ONLINE ISSN 2069-7430
ISSN-L 1841-4737
PRO LIGNO
www.proligno.ro
Vol. 10 N° 4 2014
pp. 25-34

# CHINESE LACQUER – SHORT OVERVIEW OF TRADITIONAL TECHNIQUES AND MATERIAL CHARACTERISATION FOR CONSERVATION PURPOSES

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***Abstract:***

*Chinese lacquer (urushi) is an ancient natural finishing material obtained from the sap of the lacquer trees (Rhus vernicifera). This has been used for millennia to protect and decorate furniture and various artefacts made of wood or other materials. Lacquered objects are important components of Chinese and world cultural heritage and their conservation imposes a good knowledge and understanding of the material and traditional techniques. This paper presents basic information on the Chinese lacquer as material and lacquering techniques in their historic evolution during different dynasties.*

*The experimental part looked at the physical properties of raw urushi lacquer, respectively aspect, solids content and miscibility with different solvents. A limited compatibility with white spirit as potential thinner was demonstrated. Furthermore, the microstructure of the cured film and characteristic chemical features of raw urushi lacquer as liquid and cured film were investigated. The cured film of raw urushi presents a characteristic microstructural pattern. FTIR spectroscopy revealed a partial oxidation and polymerization following processing by Kuromisation. The further curing accentuated these changes, visible as a decrease of hydroxyl absorptions bands (3400 and 1360cm$^{-1}$) occurring in parallel with an increase of carbonyl band (1740cm$^{-1}$). A strong decrease of the absorption band at 1270cm$^{-1}$, which nearly disappears for the cured film, was also observed.*

***Key words:*** *chinese lacquer; raw urushi; Kurome urushi; miscibility; microscopy; FTIR.*

## INTRODUCTION

Lacquer objects (lacquerware) are among the most valuable artefacts of different Eastern Asian civilizations (e.g. China, Japan, Vietnam, Burma, Thailand). These are objects decoratively covered with oriental lacquers, a group of ancient natural finishing materials obtained from the sap of the different lacquer trees (e.g. *Rhus* spp). Usually the lacquered objects are reddish-brown or black and very glossy due to a complex technique involving application of several thin layers of lacquer and polishing. Depending on the composition of the lacquer prepared for application, containing sometimes drying oils and/or pigments, transparent or opaque curred finishing layers with outstanding resistance and durability are obtained. The lacquer is sometimes inlaid (e.g with mother of the pearl, semi-precious stones, gold leaf) or carved. The process of laquering is very long and the objects very precious. Lacquerware includes boxes, tableware, furniture pieces, screens, buttons and even coffins painted with lacquer. The substrate for lacquering may be wood, but often other materials (e.g woven bamboo strips, leather, textiles, terracota, silk) were also employed (Zong 1985, Wei *et al*. 2011).

Lacquered objects and decorative panels were imported to Europe since 16$^{th}$ Century (Andrews 1997), and sometimes inserted in precious European furniture pieces, especially in 18$^{th}$ Century (Derrick et al 1985, Westmoreland 1985) Moreover, finishing techniques were developed in Europe to imitate the exquisite aspect of oriental lacquered objects. Such techniques are known as Vernis Martin in France and Japanning in England and America (Westmoreland 1985). Understanding the differences between these techniques and materials /techniques identification by adequate investigation methods is important for art historians, curators and restorers (Derrick et al 1985, Westmoreland 1985, Niimura 2009).

Chinese lacquer, as a comprehensive term including the actual finishing material (Urushi), the traditional preparation and application techniques and the valuable lacquered objects, refers therefore to

important world cultural heritage. Conservation of such objects is extremely complex and strongly related to material investigation and understanding. Chinese lacquer (Urushi) is a natural material with a complex and variable chemical composition (Kenjo 1985, Kumanotani 1985, Niimura 2009) and its curing through a complex bio-chemical process requires special environmental conditions (Kenjo 1985, Honda *et al.* 2008). Many aspects related to the physical and chemical properties need further investigations.

The present paper aims at offering to the reader less familiar with this subject the basic information on the Chinese lacquer as material and techniques from a historic perspective. Furthermore, an experimental laboratory study looked at some physical properties of raw Chinese lacquer and chemical features of raw material and cured film, as revealed by FTIR spectroscopy. This is part of a more extended research project on the comparative properties and ageing performance of traditional finishing materials from Europe and China, aiming to bring a contribution to the relevant scientific information needed for conservation of historic furniture.

## CHINESE LACQUER: MATERIAL AND TRADITIONAL TECHNIQUES

Chinese lacquer originates from the sap of a species of lacquer trees, *Rhus vernicifera (verniciflua)*, being tapped from the tree (Fig.1a). Sap collection starts in May - June, depending on the region and climatic conditions, best in “55 days after cherry trees blossom” (Kenjo 1985, Skalova 1985), and lasts until September-October. Three types of raw *urushi*, in decreasing order of quality, are obtained depending on the harvesting period: *hatsu urushi*, *sakari urushi* and *ura urushi.* A tree can be tapped every two or three years, the procedure being repeated for maximum 4-5 times (Du 1985). This origin of Chinese lacquer is actually depicted in the Chinese character for lacquer, resulting as a combination of three symbols meaning: wood, water and cutting marks (Fig. 1b).

The collected sap from *Rhus vernicifera* is fluid, milky white and represents a double emulsion water / *urushiol* / water. After a maturation period in dark conditions (e.g. 6 months) the external aqueous phase will be separated and the *raw urushi* lacquer is obtained (Kenjo 1985). This is viscous, milky white /cream and change rapidly colour to brownish when exposed to air (Fig.1c). Raw urushi represents in fact a colloidal system of water in oil emulsion (Burmester 1985). The oil phase is represented by a mixture of phenols, pyrocatechols (1,2-di-hydroxy–benzene) substituted in position 3 with long aliphatic chains (C15, C17), saturated and unsaturated (dienes, trienes), known under the name of *urushiol* (60-65%). The water phase dispersed in the oily phase is represented by droplets of water (20-25%) containing mono-, oligo- and polysaccharides (gummy compounds 6.5-10%) and laccase enzyme (0.1-1%). Furthermore, glycoproteins (1.2-2.8%) acting as emulsifiers are present in both the oily and water phase (Du 1985, Kumanotani 1985, Honda *et al.* 2008, Niimura 2009).


**a.** **b.** **c.**

***Fig. 1.***
***Origin, symbolisation and aspect of Chinese lacquer:***
***a - Tapping of sap from Rhus vernicifera trees; b - Chinese character for Lacquer; c - Chinese raw lacquer – raw Urushi (after different times of air exposure) and processed Kurome Lacquer.***

In order to ensure formation of a quality film in a reasonable time, raw urushi lacquer should be further processed before application as a coating material. One of the most frequently procedure used for this purpose is *Kurome*. This consists in stirring the raw material at room temperature for 1.5h, followed by heating to 40°C and further stirring for 2-4h. Special devices to ensure a thin film, good contact with oxygen,

while avoiding excessive heating are needed. During this treatment, several processes occur: partial water evaporation until 3-5%, partial polymerisation of urushiol components catalysed by laccase enzyme, separation and further fine dispersion of polysaccharides into the oily phase, alongside partial oxygen induced urushiol polymerisation (Kumanotani 1985, Honda *et al.* 2008).

Curing of Chinese lacquer is a complex oxidative polymerisation process leading to a cross-linked structure, which explains the resistance of the resulting film to solvents, water, temperature, as well as its mechanical strength, all explaining durability of lacquered objects. Two pathways were identified in the chemistry of the curing process: enzymatic polymerisation promoted by the enzyme laccase – a copper containing glycoprotein acting as oxidoreductase, and oxygen induced polymerisation (Kumanotani 1985, Honda *et al.* 2008, Niimura 2009).

The quality of the cured film, including transparency and colour, and the time of curing depends on both the quality of the lacquer (raw, Kurome, otherwise processed) and the curing conditions. Generally, temperatures of 20-30°C and high atmospheric humidity of 70-80% are employed for adequate curing of thin lacquer layers. Traditionally, an "*urushiburo*", which was a wooden box kept permanently moist by spraying with water, was employed to ensure these conditions for film drying (Kenjo 1985).

The lacquering technique has known a millenary history, evolution and diversification in China. However, the scientific investigation (e.g microscopy of cross-sections) of lacquerware from different periods demonstrated the same principle phases. These are: substrate preparation, application of a black ground containing raw urushi and fillers (black earth, ash), application of a second levelling ground, often yellow ground containing oripiment ($As_2S_3$) and more urushi, and formation of the ornamental zone composed of many thin layers of lacquer, sometimes of different colours, especially red or black, or containing silver or gold leaf. The common red pigments were Cinnabar or Vermillion ( $\beta$-HgS), while carbon black, black iron oxide and meta-cinnabar ($\alpha$-HgS) were used as black pigments. Fine intermediate polishing was done between layers (Burmester 1985).

Chinese lacquer is one of the three most famous handicraft arts of China, the other two being pottery and silk (Tian 2007). Chinese people produced and used lacquer objects since primitive times. In the Neolithic Age (10200-4500 BC) lacquer was known and used but the technique was at an exploring stage. The oldest lacquer object ever found in China is a reddish wooden bowl, dated 5000 BC, unearthed from a site of the Hemundu culture in Zhejiang (Huang and Duo 2006, Tian 2007, Zhang 2012, http://en.wikipedia.org/wiki/Lacquerware). From the Xia (2070 –1600 BC), Shang (1600–1046 BC), and Western Zhou (1046–771 BC) dynasties to the Spring and Autumn Periods (771–476 BC), lacquering techniques evolved and lacquerware experienced its initial prosperous period. Production and trading centres were developed. During the Shang Dinasty the sophisticated techniques used in the lacquer process were first developed and it became a highly artistic craft. During the Western Han dynasty (206-24 BC) lacquered painting was the main characteristic of wood furniture. Lacquer trees started to be cultivated during those times. Later, during the Tang dynasty (618-906 AD), techniques such as inlaid gold, silver or shells lacquer and coloured painting were widely used for large pieces (Zhang 2009). The Ming (1368 - 1644 AD) and Qing (1644-1912 AD) dynasties were considered as the peak of lacquerware history (Zhang 2000, Wang 2003): many very diverse and complex techniques were employed to produce objects of exquisite beauty and value (Wang 1990, Ding 2012). Table 1 highlights and exemplifies some important moments in the millennia long history of Chinese lacquer technology development.

The characteristic properties of lacquer durability and workability were utilized for various purposes. Its strength and durability provided an excellent protection for the materials used as a base, such as wood and metal, against the effects of weather or insects. Its workability made possible grinding; polishing; carving in deep and shallow relief; colouring first with black and red, and later with other colours; layering in various thicknesses; and inlaying with various materials (Skalova 1985, Zhang 2008, Wei *et al*. 2011).

ONLINE ISSN 2069-7430
ISSN-L 1841-4737
PRO LIGNO
www.proligno.ro
Vol. 10 N° 4 2014
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*Table 1*

***A short history of Chinese lacquer technology development***

| **Periods** | Cca.***5000 – 256 BC*** | ***Sui Dynasty*** (581-618 AD) ***Tang Dynasty*** (618-906 AD) | ***Ming Dynasty*** (1368-1644 AD) | ***Qing Dynasty*** (1644-1912AD) |
|---|---|---|---|---|
| Discription | People started to use lacquer for finishing; the lacquered objects' colour was black and red. | Technique development: gold, silver, shell inlaid lacquer and carved lacquer appeared. | More than 15 different lacquering techniques were employed: carved cinnabar lacquer, incised and gilded lacquer, filled-in lacquer, Coromandel, shells and mother-of-pearl inlaid lacquer | |
| Examples |  ***a*** |  ***c*** |  ***d*** |  ***f*** |
| |  ***b*** | |  ***e*** |  ***g*** |

*a. Wooden bowl with red lacquer from He Mu Du site (about 5000BC)*
*b. A painted lacquered screen from Warring states period (476-256 BC) – from (Zhang 2009)*
*c. A lacquered Chinese lute inlaid with gold and shells from the Tang dynasty (618-906 AD) –from (Zhang 2012)*
*d. Ming Dynasty (1368 - 1644 AD) table finished with shells and mother-of-pearl inlaid lacquer – from (Ding 2012)*
*e. Ming Dynasty (1368 - 1644 AD) table finished with red carved lacquer –from (Ding 2012)*
*f. Cabinet from Qing dynasty (1644-1912) – lacquer with gilding and colour painting with flower and bird motifs (Nei 2012)*
*g. Qing 'officiu hat' armchair finished with gold inlaid lacquer (Ding 2012)*

## MATERIALS AND METHODS

In the experimental part, the physical properties of raw urushi lacquer, respectively aspect, solids content and miscibility with different solvents were determined. Furthermore, the microstructure of the cured film and characteristic chemical features of raw urushi lacquer as liquid and cured film were investigated. The materials and methods employed are briefly presented.

### *Materials*

Raw urushi was purchased from East Lake Oil Co., Ltd., Jiang Su province China. Different organic solvents presented in Table 2, were employed in the miscibility tests.

*Table 2*

***Solvents for miscibility tests***

| **Solvent** | **Code** | **Purity/concentration** | **Source** |
|---|---|---|---|
| Acetone | AC | Chemical grade 99% | Chemical company - Iasi |
| Methyl acetate | MA | Chemical grade 99% | Chemical company- Iasi |
| Ethanol | ET | Chemical grade 96% | Chemical company- Iasi |
| White spirit | WS | Technical 50% | SC TURNIR- Tirgoviste |

***Macroscopic and microscopic aspect***

The raw urushi lacquer was first homogenised by thorough stirring with a glass rod. The product was initially of creamy colour but rapidly changed to brownish colour in contact with air due to rapid oxidation, behaviour characteristic to phenols. Therefore, a small amount necessary for the envisaged physical and chemical tests was taken from the original container, which was sealed back for further use.

Thin and thicker films were applied freely by hand on microscopic glass lamellas and allowed to cure at 25-30°C and 75-80% RH for minimum 48h. The microstructure of the cured film was observed in transmitted light at a stereomicroscope OPTIKA SZM fitted with image capturing system (OPTICAM PRO3) at different magnifications (40x, 80x ).

***Solids content***

The solids content of the raw urushi lacquer was determined by the classic gravimetric method. An amount of about 4g product was weighted in a weighing ampoule and then allowed to dry in a laboratory oven at 103±2°C until constant weight. The percentage of dry residue reported to the initial weight of product was calculated.

***Miscibility with solvents***

The tested raw urushi lacquer was very viscous, more like a paste, thus some miscibility tests with different solvents were necessary.

An original testing procedure, as schematically presented in Fig. 2, was adopted:

- Step 1: a drop of raw lacquer (CL) was put on a microscope glass lamella (2 lamellas/solvent);
- Step 2: 2-3 drops of solvent were thoroughly added on top of CL from a pipette and allowed to interact;
- Step 3: glass lamellas from step 2 were divided into two groups: A, B, for which the procedure was slightly different. For the A group (spontaneous miscibility) it was observed the phenomena occurring when the solvent came into contact with CL, respectively evidence of solubilisation and migration of lacquer components or evident incompatibility resulting in precipitation / whitening.

For the group B (forced miscibility) the two components (CL and solvent) were stirred by a glass rod and further laid as a film on the glass lamella. Compatibility (easy to mix and formation of a homogeneous mixture) or non-compatibility (tendency of separation, precipitation) phenomena were observed during stirring.

- Step 4: after a period of about 24h (necessary for solvents evaporation) all the glass lamellas were examined under stereomicroscope (same equipment as above) in transmitted light (TL). From each sample there were taken several images at magnifications of 40x and 80x. The microscopic examination looked at the boundary area between lacquer and solvent (area A) were the formation of a film would be the result of solubility of CL components into the solvent, respectively to the aspect / continuity / homogeneity of the film resulting from the forced mixing of CL with the solvent (area B).

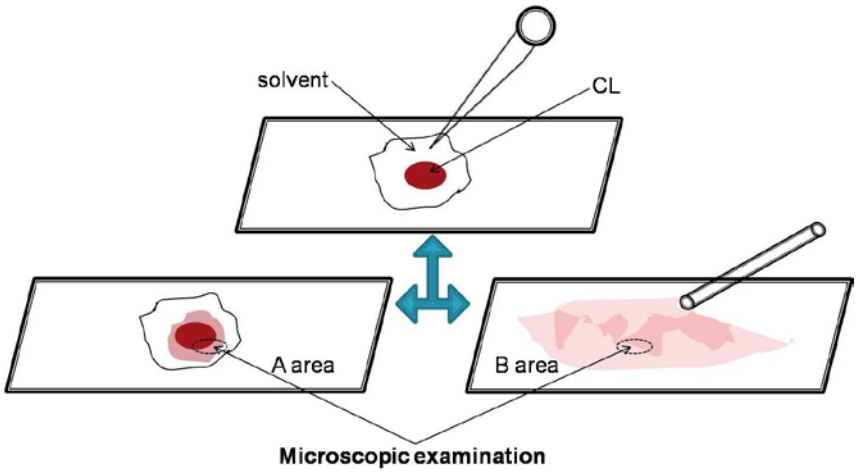



***Fig. 2.***
***A sketch of miscibility testing.***

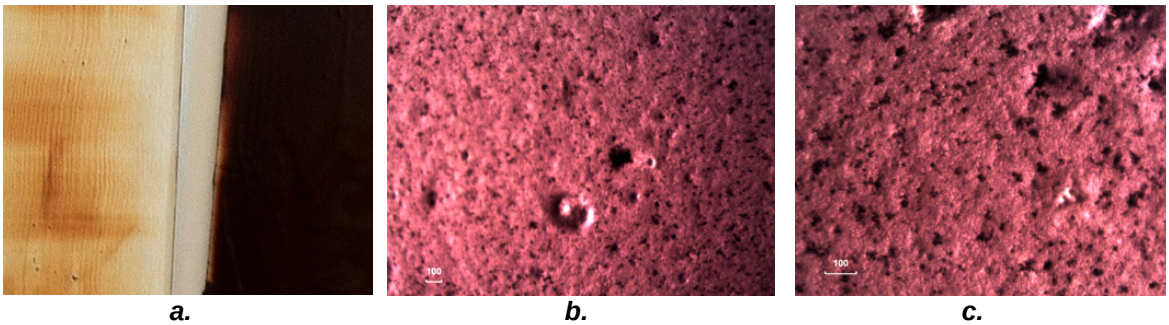
***FTIR investigation***

FTIR (Fourier Transformed Infra Red) investigations were performed on liquid raw urushi, Kurome urushi and the cured film resulted on glass lamella. FTIR spectra were recorded employing an ALPHA FTIR Spectrometer (BRUKER) equipped with ATR (attenuated total reflection) module. The spectra were recorded in the range 4000 – 400cm$^{-1}$ at a resolution of 4cm$^{-1}$, each spectrum resulting from 32 scans. Spectra were further processed for baseline correction, smoothing and normalisation employing the equipment software OPUS 7.2.

**RESULTS AND DISCUSSIONS**

***Solids content, macroscopic and microscopic aspect***

The raw urushi lacquer had a solids content of 45,8%. As already mentioned, the initial light creamy colour changed rapidly to brownish in contact with air. The liquid films on the glass lamellas (brown opalescent) turned into reddish – brown, semitransparent hard and glossy films by curing within 48h at around 25°C and 75% RH. The microscopic examination of the cured films showed some surface roughness and sort of a granular structure, as presented in Fig. 3. This agrees with similar research and can be correlated with the film formation mechanism and speed of water evaporation (Honda *et al.* 2008), and also related to the complex chemical composition of the lacquer, respectively the presence of two non-miscible phases: the polymerised phenols and inclusions of polysaccharides, which may have been not well dispersed (Kumanotani 1985).



***a.*** ***b.*** ***c.***

***Fig. 3.***
***Macroscopic aspect of thin and thick liquid films of raw Chinese lacquer (a) and microscopic structure of solid films at magnification 40x (b) and 80x (c).***

***Miscibility with solvents***

The tests with solvents revealed differences of compatibility between the raw Chinese lacquer and the solvents considered. According to the micrographs in Fig. 4 and the phenomena observed during the experiments, the following aspects related to the miscibility of Chinese lacquer could be noted:

☐***Acetone***: some components of CL are spontaneously dissolved in acetone and form a film, whilst other components precipitate, forming a white ring in the boundary A area. By stirring, acetone and Chinese lacquer mixed uniformly, so an homogeneous film resulted in B area. This is in accordance to previous research (Kumanotani 1985, Du 1985) showing that acetone can be used to separate urushiol (the mixture of phenols) from the polysaccharides and glycoproteins which precipitate.

☐***Methyl acetate***: when the solvent was added a tendency of dissolution was evident, but after a while precipitation occurred and a white zone appeared in A area. By stirring, methyl acetate and Chinese lacquer mixed uniformly but viscosity increased; a uniform film resulted in B area.

☐***Ethanol:*** a white zone appeared in A area, the mixture become viscous. By stirring, ethanol and Chinese lacquer could be mixed uniformly, but they separated after several minutes; the film obtained in B area denotes some roughness and non-uniformity. These denote incompatibility of CL with ethanol.

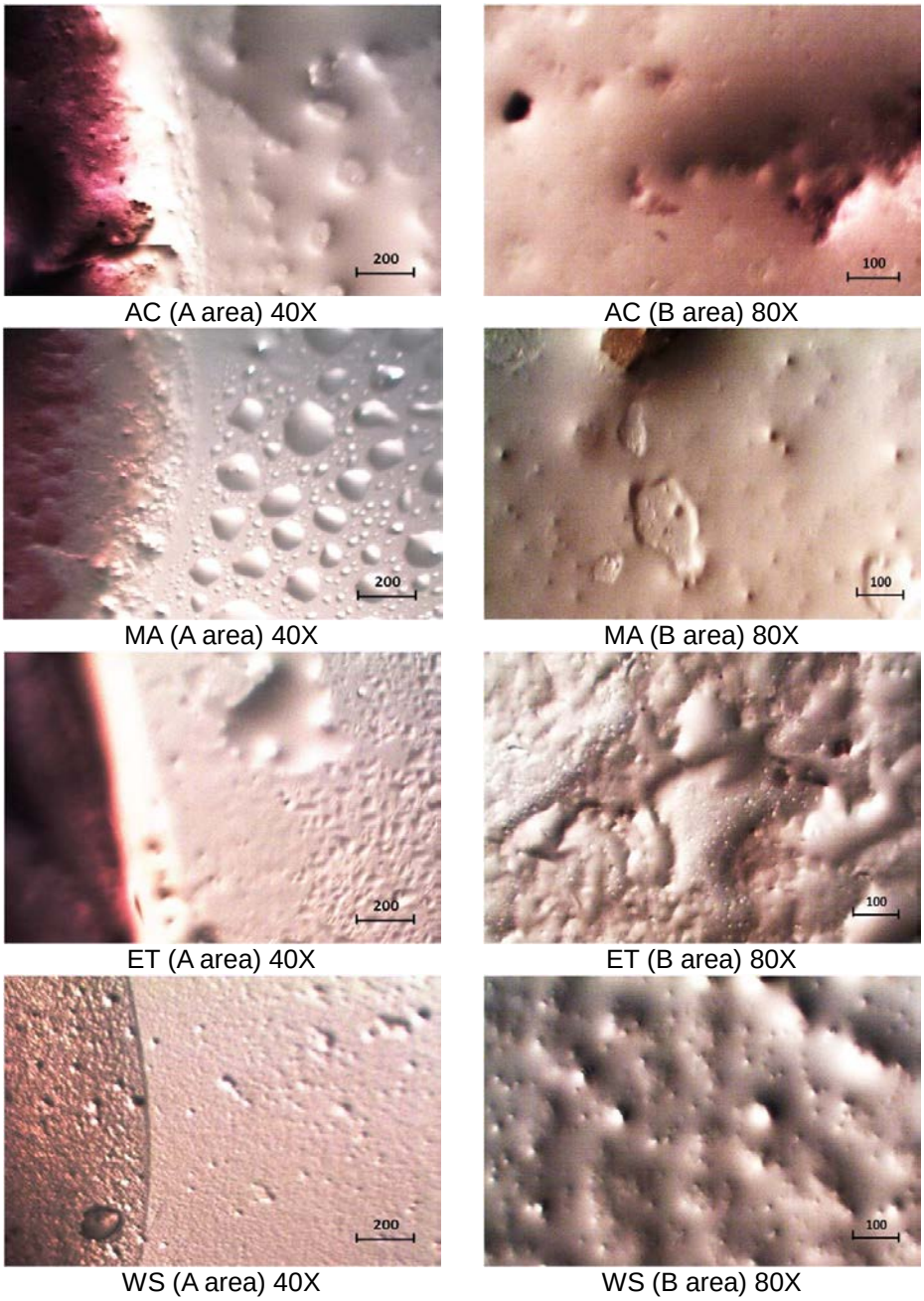



***Fig. 4.***
***Micrographs illustrating the miscibility / limited compatibility of raw Chinese lacquer with different solvents.***

□ ***White spirit:*** a tendency of migration of CL components into white spirit is observed resulting an homogeneous but opalescent film in A area. By stirring, white spirit and Chinese lacquer mixed well and a quite uniform film was obtained in the B area.

From these tests resulted that raw Chinese lacquer would be compatible with white spirit. This is in accordance with some literature data (Tan 2005, Zhang 2000). However, mixtures of raw urushi and white spirit, prepared at a ratio of 1:1 (volume based) were steady only for about less 24h at room temperature. After this period the mixture separated in two phases; a precipitate and a clear liquid. Thus, it could be concluded that the compatibility of Chinese lacquer with white spirit is limited and more experiments on the miscibility limits would be necessary.

***Chemical features revealed by* FTIR *investigation***

In Fig. 5 are presented the FTIR–ATR spectra experimentally recorded for Chinese lacquer (raw urushi, Kurome lacquer, cured lacquer), while in Table 3 are summarised the main absorbance bands in these spectra and their assignment in accordance to the literature. To facilitate understanding of bands assignment to different functional groups/structural elements, the chemical structure of Urushiol mixture, as presented in literature, is also included.

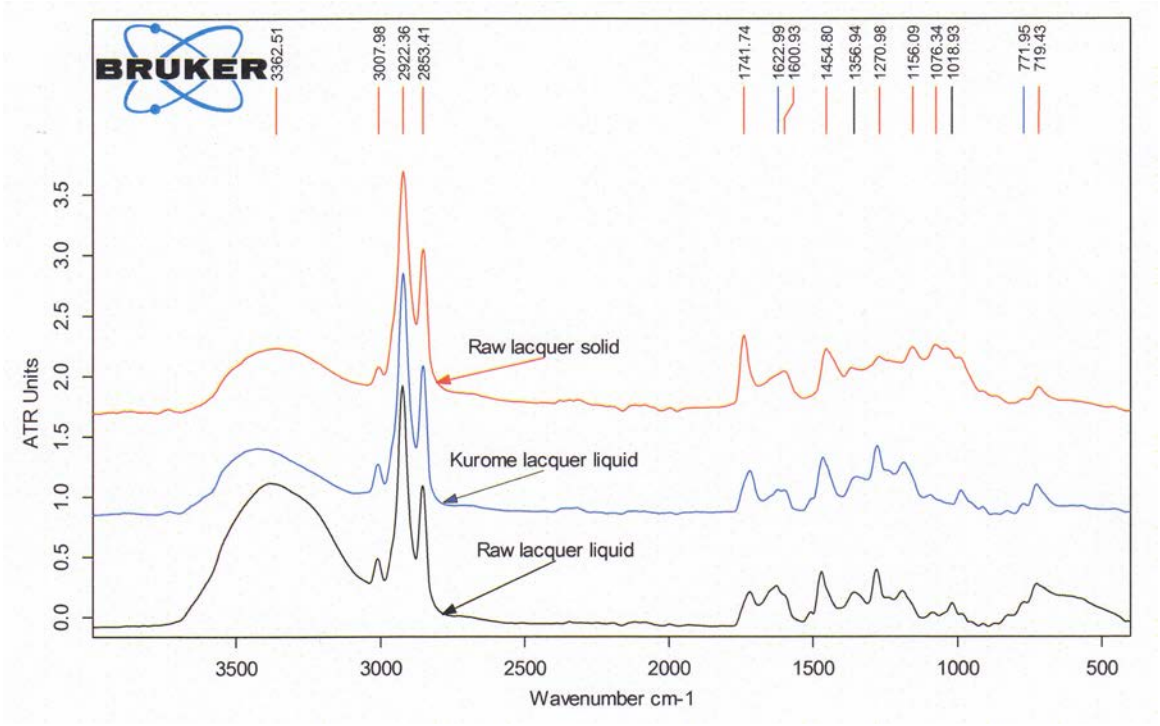


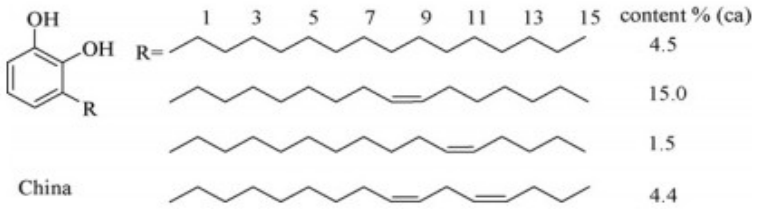

***Fig. 5.***
***FTIR –ATR spectra of Chinese lacquer: raw urushi (black), Kurome urushi (blue) and hardened film (red).***

The FTIR spectra reflect the chemical structural features of Chinese lacquer and changes occurring during Kuromisation and curing. The main absorption bands in the raw urushi lacquer spectra are those for hydroxyl (~ 3400cm$^{-1}$ and ~ 1360cm$^{-1}$), methylene (2922 and 2853cm$^{-1}$) and the aromatic ring (~1600, 1455 and 920cm$^{-1}$). They denote a phenolic structure with a long aliphatic side chain, as shown in the structural formula above. Presence of aliphatic double bands C=C is highlighted by absorptions at 1623 and 720cm$^{-1}$, while carbonyl groups determine the small absorption band at ~ 1740cm$^{-1}$. Absorptions bands at 1280, 732 and 698cm$^{-1}$ were assigned by Honda et al (2008) to catechol in monomer form.

Comparing the spectra of raw lacquer with those of Kurome lacquer and cured film some alterations are visible. The most obvious are a decrease of hydroxyl related bands (3400 and 1360cm$^{-1}$) occurring in parallel with an increase of carbonyl band (1740cm$^{-1}$). These are due to the laccase induced oxidation process when phenolic hydroxyl groups are transformed into quinone groups. Furthermore, through a radicalic mechanism C-C and C-O coupling reactions occur. The vibrations characteristic to aromatic C-H stretching and bending (3010 and 920cm$^{-1}$) and C=C (1623, 720cm$^{-1}$) are slightly decreasing. A strong decrease of the absorption band at 1270cm$^{-1}$, which nearly disappears for the cured film, is in accordance to previous research (Honda *et al*. 2008).

*Table 3*

***Bands assignment for the FTIR spectra of Chinese lacquer (experimental vs. literature)***

| **Chemical structure of URUSHIOL**<br><br>(proposed by Lu *et al*. 2006) |  | | |
|---|---|---|---|
| **Absorption band position, cm$^{-1}$** | | **Assignment** | **References** |
| **Experimental** | **Literature** | | |
| 3392 | 3600-3200 | -OH stretching | Derrick *et al*. 1999<br>Honda *et al*. 2008 |
| 3008 | 3030<br>3100-3000 | =CH stretching<br>Aromatic C-H stretching | Tellez *et al*. 2009<br>Derrick *et al*. 1999 |
| 2922 | 2925 | C-H stretching (asymm) in aliphatic methylene | Derrick *et al*. 1999 |
| 2853 | 2850 | C-H stretching (symm) in aliphatic methylene | Derrick *et al*. 1999 |
| 1742 | 1730 -1740 | Unconjugated C=O stretching (ketones) | Derrick *et al*. 1999 |
| 1601<br>1623 | ~1600<br>1650-1640 | Aromatic skeletal vibration<br>C=C stretch | Derrick *et al*. 1999<br>Tellez *et al*. 2009 |
| 1455 | 1450-1465 | Aromatic skeletal vibration<br>C-H bending for methylene and methyl | Derrick *et al*. 1999 |
| 1357 | 1420-1330 | -OH bending | Derrick *et al*. 1999<br>Honda *et al*. 2008 |
| 1271<br>1156<br>1076 | 1000-1260<br>1270 | C-O- stretch<br>Catechol | Derrick *et al*. 1999<br>Honda *et al*. 2008 |
| 920 | 920 | C-H bending vibration aromatic ring | Derrick *et al*. 1999 |
| 720 | 720<br>730 | Stretching of cis CH=CH<br>C-H rocking in methylene | Tellez *et al*. 2009<br>Derrick *et al*. 1999 |

## CONCLUSIONS

Chinese lacquer (urushi) is an ancient natural finishing material obtained from the sap of the lacquer trees (*Rhus vernicifera*). This has been used for millennia to protect and decorate furniture and various artefacts made of wood or other materials. Lacquered objects cumulate exquisite aesthetics with tradition, craft, technical skills and important historic information, being therefore important components of Chinese and world cultural heritage. Their conservation imposes a good knowledge and understanding of the material and traditional techniques, which can not been achieved without research and investigations. The complex chemical composition of urushi and its curing through a bio-chemical process are still a scientific challenge for researchers around the world.

The experiments presented in this paper looked at the miscibility of the raw lacquer with four organic solvents and showed a limited compatibility with white spirit as potential thinner. The cured film of raw urushi presents a characteristic microstructural pattern. Processing of raw urushi by stirring and heating at 40°C, known as Kuromisation, brought about not only physical changes (colour, viscosity, water content), but also chemical changes. These were revealed by FTIR spectroscopy and resided in a partial oxidation and polymerization. The further curing accentuated these changes, visible as a decrease of hydroxyl absorptions bands (3400 and 1360cm$^{-1}$) occurring in parallel with an increase of carbonyl band (1740cm$^{-1}$). These are due to the laccase induced oxidation process when phenolic hydroxyl groups are transformed into quinone groups. A strong decrease of the absorption band at 1270cm$^{-1}$, which nearly disappears for the cured film, is in accordance to previously reported research.

Further scheduled research will look at the ageing resistance of the cured films.

**ACKNOWLEDGEMENTS**

*The authors acknowledge the structural funds project PRO-DD (POS-CCE, O.2.2.1., ID 123, SMIS 2637, ctr. No 11/2009) for providing the infrastructure used in this work.*

*We are also grateful to professor YI Song Lin, from Bei Jing Forestry University for providing the raw Chinese lacquer for experiments*

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