Characterisation of the “specific polarity” of aged terpeneous resin coatings using IR spectroscopy to predict the solubility of varnish materials

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Project focus

The main point of research of this scientific conservation-restoration MA project at the Bern University of Applied Sciences is to optimise the infrared spectroscopic (FTIR) methodology in order to generate basic information for conservation treatments. Of particular interest is the correlation of the oxidation state to the change in solubility of a varnish material, which is relevant to the choice of effective solvents in surface cleaning treatments. Spectral information is hereby helpful to better understand the material properties. For example, the knowledge on the „specific polarity“, which relates to the functional groups present, is of direct relevance in this context. The various functional groups exhibit different intermolecular interaction capacities, e.g. polarity / polarisability, as well as aprotic and protic properties. Thus, such spectral data delivers powerful information to predict the solubility of a varnish material.

Infrared spectroscopy

The distinction of oxidised terpenes is, unfortunately, hindered by the interference of similar IR-bands summing up to a non-distinctive broad carbonyl signal. Thus, a derivatisation technique involving gaseous sulfur tetrafluoride SF_4 was applied to discriminate interfering bands. This method, presented elswhere (Zumbühl et al. 2014), reliably achieves identification and quantification of primary acids, α,β-unsaturated acids, tertiary acids, peroxy acids, esters, ketones, and α,β-unsaturated ketones - even in complex mixtures of terpenes (**Fig. 1**). This on-going project explores the ageing behaviour of varnish materials. Accelerated light ageing under indoor (glass filter LP 330mm) and museum conditions (UV-filter LP 390mm) revealed, that various functional groups are being formed at different proportions during individual stages of the treatment (**Fig. 2**).

Zumbühl, S. et al. (2014). Applied Spectroscopy 68(4): 458-465.

Terpeneous varnish solubility

The kinetics of dissolution were studied within the context of surface cleaning. A new parametrisation system (Zumbühl 2014) was used to describe the material solubility. The current study explores the change in solubility of various terpeneous varnish materials in relation to ageing under different light conditions. It systematically documents the behaviour of dammar, mastic colophony and sandarac upon interaction with 21 selected solvents along the polarity scale. The result is visualised graphically in **Fig. 3**. Changes in solubility between fresh and aged materials (**Fig. 3**) can be linked to progressive development of functional groups during accelerated ageing (**Fig. 2**). The study reveals that the characterisation of the „specific polarity“ using infrared spectroscopy on micro-samples delivers helpful information to responsibly plan the cleaning treatment of artwork.

Zumbühl S. (2014). ICOM-CC Melbourne, Art. 1610, 11p.

Effects of SF_4 derivatisation on FTIR resin analysis

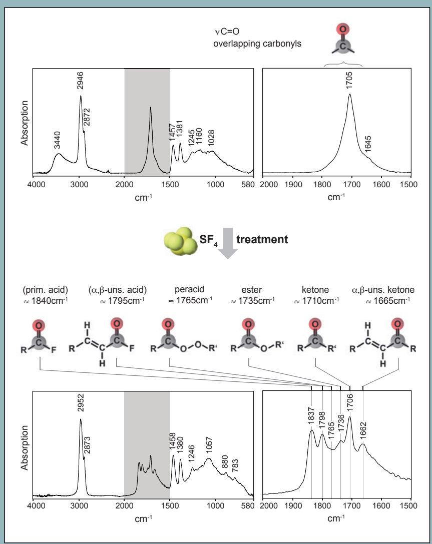

Figure 1: Derivatisation of mastic resins using sulphur tetrafluoride (SF_4). This triterpene resin has multiple functional groups within the raw material already. Spectra in the top row show the FTIR response prior to derivatisation, whereas the spectra in the bottom row document the FTIR response upon treatment with SF_4

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Functional groups formed during ageing

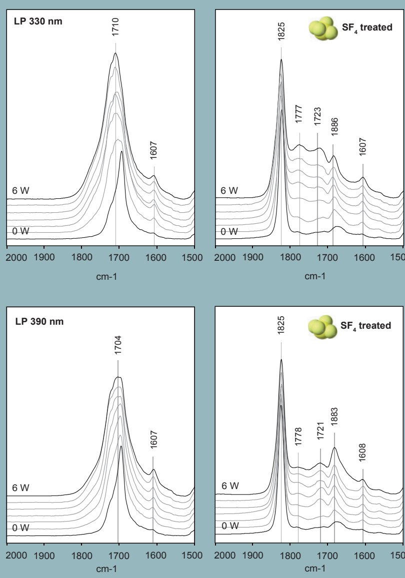

Figure 2 (above): Infrared spectra of colophony varnish afer accelerated light ageing behind window glass (top row LP 330nm) and UV-filtering glass (bottom row LP 390nm) for 6 weeks (6W). The graphs show the carbonyl region before (left column) and after derivatisation using SF_4 (right column).

Figure 3 (right): Solubility plot of fresh (top, 0W) and matured colophony varnish aged (6W) under different light conditions (center LP 330nm; bottom LP 390nm). The rate of solubility normalised to the fastest acting solvent is presented as variably sized circles, whereby the diameter corresponds to the relative intensity of action of the different solvents.

Changes in solubility of resins upon ageing