Field microscopy applied to the understanding of the technology and conservation of wall paintings

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Abstract. Wall paintings are complex, heterogeneous structures open to the environment and composed of multiple layers. Additionally, they are frequently located in remote areas, where basic infrastructure is not available. The causes and mechanisms involved in the complex deterioration phenomena, which wall paintings are often subjected to, are therefore difficult to identify and address. Developments have been made to target conservation issues in situ with non-invasive and portable investigative techniques, however standard practice to investigate stratigraphic information still involves analysing samples in a laboratory setting. In remote sites this approach is inefficient, as in many cases decisions on treatment cannot wait for the results of the laboratory examinations. This may have serious consequences on the integrity of the painting. This paper describes adaptation of classical methods for the in-situ preparation of cross-sections, thin-sections and dispersions utilising custom-made equipment. The methodology allows for a direct examination of samples in-situ, using a portable polarizing microscope (field microscopy). The system was adapted and developed to not only capture incident and transmitted light, but also to capture multispectral images (infrared-, ultraviolet-reflected and ultraviolet- and visible-induced luminescence), using external radiation sources. Comparisons with benchtop equipment are made to demonstrate the efficacy and usefulness of the process.

1. Introduction

Incident light microscopy is one of the most useful tools employed in art conservation. A great deal of information may be gained quickly, with a relative small capital investment and without the need for specialist technicians. Microscopy, defined by McCrone as ‘the application of any enlarged-image process enabling visualization of objects nominally invisible to the unaided human eye’ [1], permits a skilled and trained conservator to identify aspects of original technology or of condition that would otherwise be missed. The polarised light microscope (PLM) takes this process forward, allowing visualisation, interpretation and chemical identification of microscopic objects through their examination between polarising filters. Optical properties not visible in a standard incident light microscope may thus be observed. Materials identifiable with this micro-analytical tool include pigments, fibres, plaster components and alteration products [2].

In the conservation of wall painting, samples are collected from a site, which is often remote and only accessible for investigation for a limited period of time [3]. In broad terms, two types of samples for microscopy are commonly collected:
1. A first sample typology is collected to investigate the original technology (to observe the composition and complexity of paint and plaster layers, for example) and to understand past conservation/restoration interventions (such as the presence of a later varnish or repainting); and
2. A second typology is researched to scrutinize conservation issues and the effectiveness of current conservation interventions (for instance to observe freshly consolidated plaster, or the composition of experimental grouts or of salt species).

Under standard working conditions, and following appropriate visual observations and non-invasive investigations [4], microscopic samples are taken back to a laboratory for examination. Considerable time may pass between the collection of samples and their analysis, with an inevitable lack of continuity in the conservation process. In addition, as the conservation process is iterative in nature [5], questions raised during the analysis cannot be addressed before a new campaign takes place, and new samples are collected [6]. Moreover, in transport friable samples may suffer from mechanical damage due to vibration.

1.1. Cross-sections, thin-sections and dispersions

A wall painting, in the field of conservation, is considered a complex, heterogeneous structure made up of multiple layers. In order to study such an intricate object, it is common practice to collect a minute sample and mount it as a cross-section. This enables a conservator to view the stratigraphy of the wall painting under a microscope, and to gain crucial information on the technology and state of conservation.

To obtain a cross-section, the sample is embedded into a mounting medium, commonly an organic resin, and polished on one side until the paint stratigraphy is exposed. Alternatively, the sample may be cut using a microtome. By observing various characteristics of the particles in the paint layers, such as colour, size, it may be possible to preliminarily characterise the pigments [7], as well as to obtain important information on optical and physical properties related to their change and ageing [8].

Histochemical tests may be used to investigate organic binders [9][10].

A thin-section may be obtained from a cross-section adhered to a glass slide and polished to a standard thickness of 30 µm [11][12]. In conservation literature, a range of thicknesses between 5 and 50 µm is reported [13]. This thickness is estimated by comparing the birefringence colours of known minerals within the section to the Michel-Lévy chart. Quartz grains are often added next to the section to be used as a control. Samples prepared in such a way are more commonly analysed under PLM, and have traditionally been used in petrography and micromorphology studies [14], to identify inorganic materials and locate their source, to group items with a common source, or, less frequently, to provide information on conservation treatments and on the state of deterioration [12]. In wall painting conservation, thin-sections are usually prepared for plaster analysis. Observed microscopically and coupled with XRD analysis, mineralogical components and their origin may be identified [15]. Moreover, thin-sections of grouts allow to observe their morphology, particle distribution and, when impregnated with coloured resin, their porosity [16].

Pigment dispersions are created by immobilising pigment particles on a microscope slide with a resin of a known refractive index [2]. Their microscopical observation may aid in the identification of the pigments present and their source, through the study of the combination of their optical properties visible in plane- and crossed-polarised light [17][18]. Pigment dispersions may be especially useful for the differentiation between natural and synthetic pigments [8].

2. Embedding resins for in-situ preparation of samples: characteristics and field constrains

Many embedding materials and equipment for sample preparation are available. In order to select the most appropriate ones, required properties for resins used in the preparation of cross-sections and thin-sections in situ need to be identified. These are based on a literature review of the most frequently used materials and on the field constraints considered below.

2.1. Definition of field constrains and of resin properties

Preparation of samples in situ may be hindered by field constraints, outlined below:
• **Availability of materials**: materials involved should be easily available (dependant on countries);
• **Portability**: equipment must be portable, lightweight, not bulky, and durable;
• **Transportability**: equipment and materials must comply with relevant health and safety regulations for transport. This must be checked with the competent authorities on a case-by-case basis;
• **Ease of use**: the methodology should be relatively simple and reproducible, without the need for highly specialized skills and resources. Ideally, it should be possible for the methodology to be implemented even with scarce resources, such as when water or electricity are not available; and
• **Cost**: cost should be reasonable and necessary material and equipment should be affordable by small private conservation firms/individuals. Ideally, equipment and materials can be sourced in the country where the measurements take place.

The characteristics that should be kept in mind while choosing an adequate embedding material pertain both to its immediate use, so to its behaviour as soon as it interacts with the sample (working properties), and to its performance in the future with respect to the sample, after its initial use and manipulation (performance characteristics). Working properties include:

• **High transparency**: allows visibility of the sample during preparation;
• **Low viscosity**: maximises penetration within the pores of the sample;
• **Fast curing time**: ensures that samples can be analysed within tight time constraints;
• **Simple preparation methodology**: facilitates the preparation of the samples in adverse conditions;
• **Long shelf life**: minimizes costs and resources; and
• **Health and safety**: the resin must be safe to use for the users and the environment.

Performance characteristics include:

• **Hardness**: a hard resin protects the sample, and allows polishing without sample deformation;
• **Uniformity**: colour uniformity increases reproducibility of the results;
• **Low shrinkage**: minimizes the risk of sample deformation;
• **Cold-curing mechanism**: prevents overheating and subsequent damage of the sample;
• **Low luminescence**: maximises the detection of weakly luminescent compounds in the sample; and
• **High stability**: minimises colour change and deterioration over time, allowing future analysis of the sample.

2.2. **Common embedding resins**

The sensitivity and fragility of samples of cultural heritage materials require the use of cold-curing embedding resins. The process of curing, however, even in cold systems, usually produces heat to different extents associated with the polymerization process. Generally, slow-setting resins produce less heat than fast-setting resins, which can reach temperatures as high as 100 °C [22]. Cold systems can be further classified as thermosetting, (including epoxy and polyester resins), and thermoplastic, (Canada balsam, acrylics and cyanoacrylates) polymers.

Thermosetting polymers are more resistant to solvents, abrasion and temperature, so they are generally considered to perform better [13]. For thin-sections, the resin functions as an adhesive—to hold the sample on the glass slide and to allow manipulation and storage—as well as a consolidant, especially in cases of powdery samples, to preserve its structure during polishing [23]. Epoxies are frequently deemed the best choice due to general performance characteristics [22][13], although polyester resins are often preferred due to their minimal shrinkage, low cost, moderate toxicity, ease of preparation and availability [24]. Both these types are suitable for luminescence imaging [13].
Identification of pigments in dispersion relies on comparison with known references, frequently prepared with Cargille Meltmount™, which is by far the most commonly used medium [19][20][21].

3. Method for in-situ sample preparation

Much of the equipment needed for the preparation of cross-sections, thin-sections and dispersions is already portable. For the few exceptions, compact alternatives were sought. For cross- and thin-sections, the main points to consider are the way in which the sample is embedded in the resin, the grinding, polishing and vacuum-impregnation processes.

Preparation of cross-sections in situ follows standard procedures [7], except for the grinding and polishing which will have to be done manually (and evenly) on wet polishing cloths of various grades (120, 500, 1000, 2400, 4000, 8000 and 12000). If the section will be further polished to a thin-section after examination and imaging, some quartz grains must be placed in the sample-holder adjacent to the sample. The grains must be exposed when the cross-section is finalised, then the exposed side may be glued to a microscope glass-slide and further polished on the other side. The section must be frequently checked under cross-polarised light, until the quartz grains reach the colours in accordance with the right thickness (according to the Michel- Lévy birefringence chart). For 30 μm, the quartz should appear beige-grey. The adaptation of laboratory equipment to on-site equipment for dispersion was very straightforward, with the only point to consider being the heating process for the resin. A cup warmer, able to heat above 65 °C in order to melt the resin, was selected as a lightweight and compact alternative to a standard hot plate.

The quality of samples prepared is comparable to that obtained using standard laboratory equipment. The benefits of preparing cross-sections, thin-sections and dispersions anywhere with minimal equipment, allowing a conservator to gain information quickly and effectively, greatly outweigh the longer time required to prepare samples with this method, compared to laboratory preparation.

3.1. Custom-made sample holders

Manual polishing of sections requires more time than with the use of polishing wheels or microtomes [7]. It follows that samples should be embedded in a reduced amount of resin, to decrease the amount of polishing required. Moreover, smaller resin blocks will also reduce the cost, the amount of resin and hardener to be transported and the health and safety concerns1. For this study, sample-holders were thus designed and converted in a CAD file using AutoCAD software. They were then 3D printed in acrylonitrile butadiene styrene (ABS), a general-purpose, low-cost plastic material. Finally, they were replicated with silicone moulds, and reproduced with resin (figure 1).

Figure 1. Sample holders in resin (a) and their measurements (b).

The sample-holders are thin rectangular blocks with an elliptical well reaching the middle of the block. The sample is placed in the well, which is then filled with resin. The rationale behind this design is the following:

- its thinness (5 mm) minimises the amount of resin needed for embedding and polishing the sample. The sample-holder is at the same time sufficiently large to be conveniently handled;

1 Valentine Welsh [VW Fecit] also designed ready-made sample holders (easysections.com). The holders designed for this study allow for the positioning of the sample in the middle of the holder, minimizing the risk of overpolishing.
• the shape and width of the well (3 mm) allows placement of quartz or calcite crystals alongside the sample, needed for the preparation of thin-sections;
• crucially, it allows for the positioning of the sample in the middle of the block, minimising the risk of over-polishing when manually preparing thin sections;
• the sample holders are small and lightweight and can be prepared in the laboratory and carried ready-made on site.

3.2. **Vacuum pump and chamber**

Thin-sections might require thorough embedding of the sample in the resin, especially for decohesive or very porous samples. This serves to stabilise the sample during its preparation as a thin-section [25]. Samples are thus frequently embedded under vacuum [14]. Whether a vacuum is desirable, or required, also depends on the fluidity of the resin chosen. Vacuum removes air from the pores in the sample, and also the bubbles that might have formed within the resin. The more viscous the resin, the easier it is for air bubbles to remain trapped. If present, they may obstruct the observation of the sample: with resins of lower hardness than epoxy, air bubbles may be enlarged during grinding due to the pressure created within them by loose resin particles, which may also remain trapped in the air bubbles. For our purposes, a high-level of vacuum such as that achieved by a rotary vane pump used in laboratories is not required, thus it was deemed suitable to use a hand-held vacuum pump (used to bleed breaks in cars and motorcycles). This pump is able to reach maximum 760 mmHg pressure, about 1 bar. A small, modified Tupperware container was initially used as a vacuum chamber [14], but an economical mini-vacuum desiccator was later found to perform better (figure 2).

![Figure 2](image)

**Figure 2.** Materials required for in-situ preparation of cross- and thin-sections (a), including from left and clockwise: vacuum-pump and Tupperware used as a vacuum-chamber, epoxy glue to adhere the cross section to the polishing slide, resin to embed the section, quartz grains to determine the thickness of the thin-section, polishing cloths, glass-cutter to cut regular glass slides to size to fit into the thin-section holder. In (b), a mini-vacuum desiccator, which connects to the vacuum pump in (a).

4. **Examination of samples: comparison between portable and benchtop microscopes**

Once the samples are prepared, they can be examined in situ with the Goren-Pol microscope. This portable polarising microscope is principally designed for observation of samples in transmitted light; however, incident illumination can easily be provided externally by placing a torch on a stand. A high-end benchtop Leica DMRD was utilized to compare the quality of the examination and imaging of samples obtainable with the Goren-Pol. By removing the left eyepiece, a camera may be fitted on the Goren-Pol, to allow capturing of images both in incident and transmitted light configurations.
Figure 3. Plane-polarised light image of a thin-section (a and b). Degradation of plaster due to salts. The sample was first mounted as a cross-section (c).

Figure 4. Particle of azurite imaged in cross-polarised light with the Goren-Pol (a) and Leica (b), at 600x and 500x respectively.

Figure 5. Incident light images of a cross-section of ultramarine.

**Transmitted light:** In general, examination with the Goren-Pol yields excellent results, comparable in quality with the Leica, especially in transmitted light (figures 3 and 4). The light source of the Goren-Pol (LED) is whiter than that of the Leica (halogen) and therefore provides a better colour rendition of the various phenomena used for the identification of materials. **Incident light:** Examination with both microscopes in incident light is limited to about 200-400x. While very flat samples yield better results, in general, images in incident light captured with the two microscopes can be different due to the direction of the incident light and the difference in type of source. The light for the Goren-Pol, being external, reaches the sample at a different angle than it does with the Leica, equipped with an internal source (figure 5). This may result in a more raking view of the sample, depending on the working distance of the objective. Nonetheless, incident light examination of thin-sections and dispersions is often clearer with the Goren than with the Leica, especially at high magnifications, as images appear less hazy and better defined, even if slightly raking.

**5. Adaptation of the Goren-Pol to Multispectral Imaging (MSI)**

The Goren-Pol microscope was adapted for multispectral imaging of microscopic samples, easily done by using different radiation sources, filters and modified cameras. Set-ups for imaging of cross-sections in infrared-reflecting (IRR), visible-induced infrared luminescence (VIL) and ultraviolet-induced visible luminescence (UVL) are briefly described below (Figure 6). Further avenues of investigation include using more advanced and sensitive cameras and a more specific system of filters.
Figure 6. Set-up with the Goren-Pol: (a) a filtered UV torch (5W, 365 nm, Schott UG11 filter) illuminates the sample; the camera, equipped with a B&W420 filter, is connected to a computer through tethering software. In (b) a standard filtered visible torch (400-700 nm, 240 lm, Schott BG38 filter) excites the sample with visible light, re-emitted in the IR range as seen on the camera screen. The camera is equipped with a Schott RG830 filter.

5.1. Ultraviolet-induced luminescence imaging in the visible range (UVL)
The Goren-Pol requires an external UV light source for UVL observation and imaging, while the Leica is equipped with an internal one. A 5W LED UV torch with an emission centred at 365 nm was used for this research as the external UV radiation source. A Schott UG11 excitation filter was positioned in front of the torch, to remove the unwanted violet-blue radiation. The UV radiation excites luminescence from the sample, which can be observed through the eyepiece. For visual observation, the eyepieces must be equipped with an emission UV-blocking filter, in order to block the reflected UV radiation and avoid damage to the unprotected eye. During image capture, a B&W420 emission filter was placed between the relay lens and the camera sensor. Difficulties in focusing weakly fluorescent samples can be overcome by using a visible LED torch.

Up to 200x, excellent results were obtained with the Goren-Pol, only requiring slightly more adjustment of exposure to obtain accurate images comparable to those of the Leica (figure 7).

Figure 7. UVL image of a cross-section with a fluorescent paint layer containing particles of bright yellow (Indian yellow) [26].

5.2. Infrared-reflected (IRR) and visible-induced infrared luminescence (VIL) imaging
When equipped with a set of filters and a modified CCD camera, sensitive in the IR range, the Goren-Pol microscope can capture images in the IR range. A Nikon Coolpix 995, following the removal of the inbuilt UV&IR-blocking filter, and equipped with an IR cut-on filter (Schott RG715 or RG830), was used to capture images in the IR range. The function of this filter is to allow IR radiation to reach the sensor and to block any visible light.

For IR-reflected imaging, a 5W IR torch, centred at 850 nm, was used as an external radiation source. For visible-induced luminescence imaging, a visible LED torch, equipped with an excitation IR-
blocking filter Schott BG38, was used as a source of visible light. The visible light is absorbed by luminescent compounds and re-emitted in the IR range.

Figure 8 shows the IRR and VIL images of a cross-section of a sample from a 4th Century BCE temple in Cumae, Naples, Italy, and consists of a calcium carbonate ground and a paint layer containing particles of Egyptian blue. This pigment is known to emit IR radiation when excited in the visible range [27][28]. Focusing, for both reflected and luminescence images, was achieved using the 5W IR torch.

Figure 8. Visible (a), IRR (b), and VIL (c) images of a cross-section containing Egyptian blue.

6. Conclusion
Preparation and examination of cross-sections, thin-sections and dispersions for the conservation of wall paintings is more often carried out off-site, in a laboratory with state-of-the-art, but expensive equipment. However, this discipline would greatly benefit from the possibility of carrying out this type of examination in situ, using portable, low-cost and versatile equipment that may be tailored for specific needs. The present paper illustrates the possibility of achieving this by modifying standard preparation procedures to suit the examiner’s needs, designing custom-made equipment and by using a suitable portable microscope, such as the recently developed Goren-Pol. Comparatively to a state-of-the-art Leica DMRD, the results obtained with the Goren-Pol are excellent, bearing in mind its compact size and much lower cost. The set-up could easily and inexpensively be altered, to examine samples in incident light, UV and IR radiation.

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References
[1] McCrone W C 1994 Journal of the American Institute of Conservation 33 (2) 11, 14
[2] McCrone W C 1982 Journal of the International Institute for Conservation - Canadian Group 7 (1-2) 11-4
[3] Cather S 2004 2nd Int. Conf. on the Conservation of Grotto Sites (Dunhuang) ed N Agnew (Los Angeles: The Getty Conservation Institute) pp 22-32
[4] Pique’ F and Verri G 2015 Project Report: Organic Materials in Wall Paintings (Los Angeles: The Getty Conservation Institute) p 13
[5] Cather S 2004 Proc. Int. Sem. Diagnosis, Conservation and Restoration of Far Asian Mural Paintings (Ravenna) ed R Mazzeo pp 28-29
[6] Hunt P N and Griffiths D R 1989 World Archaeology Ceramic Technology 21 (1) 165-72
[7] Plesters J 1955-6 Studies in Conservation 2 110-13
[8] Silva C L 2006 ‘A Technical Study of the Mural Paintings of the Interior Dome of the Capilla de la Virgen del Rosario, Iglesia San José, San Juan, Puerto Rico’, Theses (Historic Preservation) pp 16, 40
[9] Messinger J M 1992 Journal of the American Institute of Conservation 31 (3) 267
[10] Magrini D, Bracci S and Sandu I C A 2013 Procedia Chem. 8 194
[11] Gribble C and Hall A 1995 Optical Mineralogy: Principles and Practice (London: UCL Press) p 32
[12] Reedy C L 1994 Journal of the American Institute of Conservation 33 (2) 115
[13] Sandu I C, Schafer S, Magrini D, Bracci S and Roque C A 2012 Microsc. Microanal. 18 (4) 862-3
[14] Goren Y 2014 New York Microscopical Society Newsletter pp 2, 3, 7, 10
[15] Zizola C 2005 La conservazione e il restauro delle pitture murali della Madrasa Amiriya, Radà, Yemen Centro di Conservazione Archeologica (Roma) 2
[16] Griffin I 1997 ‘Pozzolanas as additives for grouts: an investigation of their use in wall painting conservation’ Unpublished dissertation (London: The Courtauld Institute of Art) p 37
[17] Santopadre P and Verita’ M 2006 Studies in Conservation 51 (1) 32
[18] Gaetani M C, Santamaria U and Seccaroni C 2004 Studies in Conservation 49 (1) 16
[19] Berrie B H, Leona M and McLaughlin R 2016 Heritage Science 4 (1) 8
[20] Yong L and Shiwei W 2014 Studies in Conservation 59 (5) 315
[21] Stoner J H and Rushfield R 2013 Conservation of Easel Paintings (Oxford: Taylor & Francis) p 315
[22] Bousfield B 1992 Surface preparation and microscopy of materials (Chichester: Wiley) pp 21, 167
[23] Pouyet E, Lluveras-Tenorio A, Nevin A, Saviello D, Sette F and Cotte M 2014 Anal. Chim. Acta 822 53
[24] Derrick M, Souza L, Kieslich T, Florsheim H and Stulik D 1994 Journal of the American Institute of Conservation 33 (3) 228
[25] Sabatini G, Giamello M, Pini R, Siano S and Salimbeni R 2000 J. Cult. Heri. 1 S13
[26] Tamburini D, Martin de Fonjaudran C, Verri G, Accorsi G, Accocella A, Zerbetto F, Rava A, Whittaker S, Saunders D and Cather S 2018 Microchem. J. 137 242
[27] Verri G 2009 Anal. Bioanal. Chem. 394 1011
[28] Aramini F, Sidoti G and Santopadre P 2013 Bollettino ICR 27 20