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Combination of a Solvent-dispensing and Micro-aspiration Device for Removal of Varnishes on Painted Surfaces

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ABSTRACT

The use of organic solvents in treatments for the removal of varnishes on easel paintings is still a common practice among conservators worldwide. The more polar solvents required for aged, oxidized varnishes may promote leaching of fatty acid components of oil-based paints. The strategy tested in this study to reduce the diffusion of the solvents, in an attempt to minimize interactions, relied on the use of a system composed of two devices: a pump for delivering a precisely controlled amount of solvent to the surface, and a solvent-proof membrane pump to quickly aspirate it. This method was compared with two traditional methods of solvent application, to remove the varnish on two oil paintings on canvas, one from the twentieth century and the other dated 1872: swab-rolling and application through a Japanese paper sheet. The extent of leaching was measured by GC–MS analysis of the paint samples taken from the treated areas. The profiles of the fatty acids before and after varnish removal could thus be compared.

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Easel paintings; varnish removal; organic solvents; controlled-dispensing; micro-aspiration

Introduction

One of the authors previously reported on an innovative system composed of a syringe-pump and a surgical micro-aspirator for wet cleaning of sensitive painted surfaces (Cremonesi 2018). It basically represents a 'safer' way, as compared to more traditional swab-based techniques, to surface clean water-based contemporary paints, as well as any other artworks in which either the support or the paint and priming layers may be affected by contact with water. This successful strategy relies on the combined action of the two units: the pump delivers a controlled amount of solution to the cleaning brush acting on the surface, and the aspirator immediately removes it, thus preventing flow under the surface. Water-sensitive tempera paints could be cleaned, and the treatment resulted in lower interaction in terms of swelling and paint loss, as compared to traditional swab-rolling. Furthermore, analytical evidence was collected in the surface cleaning of a contemporary unvarnished oil painting, demonstrating that the system caused less interaction in terms of leaching of the paint layer, in comparison with traditional swab rolling (Casoli et al. 2020). A different device for dispensing the aqueous solution was also used, an air powered manual dispenser with a digital mechanism (Cremonesi and Héritier 2017; Casoli et al. 2020). Various cleaning treatments of nineteenth and twentieth century oil and acrylic

paintings were performed, with aqueous solutions in free and gelled form. Even agar and agarose gels, while in the semi-solid state, could be applied and aspirated with these devices. Analytical evidence was also collected demonstrating lower mechanical impact on fragile painted surfaces, as compared to traditional swab-rolling (Héritier 2020).

A further development of the system was necessary to enable it to work with organic solvents: its components had to be changed. As for the liquid delivery unit, the syringe driver could no longer be used: the pump would frequently stop, even when fitted with all-plastic syringes, devoid of the rubber gasket on the plunger; furthermore, leaks of solvent could pose a safety risk, considering that the pump is not solvent-proof. The latter issue was also crucial for the aspiration system; the previously used surgical micro-aspirators had to be replaced by other instruments, being only compatible with aqueous fluids, and not solvent-proof.

Aim of this work

In preliminary testing of the controlled-delivery and micro-aspiration system with organic solvents, to perform removal of organic film-forming materials like varnishes and coatings, only visual evaluation of the results was made, and the conditions of the

treated surface were only assessed at a microscopic level (loss of paint, blooming, *craquelure*). It was clearly evident that this way of delivering solvents to the surface was effective, and it was reasonable to assume that lesser interaction with the paint media could be obtained, in terms of swelling and leaching. It is indeed well established that shorter contact time of solvents can reduce the interaction with oil- or alkyd-based paint media (Sutherland and Shibayama 1999; Sutherland 2001; Fuesers and Zumbühl 2008). The present study was designed to verify this assumption at a more precise analytical level – whether the quick removal of the solvent from the surface, and the consequent decrease in diffusion under the surface, could lead to a lesser extent of leaching out components from the paint medium, in comparison with more traditional means of applying organic solvents to remove varnishes. To address this, the binding medium of two oil paintings was first characterized by gas chromatography coupled with mass spectrometry (GC–MS) and the varnish by infrared spectroscopy (FTIR). The varnish was then removed in three selected areas, using the same solvent with the controlled-delivery and micro-aspiration system, and in two other more traditional means. Paint samples from the three tested areas were then analyzed by GC–MS. The profiles of the fatty acids before and after varnish removal could thus be compared, to assess the extent of interaction.

Two artworks were chosen for the experimentation: an oil painting on canvas, dating from the first half of the twentieth century, with a strongly discolored varnish layer, and an oil painting on canvas by Uranie Alphonsine Colin-Libour dated 1872, with a thick yellowed varnish.

Experimental procedure

Samples for GC–MS analysis

From the original varnished surface

Samples 1 and 2 were taken from two different color areas of the twentieth century painting, one adjacent to the area where the varnish removal tests would then be made and the other opposite to that. Samples 3 and 4 were taken from two different color areas of the 1872 painting, each adjacent to the area where the varnish removal tests would then be made; precisely, sample 3 was taken from the brown background and sample 4 from the blue dress of the female figure. 0.6 mg of each sample was then used for GC–MS analysis to characterize the binding medium.

From the paint layer after varnish removal

For both paintings, letters indicate the three designated solvent-application techniques:

Sample A (*Aspiration*), after solvent applied with controlled-delivery and micro-aspiration system;

Sample P (*Paper*), after solvent applied on Japanese paper;

Sample S (*Swab*), after solvent applied through cotton swabs.

These samples were analyzed by GC–MS to determine their relative content of lipid material.

0.6 mg of each sample was used for GC–MS analysis to monitor the leached binding medium.

From the Japanese paper after the solvent treatment

An additional sample (L, *Leached*) was taken from the Japanese paper used in one of the treatments on the twentieth century painting, and analyzed by GC–MS.

Samples for FTIR analysis

A sample of both varnishes was scraped from the original varnished surfaces and analyzed by FTIR to identify the nature of the applied varnish.

Samples for cross-section analysis

Only the twentieth century painting was sampled for this analysis. A micro-fragment was sampled from the original surface, in an area adjacent to that of the subsequent solvent treatments.

Instruments and methods

Gas chromatography–mass spectrometry (GC–MS)

GC–MS analysis was carried out on an Agilent Technologies 7820A gas chromatograph (Agilent Technologies, Palo Alto, CA) coupled to an Agilent Technologies 5977B mass spectrometer. The carrier gas was helium. The injector was kept at 280°C. The injection mode was split (20:1). MS conditions were as follows: interface temperature, 280°C; ion source temperature, ca. 190°C; electron impact mode, 70 eV, scan mode in the range of m/z 40–400. A low-polarity capillary column SLB-5 ms (Supelco, 5% phenyl groups, 30 × 0.25 mm, f.t. 0.25 μm,) was used.

The sample was treated with 4N-HCl in methanol (0.4 ml) and n-hexane (1 ml) for 1 h at 70°C, with addition of 10 μl 2.5 mM tetracosane (internal standard). The n-hexane phase, which contains fatty acid methyl-esters, was used for gas chromatographic analysis (1 μl). Separation of the methyl ester of fatty acids was done following this temperature program: isothermal at 60°C for 2 min, with 20°C/min heating up to 220°C and isothermal at 220°C for 8 min, with 20°C/min heating up to 280°C and final isothermal conditions at 280 °C for 4 min.

Qualitative analysis was performed to identify lipids contained in the painting, and the average amounts of fatty acids relating to the internal standards were considered.

Cross-section analysis

One micro-sample was collected to prepare a cross-section (Plesters 1956) by embedding it in epoxy resin (EpoFix™ Struers) and dry polished with silica abrasive papers (grit p120 to p4000). Cross-section analysis was performed using visible and ultraviolet (UV) light microscopy, followed by scanning electron microscopy (SEM) coupled with an electronic microprobe (EDX). A Nikon TK-1270E optical microscope was used in reflected light. Dark field observations were performed using fixed oculars of 10x and objectives with different magnifications (5, 10, 20, and 40x). Photomicrographs were recorded using an Olympus DP70 digital scanner camera directly connected to the microscope. An environmental SEM (Philips Quanta FEI 200) equipped with an energy dispersive X-ray spectrometer (EDX) by Link Analytical Oxford (Link, UK), model 6103 was also used. Several analyses were performed on each layer of the cross-sections and the elemental analysis was carried out at 30 kV acceleration voltages, with a 100 s lifetime and 30 mm working distance.

FTIR spectroscopy analysis

FTIR spectroscopy measurements were performed using a Bruker Hyperion FTIR microscope equipped with a cryogenic mercury-cadmium telluride detector and a germanium (Ge) crystal. A few micrograms of sample were placed in a diamond cell and then placed into the instrument. The analysis was



Figure 2. Luer-lock brushes of different shape, size, and type of bristles.

performed in transmission mode, and spectra were collected from 400 to 4000 cm^{-1} with 64 scans of acquisition with a resolution of 4 cm^{-1} .

The dispensing/aspiration system for solvent application

The dispensing/aspiration system was composed of the following units, shown in Figures 1–3. A laboratory membrane pump [1 in Figure 1] was selected as the micro-aspiration device. This pump, certified to aspirate air contaminated by hydrocarbon vapors, operates oil-free thanks to its mechanics: a vibrating membrane that generates suction on the inlet port and



Figure 1. The micro-aspiration device: the membrane pump connected to the luer-lock brush, and the trap inserted within the suction tubing.

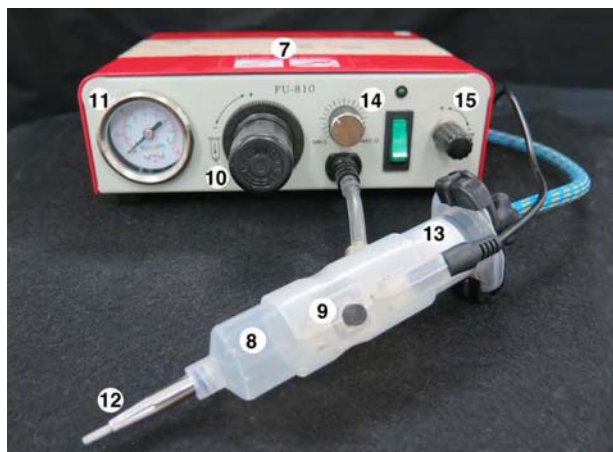


Figure 3. The electric/pneumatic solvent-delivery device connected to the syringe-tank fitted with a luer-lock brush and the finger control.

compressed air on the outlet port. It is virtually maintenance free. Membranes, or diaphragms, are available in different materials; poly-Tetrafluoroethylene (PTFE, Teflon™) was selected, because of its highest compatibility with organic solvents of different polarity, including hydrocarbons. The specific pump was the LABOPORT™ Mini Laboratory Pump, model N86KT.18 by KNF Neuberger GmbH (KNF 2018). This entry-level model in the series of pumps has the lowest delivery rate – 5.5 litre/min. However, the aspiration produced was sufficient to quickly remove from the surface a moderate amount of delivered solvent. In spite of the small size, the pump is highly priced if the PTFE membrane is selected. A more powerful pump model would be preferable for a larger amount of solvents – the LABOPORT™ model N840FT.18, with 34 l/min delivery rate (KNF 2019). A polypropylene Drexel bottle [2] inserted into the suction tubing acts as solvent-trap, to prevent the solvent in liquid form from reaching the membrane chamber; a luer-lock brush is inserted into the open end of the suction tubing to aspirate the solvent from the treated surface [3]. A length of soft silicone tubing [4] is also inserted around the ferrule of the brush, to prevent scratching the aspirated surface. The pump is extremely simple to operate, having only the on/off switch [5]. It works quietly; further reduction of the noise is achieved by inserting an additional silencer [6] in the outlet port.

Luer-lock brushes of different shape, size, and type of bristles are available (Figure 2). The brushes are also color-coded, according to the international classification for different gauge volumes.

An electric/pneumatic tool manufactured for precision application of fluids and pastes was selected as solvent-delivery device [7 in Figure 3] – a compressed air-powered manual dispenser with digital controls. Specifically, the model used was the Barrel Dispenser FU-810 by Adhesive Dispensing Ltd (Adhesive Dispensing 2019). A syringe-tank [8] connected to the

dispenser is the fluid reservoir. Impulses sent by a pedal or finger control [9] release a certain amount of pressure, precisely regulated by a doser [10] and monitored by a gauge [11], onto the piston of the syringe, which then dispenses controlled amounts of the fluid to the surface through a luer-lock brush [12]. Syringe-tanks are available in different volumes (3, 5, 10, 30, and 55 ml). A double wiping edge [13] on their polyethylene pistons guarantees a good seal without a rubber gasket. The amount of dispensed fluid is precisely regulated by digital control [14] of the duration of the impulse, between 0.1 and 9.9 seconds. The dispenser also provides an adjustable [15] anti-drop system, operating by Venturi effect, which prevents the fluid from flowing between impulses. Normally the dispenser is powered by an air compressor, and a fairly sophisticated on-line filtering system is then required to purify the compressed air from contaminant water, oil, and particles from the compressor. Alternatively, for ease of transport and operation, an inert gas cylinder may be used in place of the air compressor to power the dispenser; this was the actual hardware configuration used in our treatment, relying on an 5 kg argon cylinder as a source of pressure.

The Japanese paper, the cotton swabs, and the syringe-tanks were thoroughly rinsed by sonication in water, dried, sequentially washed with trichloroethane and acetone and dried, prior to use.

Results and discussion

As an opening remark, researchers often must make this choice when addressing this type of study: experiment with real artefacts, which allow only limited sampling, or simulated artefacts, which instead would allow extensive and comprehensive sampling. The first possibility, unlike the second, cannot produce statistically valid results in the sense of repeatability, but has the fundamental value of representing a ‘real case’ in terms of the complexity of the materials and their aging. Thus, although both are legitimate, the two possibilities are equally affected by limits.

In the work presented in this contribution, with a mainly applicative value, the authors privileged the second choice, well aware of its limits. A different form of validation is thus pursued – not so much via statistics, but rather applicability, with a comprehensive study currently underway on a series of ‘real cases.’ In other words, this corresponds to giving up a little scientific rigor in favor of closer relevance to the infinite variability of the artistic artefact.

Characterization of the paintings

Cross-section analysis of the sample from the twentieth century painting showed a rather simple structure

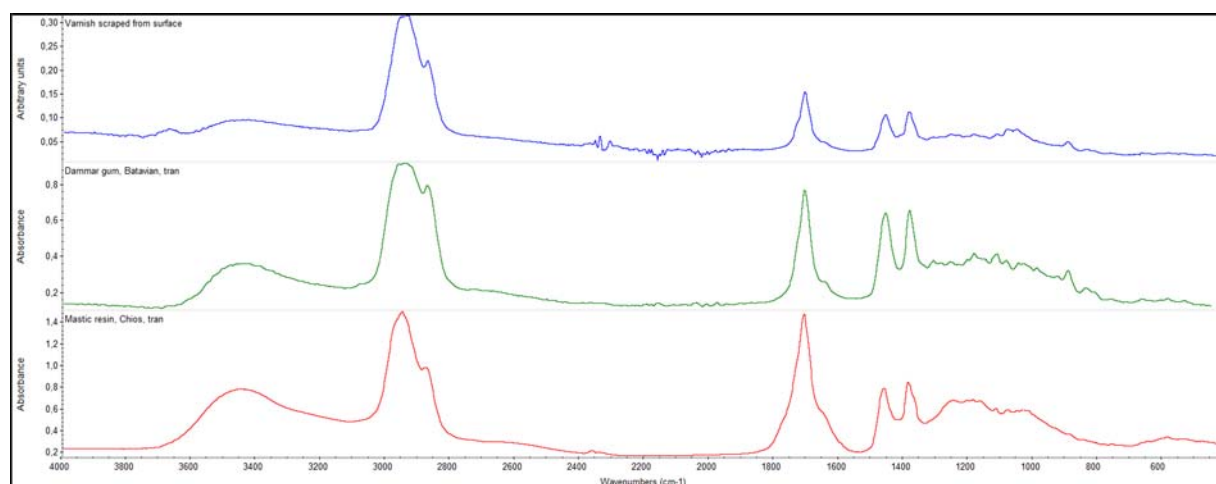


Figure 4. Characterization of the varnish of the twentieth century painting. FTIR spectra of (top to bottom): the sample from the painting; a reference spectrum of dammar; a reference spectrum of mastic.

for the painting – the canvas, a layer of protein-base material, a thin light color film, and a varnish layer. Optical microscopy under UV light and SEM observations showed no actual priming layer, but rather a proteinaceous coating, likely animal glue, applied to reduce the porosity of the canvas support. The pictorial layer is about 30 μm thick and presents several micro-cracks that suggest an initial loss of cohesion. From a chemical point of view, it is composed of a titanium dioxide white matrix in which many small, variously colored particles are dispersed. EDX microanalysis identified the following elements in these particles: animal carbon black (ivory or bone black), cobalt blue, barium sulphate and zinc white (maybe lithopone), dark natural earths rich in manganese oxide, and traces of chromium (maybe chromium oxide greens). Staining tests revealed the presence of significant quantities of fats and proteins.

A thin (<10 μm), UV fluorescent, varnish film is visible over the pictorial layer. FTIR spectroscopy analysis characterized this varnish as a terpenoid resin; dammar has the best matching spectrum (Figure 4).

Similarly, the varnish sampled from the 1872 painting, analyzed by FTIR spectroscopy, was characterized as terpenoid resin: dammar (85.06 match, ref. NR00234 IRUG database). No cross-section analysis was done for this painting.

Binding medium analysis

The fatty acids identified by GC–MS analysis of the paint samples from both paintings (samples 1, 2, 3, and 4) indicated the presence of a mixture of oils. The results obtained are shown in Table 1 for the twentieth century painting, and in Table 2 for the 1872 painting. The very low value of azelaic acid in the samples from the twentieth century painting may indicate the presence of a siccative oil in mixture with other vegetable oils, presumably one or more semi-

siccative oils. As for the 1872 painting, the results obtained indicate a probable mixture of siccative and semi-siccative oils.

Varnish removal tests

Although a volatile polar solvent like acetone could have been effective in removing the varnish, the less volatile 2-butanone (methyl ethyl ketone, MEK) was deliberately chosen. Its stronger swelling power toward oil media could make the difference more significant among the three selected methods of solvent application, in terms of interaction with the paint medium.

On selected color areas of the painting, with an even distribution of the varnish, the solvent was applied in the three selected ways, each for the same length of time (about 20 s on the twentieth century painting and only 10 s on the 1872 painting): swab rolling, wetting a Japanese paper sheet with a solvent-loaded cotton swab, and with controlled delivery/micro-aspiration. In the first two applications care was taken to pre-dry the solvent-loaded cotton swabs on absorbent paper, to avoid an excess of solvent.

Figure 5 illustrates the varnish removal tests on the 1872 painting. For each application, the areas to be treated were precisely defined by cut-out frames of identical size. Complete removal of the varnish was assessed on all areas by examination under UV light (Reskolux lamp, 365 nm).

After some hours, the treated areas were sampled for GC–MS analysis. The samples were then weighed, and 0.6 mg of each sample was used for GC–MS analysis. An internal standard was added to each sample for quantitative results: 10 μl 2.5 mM tetracosane, RT = 19.89 min.

The chromatographic profiles of the three samples 2S, 2P, and 2A from the twentieth century painting

Table 1. Fatty acid amounts in samples 1, 2, 2S, 2P, and 2A taken from the twentieth century painting, from GC–MS analysis, calculated in relation to tetracosane internal standard.

Fatty acid RT (min)	tR (min)	Sample 1 (µg)	Sample 2 (µg)	Sample 2S (µg)	Sample 2P (µg)	Sample 2A (µg)
Azelaic Acid	9.7	1.2	1.2	0.9	0.5	0.8
C10:0	10.2	0.4	0.4	0.4	0.3	0.4
C14:0	10.5	3.3	3.5	0.7	0.3	3.3
C15:1	10.8	0.4	0.5	0.4	0.3	0.6
C15:0	11.1	1.4	1.1	0.8	0.6	1.2
C16:1	11.7	0.7	0.7	0.4	0.5	0.6
C16:0	11.8	28.7	28.8	23.5	11.9	28.2
C17:0	12.8	1.3	1.3	1.0	0.7	1.2
C18:1	13.7	4.8	4.9	1.9	1.7	4.9
C18:0	14.1	27.4	27.6	21.0	11.5	27.3
C20:0	18.2	0.4	0.4	0.3	0.2	0.3
C22:0	20.7	0.5	0.4	0.4	0.2	0.5
Total (µg)		70.5	70.8	51.7	28.7	69.3

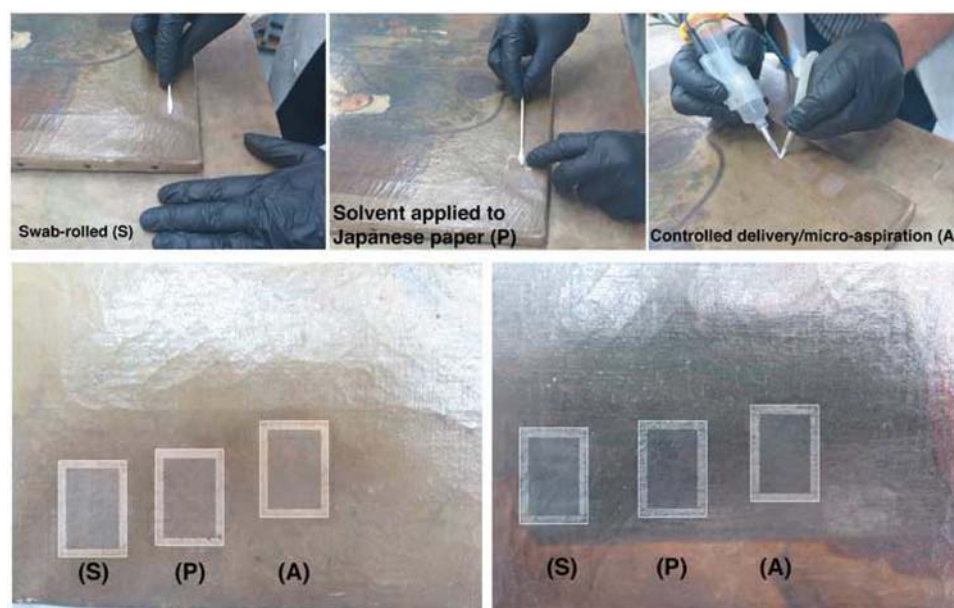
Table 2. Fatty acid amounts in samples 3, 3S, 3P, 3A, 4, 4S, 4P, 4A taken from the 1872 painting, from GC–MS analysis, calculated in relation to tetracosane internal standard.

Fatty acid	tR (min)	Sample 3 µg	Sample 3S µg	Sample 3P µg	Sample 3A µg	Sample 4 µg	Sample 4S µg	Sample 4P µg	Sample 4A µg
Azelaic Acid	9.7	10.2	4.2	3.6	5.4	8.4	2.4	1.8	6.0
C14:0	10.5	9.0	1.2	0.6	1.2	10.8	2.4	2.4	7.8
C15:0	11.1	4.8	–	–	–	4.2	0.6	0.6	3.0
C16:1	11.7	4.2	–	–	–	4.2	–	–	–
C16:0	11.8	35.4	14.4	11.4	35.4	36.0	9.0	9.0	33.6
C17:0	12.8	2.4	–	–	–	3.0	0.6	0.6	9.0
C18:1	13.7	3.6	2.4	5.4	10.8	13.2	3.0	3.0	7.2
C18:0	14.1	17.4	8.4	6.6	12.6	15.6	4.8	4.2	14.4
Total (µg)		87.0	30.6	27.6	65.4	95.4	22.8	21.6	81.0

show the same fatty acids identified in the original paint samples (samples 1 and 2). Their amounts are listed in Table 1, calculated in relation to the tetracosane internal standard.

As can be seen, sample A from the area treated with the controlled delivery/aspiration method contains the largest amount of fatty acids from the paint binder (69.3 µg). It can also be noted that the total amount of fatty acids detected in sample A has a value very close to that determined in the two samples of the

original, untreated paint material (70.5 and 70.8 µg). This finding shows that the micro-aspiration assisted procedure did not cause an appreciable leaching effect. Sample S follows, from the swab-rolled area (51.7 µg); and last, sample P from the area treated with the solvent applied onto Japanese paper (28.7 µg). These measured amounts are ‘residual’ amounts, after the leaching action exerted by the solvent used to remove the varnish. The higher amount in sample A thus indicates lower interaction

**Figure 5.** Varnish removal tests on the 1872 painting carried out according to the three selected methods.

on the paint media by the solvent, when applied onto the surface and quickly removed by micro-aspiration.

GC–MS analysis of sample L, taken from the same Japanese paper used in one of the paint removal tests, showed the signals corresponding to the same fatty acids identified in the samples from the paint, in similar proportions; this confirms the leaching effect exerted by the solvent applied under this method.

The results from GC–MS analysis of the treated areas of the 1872 painting substantially confirm the trend seen for the first painting (Table 2) – the paint areas treated with the solvent onto the Japanese paper (samples 3P and 4P) show a strikingly low residual amount of fatty acids, followed by the swab-treated areas (samples 3S and 4S). Conversely, the areas treated with the micro-aspiration assisted procedure (samples 3A and 4A) retain the largest amounts of fatty acids. Furthermore, in area 4 (the dress of the female figure) this residual amount is fairly close to the original amount, thus indicating the lowest leaching effect exerted by the powerful solvent when applied through this technique.

The care for the paintings prompted us to only sample one paint fragment from each treated area so as to contain our interaction. These results, although partial, still demonstrate that micro-aspiration is an effective means of decreasing the rate of diffusion of the solvent under the surface. This lower diffusion in turn ensures lower leaching of the oil-based paint medium.

There is a final consideration to note of practical interest, relating to the controlled application/micro-aspiration system. It has been used on several occasions, for thinning or removing varnishes on antique, modern, and contemporary easel paintings (Cremonesi and Héritier 2017). Working with this equipment is simple. The wide choice of brushes available allows one to find the most suitable selection for solvent application and removal both on smoother surfaces and on morphologically more complex areas, such as thick *impasto* and relief of the brushstroke. The operator always has the feeling of full control over the application, precisely because the conservator decides the amount of solvent to release; the useful anti-drop system prevents the uncontrolled leakage of solvent. Furthermore, the operator decides the contact time of the solvent with the surface, contacting it with the micro-aspiration brush. This must be periodically rinsed in a polar solvent, such as acetone, disconnecting the micro-aspiration, to prevent the accumulation of removed varnish on the bristles.

Both the solvent-delivery and aspiration instruments operate in safe conditions. The diaphragm aspiration pump is certified for the use of organic solvents and is explosion proof. As for the liquid dispenser, the solvents are in contact only with the syringe-



Figure 6. The use of the controlled application micro/ aspiration system on a painting in an upright position on the easel.

tank which is pneumatically controlled so there is no risk of fire. The tubes for the application and aspiration of solvents can be of appropriate length to work easily and without loss of performance even on a painting of relatively large dimensions, in either the horizontal or vertical position. Figure 6 shows the use of the instrumentation on a painting in an upright position on the easel.

Both devices are easy to operate and well controllable. Furthermore, both the solvent delivery unit and the aspiration pump can also manage gels of not excessive viscosity. The precision of the solvent delivery unit allows one to use much lower quantities of solvents than those required for swab application. This translates into a better quality and safety of the work environment, because exposure to solvent vapors is drastically reduced. During operation, fewer volatile solvents gradually accumulate in the trap, so that they can be properly disposed of at a later time. The more volatile ones may evaporate with the suction airflow. If it is considered prudent to use an aspirator for prolonged use or for very large amounts of solvents, the aspirator should be positioned near the membrane pump outlet valve.

Conclusions

In restoration treatments aimed to remove varnishes from easel paintings, organic solvents are likely the

most commonly used materials by conservators worldwide. The well documented risk of leaching out components from the paint media may be particularly severe for the more polar organic solvents that are generally required for aged, oxidized varnishes of natural and synthetic origin.

The results from this study substantiate our initial hypothesis: reducing diffusion of the solvent used for removing the varnish on an oil painting could effectively reduce the extent of leaching. In our expectations, a modified version of the controlled delivery of liquid and micro-aspiration systems devised for the aqueous medium could also effectively accomplish this. In comparison with two traditional means of solvent application, by cotton swab and through a sheet of Japanese paper, the system actually performed with the same efficacy, producing a lower degree of interaction with the oil-based paint media, in terms of leaching out the fatty acid constituents.

Particularly meaningful, for its practical relevance, is the finding that application of the solvents through a Japanese paper sheet produced the largest degree of leaching. This method is often used by conservators for removing varnishes, particularly from large painted surfaces.

Previous testing of the system in combination with aqueous medium for cleaning water-sensitive surfaces demonstrated the positive effect of micro-aspiration in reducing side interactions with the paint medium, like swelling and loss of paint. The current study substantially proves that the same benefits can also be obtained when working with solvents.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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