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## Solvent Vapour Use – The Unintended Consequences in Textile Conservation

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### ABSTRACT

The impact of solvent vapour from two solvents, acetone and industrial denatured alcohol (IDA), commonly used in adhesive reactivation in textile conservation, when applied to artificially soiled silk fabric through either Gore-Tex® or Reemay® membranes for exposure times of either 1 or 3 min, was measured using microscopy and ImageJ to monitor the movement of the solid particulates of the soiling, and Fourier transform infrared spectroscopy with attenuated total reflectance (FTIR-ATR) to monitor the movement of the oily components. Analysis using these techniques successfully showed the movement of large particulates through the textile and a decrease in the presence of oil. Analysis with FTIR-ATR showed that the application of solvent vapour by a poultice method produces more than just a surface change and the vapour can penetrate far enough into the fibres to cause a change in the level of oil through the entire textile. The results showed that the solvents and barrier membranes have characteristic differences which impact on the changes on the soiling. The application of acetone produced a greater change in the movement of oil on the front and back of the samples than the application of IDA, while a comparison between the barrier membranes showed a greater change occurring in the level of oil with the use of Gore-Tex® rather than Reemay®. The time of exposure to solvent vapour made little difference to the changes to the textile soiling. Quantitatively significant results were gathered from analysis of the changes in the oil measured with FTIR-ATR, and qualitative changes in the large particulate soiling, demonstrating the potential usefulness of ImageJ open access software in future historic textile soiling monitoring studies.

### ARTICLE HISTORY

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### KEYWORDS

Solvent vapour; adhesive reactivation; solvent cleaning; FTIR; microscopy; textile conservation; membranes; image analysis

### Introduction

Textiles may be exposed to solvent vapour during conservation treatments. One notable example of this is during adhesive support treatments. In the process of solvent re-activated adhesive treatments, a thin film of the chosen adhesive is applied to a support fabric and is allowed to dry. Once the support fabric is positioned on the area of the object to be supported, the adhesive film is re-activated. Blotter paper is dampened with the chosen solvent and applied to the object through a barrier membrane, applying slight pressure to ensure contact between the adhesive film and the object. Applying the solvent as a vapour through a barrier layer allows the conservator to have more control over the amount of solvent applied to the object. This can reduce the risk of staining caused by the solubilisation of ingrained soiling by liquid adhesive, and excessive softening of the film which can imbed the adhesive in the structure of the textile.

Solvent re-activation is generally used when a textile is too brittle to withstand the action of stitching, when it contains painted components, and when it proves to be technically too challenging to stitch into, for

example, a three-dimensional object. Solvents may also be used when a textile is considered to be too fragile for the amount of heat and pressure required to achieve a satisfactory bond with a heat-sealing treatment (Hillyer, Tinker, and Singer 1997). The research reported here was formed from questioning whether solvent vapour has unintended effects on textiles, specifically the movement of soiling within them.

With the understanding that an object's condition, material composition, and structure can result in the need for an adhesive support treatment, and the knowledge that the same factors can hinder the removal of ingrained soiling (Eastop and Brooks 1996; Greene 2006), a hypothesis can be made that historic textiles which are supported with an adhesive treatment may have some level of soiling retained in the fibres.

The aim of this research is to investigate whether the application of solvent vapour to a textile when used in adhesive re-activation mimics the action of liquid solvent and produces an unintended change in the soiling found in textiles.

An experimental trial was designed to investigate and measure the effects of the solvent vapour on textile

soiling. The trial aimed to mimic the action of adhesive re-activation with solvent vapour, however omitting the adhesive film. This was done as an initial study aiming to understand the behaviour of solvent vapour through membranes. This information is crucial to understand the effects on the mechanisms of the various materials. It was decided that the inclusion of adhesive at this stage could hinder the data gained from the analysis. To answer the research question the following variables were included in the experimental design:

- Investigate the potential effect that solvent vapour has on ingrained soiling in textiles using two solvents, acetone and industrial denatured alcohol (IDA), commonly used for adhesive re-activation
- Determine if there is a difference in the effect of the solvent vapour if different types of barrier layers are used by testing Gore-Tex® and Reemay®
- Determine if the effect of the solvent vapour on the soiling changes with varying lengths of exposure, using 1 and 3 min exposures times

## Materials and methods

Fabric was artificially soiled with a combination of oil and organic and inorganic particulate matter. Solvent vapour was applied to the samples replicating the process of adhesive re-activation.

The samples were analysed using light microscopy before and after the application of solvent at two different magnifications, at which point they were photographed and the images evaluated with the scientific image analysis software programme ImageJ (2016) to visually and quantitatively measure the visible particulate soiling. Analysis of the change in the oil in the soiling mixture was achieved with Fourier transform infrared spectroscopy with attenuated total reflectance (FTIR-ATR). A series of ratios using the absorbance values of the changing carbonyl peak associated with oil and a peak of consistent intensity were calculated to monitor the changes that occurred between the test groups before and after the application of solvent vapour (Garside and Wyeth 2003).

### Silk

Silk was used as the substrate textile; samples measuring 50 × 50 mm were cut from plain woven medium weight silk habotai (Whaleys Bradford batch 015263/108) with a thread count of 50 ends per 10 mm in both warp and weft directions. Silk was chosen as it is a material that is often found in a variety of historic textiles. The medium weight habotai has a slightly more open weave structure and was considered beneficial to the experimentation as the applied soiling mixture would adhere well to the fabric, being able to sit within the

interstices of the weave structure, making any movement of the soiling on the silk easily perceivable under magnification. The samples of silk were cut from the main piece of fabric in a diagonal line so that no two samples shared the same warp or weft. This was done to avoid any bias in the results due to abnormalities or flaws in a single thread (Saville 1999).

### Soiling composition

Soiling found on historic objects is a combination of heterogeneous dust, comprised of fragments of skin, textile fibres, oils, grease, and inorganic salts, and fine particulate matter in the form of dark disfiguring soiling from a number of different sources that is usually grouped together under the heading 'soot' (Grau-Bové and Strlič 2013). Based on this knowledge a mixture comprising organic and inorganic matter was created to artificially soil the textile samples to be tested.

The soiling mixture comprised:

- A Midelney subsoil (a soil type), comprising of 53% clay and a 0.41% iron oxide content (Bakhsh 1988). This particular soil was chosen because of its high inorganic content and low iron content. Low iron content was chosen to avoid adverse staining.
- Soot, which was formed of a mixture from both wood and coal burning fires. Soot was chosen as a component for the soiling mixture as it provided carbon content and a slightly greasy texture.
- Dust, a combination of the contents from four museum vacuums located at the Centre for Textile Conservation (CTC) and two household vacuums. Dust was chosen for a complex composition of organic and inorganic compounds and large particle size. The dust was sieved through a coarse net to remove large clumps of hair and fibre.
- Olive oil (Filippo Berio Extra Virgin Olive Oil®). Oil was used to provide a greasy texture and aid in the adherence of the particulate soiling to the silk samples. The long molecular chains that make up the backbone of the oil molecule provide a greater surface area than the particulate soiling, which results in a strong bond to a textile surface (Tímár-Balázsy and Eastop 1998).

A set of preparatory tests were carried out to determine the most useful soiling mixture. Various ratios of the soiling components were applied to seven silk samples which were humidified at three different relative humidity (RH) levels, and analysed with FTIR-ATR and microscopy. From these preliminary tests the ratio of the soiling components and amount of the soiling mixture used to artificially soil the samples, the method of application of the mixture to the samples, the humidity level, and techniques of analysis

with microscopy and FTIR-ATR were finalised. The final soiling mixture comprised of 30% soil, 30% soot, 20% dust, and 20% oil.

### **Solvents**

The organic solvents used were acetone (Sigma-Aldrich) and IDA (Fisher Scientific). These were chosen as the solvents as they are commonly used in solvent cleaning and also adhesive re-activation in textile conservation practice.

### **Barrier layer membranes**

Two different materials were used as a barrier layer between the soiled silk samples and the solvent dampened blotting paper, Gore-Tex® and Reemay®.

Gore-Tex® (Conservation by Design) is a semi-permeable membrane made of two different layers: a hydrophobic layer of poly tetrafluoroethylene (PTFE), and polyester felt. The PTFE layer forms the membrane quality, and has pores of differing geometries that can range from 500 nm to 8 µm in diameter, permeable to vapour but not to liquid (Tarsiche, Hopîrtean, and Ciurchea 1997; Smith, Kerr, and Cowling 2007). Gore-Tex® was chosen for this experiment as it is commonly used in the re-activation of adhesives in conservation practice.

Reemay® is a randomly spun-bonded polyester fabric (Preservation Equipment P492-2014, 34 gsm). Reemay® is used much more infrequently than Gore-Tex® in adhesive re-activation. It is most often used as a material for packing objects and creating protective covers (Kerr, Capjack, and Fedosejevs 2000), is used in interventive conservation treatments as a support aid for delicate objects during wet cleaning treatments (Mathisen 1995) and as a backing material in leather conservation (Kite, Thompson, and Angus 2006).

### **Application of soiling mixture**

Eighty samples of silk were artificially soiled in the composite mixture; each sample was soiled individually to ensure that the same amount of mixture was applied to each surface. A total of 40 g of the mixture was used to soil the samples. The four soiling components were combined in a beaker; a stainless steel spoonula spatula was used to place the mixture on the centre of each sample, which was mechanically rubbed into the front only. This was done so that any movement of the soiling between the front and back of the sample would likely be easier to detect.

The soiled samples were humidified in a chamber made from a plastic tray covered with Melinex, a polyester sheeting (Preservation Equipment Ltd, 125 micron), and sealed with brown packing tape. The samples were humidified to ensure that the soiling

mixture became cemented to the threads and thus represented historical soiling affectively. The effect of humidity on soiled textiles has been well documented in the published literature on the monitoring of dust in museums and historic houses. These studies have shown that an environment of high relative humidity (RH) and temperature fluctuations can greatly affect the level to which dust and dirt can adhere to fibres, with particulate soiling becoming cemented to a surface after less than a day with an RH over 80% (Kumar, Dave, and Srivastava 1984; Lithgow et al. 2005; Brimblecombe, Thickett, and Yoon 2009). The humidity within the chamber containing the silk samples was raised to 70% RH with an ultrasonic humidifier over the course of three hours. Cotton wool dampened with deionised water was used to maintain the humidity level. The samples were humidified in the chamber for a total of three days. After the first day it was found that the humidity in the chamber rose to 80% RH, at which point some of the dampened cotton was removed. The humidity level then naturally lowered back to 70%, and remained at this level  $\pm 5\%$  over the remaining two days of humidification. The temperature of the chamber was recorded as  $22^{\circ}\text{C} \pm 3^{\circ}\text{C}$ ; the environment within the chamber was monitored with a Hanwell ML4106 data logger (IMC Group 2017). The samples were removed from the chamber, brushed with a soft-haired brush and vacuumed with a museum vacuum on low suction. This was done to remove any loosely adhered particulate soiling, replicating the process of the initial cleaning undertaken in a conservation treatment. The samples were numbered with a 6B graphite pencil in the bottom right corner to ease identification.

The eighty samples were randomly separated into eight groups using a random number generator (Math Goodies 2017). Each group contained ten replicates; this allowed for statistically viable results to be collected taking into account the possibility for experimental anomalies. Each group was assigned a barrier layer, time length of application of the solvent vapour, and a solvent. The test groups were labelled in this manner, where Gore-Tex® is represented by (G), Reemay® by (R), acetone by (A), and industrial denatured alcohol by (IDA). The groups were labelled as: G 1 min A; G 3 min A; G 1 min IDA; G 3 min IDA; R 1 min A; R 3 min A; R 1 min IDA; and R 3 min IDA.

### **Application of solvent vapour through barrier layers**

Each sample was placed soiled side up onto a piece of silicone release paper, which was placed on top of a thick piece of polyester felt, done to replicate the process of the re-activation of an adhesive film on a flat textile. However, an adhesive film was not applied to the soiled samples for the experimentation.

This was done as the research question aimed to investigate the effect of solvent vapour on soiling, and it was thought the presence of an adhesive layer could obstruct the final visual and FTIR-ATR analysis.

A piece of blotting paper cut slightly larger than the size of the silk sample was dampened in either acetone or IDA using a solvent dispenser. Excess solvent was allowed to evaporate off the blotting paper until the paper no longer appeared wet, but was still cool to the touch. The dampened blotting paper was then placed over the selected membrane, and was weighted with a glass weight, which measured  $155 \times 95 \times 5$  mm, and weighed 219 g.

The solvent dampened blotting paper was held for two-time intervals, 1 and 3 min, as these time intervals for the application of solvent vapour in an adhesive treatment can produce satisfactory bonds between object and support. The recording of accurate lengths of exposure to solvent vapour also gives a clear picture of the materials' behaviour over specific time periods.

After the allotted exposure time to the solvent vapour, the dampened blotting paper was removed, and the sample and membrane were left under the glass weight for another four minutes. This was done to mimic the process of adhesive re-activation, as it is common to weight the sample after exposure to the solvent vapour to ensure that the support is strongly adhered to the object (Karsten and Down 2005).

## Instrumentation

### Microscopy

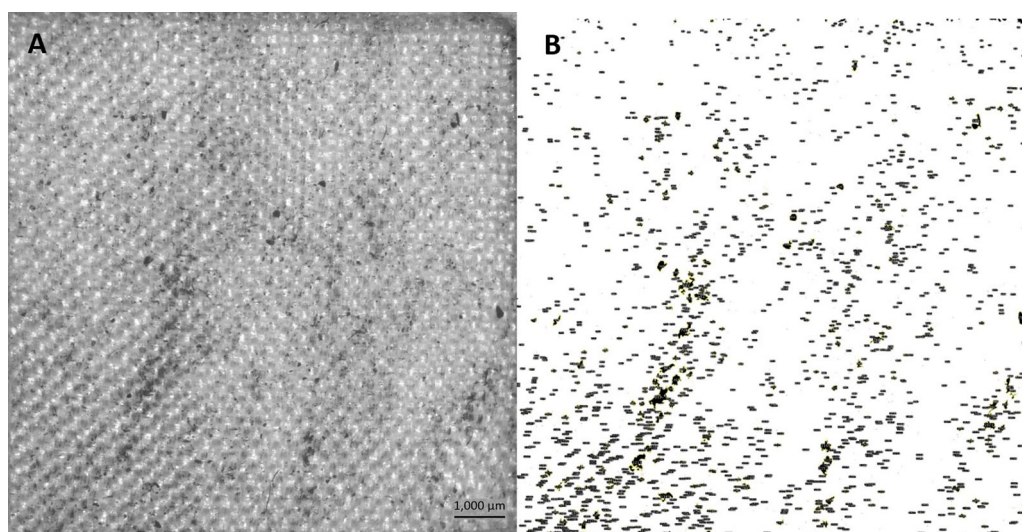
The samples were photographed under magnification using a Zeiss Stemi 2000-C stereomicroscope and Zen software, at  $4\times$  magnification and  $20\times$  magnification for both the front and back. This type of photography

allows for visual comparison and confirmation of any movement or change in the level of soiling after the application of solvent vapour. A card template was used to frame a section of the soiled fabric for the photography, with a window measuring  $10 \times 10$  mm. The positioning of the template was marked on each sample with a graphite pencil to ensure the template was placed in the same location for the photography after the application of solvent vapour, done so that the comparison of the before and after photography of the samples could accurately be assessed for any changes of the soiling occurring as a result of the experimentation.

### Particulate counting using Image J

ImageJ is an open-access image processing software that was designed by the National Institutes of Health for precise biomedical and scientific image analysis (Schneider, Rasband, and Eliciri 2012). The 'particle analysis' function automatically counts the portions of an image that have been segmented from the background (ImageJ 2017). Although used mainly in medical and scientific industries, the software was considered useful for this research as similar particle counting programmes have been used in dust monitoring in the heritage sector (Knight 2002). The 'particle analysis' function was used for the analysis of the microscopy images of the particulate soiling visible on the front and back of the samples, both before and after the application of the solvent vapour, with the aim of determining any changes in the amount of particulate after treatment.

The images of the samples were converted to black and white, and the threshold parameters of the imaging software were set to only analyse the darkest shades of black and grey, meaning the visible particulates (see Figure 1). A scale was set to measure



**Figure 1.** Black and white image of a sample (A) and the ImageJ analysis (B) with the set threshold parameters, where the dark areas are the particulate soiling that measure more than  $80 \mu\text{m}^{-2}$ .

171 pixels/1000  $\mu\text{m}$ , and particles measuring less than  $80 \mu\text{m}^{-2}$  were filtered out as these were mostly introduced by the thresholding procedure.

### **Fourier transform infrared spectroscopy (FTIR-ATR)**

Analysis was carried out using a Perkin Elmer Spectrum One FTIR Spectrometer with a Universal Sampling Attenuated Total Reflectance accessory, a diamond/thallium bromiodide (C/KRS-5) ATR crystal with a penetration depth of up to  $2 \mu\text{m}$ , and Spectrum software version 5.0.1. The spectra were collected over the region of  $4000\text{--}400 \text{ cm}^{-1}$ , at a resolution of  $8 \text{ cm}^{-1}$  and an average of 16 scans used. The samples were held on the ATR using a clamping force of approximately 50 N. The spectra were analysed using Bio-Rad Laboratories KnowItAll® software, Windows version 10.0.15063. Each spectrum was viewed as absorbance mode and baseline corrected using the linear automatic correction within the software.

The ten replicate samples from each of the eight experimental conditions were analysed 10 mm from the bottom left corner along the bottom edge and were analysed at the same location on the front and back so that any movement of the soiling through the fabric could be detected. The materials used to create the artificial soiling were analysed and the spectra were used to categorise the peaks of the spectra of the soiled silk samples. In order to determine the change in the presence of oil on the soiled samples after each solvent vapour treatment the intensity ( $I$ ) as peak height of the characteristic carbonyl ( $\text{C}=\text{O}$ ) present in the oil at  $1744\text{--}46 \text{ cm}^{-1}$  was compared against the characteristic amide I silk peak at  $1618 \text{ cm}^{-1}$  which is due to the carbonyl stretch and nitrogen–hydrogen ( $\text{N}\text{--}\text{H}$ ) of the peptide group. The intensity of the silk peak was used in the ratio calculations as it remained consistent throughout the experiment and was not affected by other material components within the sample. Comparison of FTIR spectra of unsoiled silk with soiled silk showed that the soiling did not affect the Amide I peak.

The absorbance values were measured from the baseline corrected spectra, and the ratios calculated for comparison of the front and back spectra for each test group. The peak height ratios  $I_{1744-46}/I_{1618}$  of each of the ten replicates in each of the eight conditions was carried out before and after solvent treatment. Application of the absorbance ratios facilitated the compilation of quantitative data allowing for a direct comparison of the results produced by the experimental variables.

### **Statistical analysis**

Statistical analysis was carried out using Minitab 17. Paired  $t$ -tests were carried out to determine if there

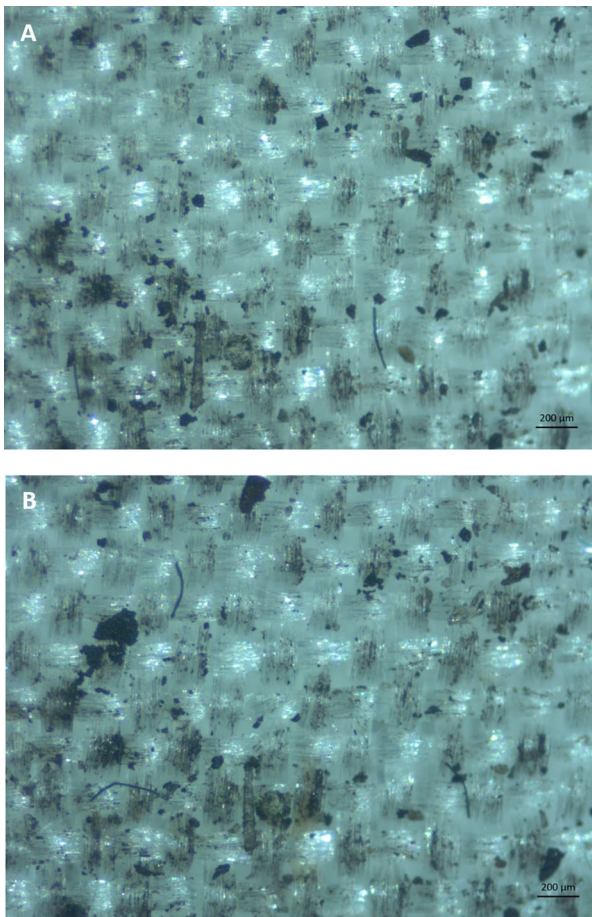
were significant differences between the samples before and after treatment. To determine the probability of a result being significant,  $p < .05$ ,  $.01$ , and  $.001$  are used, meaning significant, very significant, and highly significant, respectively, or put another way, 1 in 20 chance of being wrong (95% confidence), 1 in 100 chance of being wrong (99% confidence), and 1 in a 1000 chance of being wrong (99.9% confidence).

## **Results and discussion**

### **Microscopy**

Movement of large particulate soiling was observed during the application of the solvent vapour, as the barrier layer and silicone release paper had noticeable amounts of dark particulate soiling on both surfaces after the removal of the solvent dampened blotting paper. Movement of particulates measuring approximately  $50\text{--}200 \mu\text{m}$  was observed during visual analysis of the microscopy images. A small number of the images at  $20\times$  magnification have been considered anomalies due to the fact that the card template was not well aligned for the photography after the application of solvent vapour. As a result the before and after images are not exact representations of each other and so an accurate visual comparison could not be made; a result exacerbated by the high magnification and resulting small visible area of the sample. From the findings future research would benefit from the use of a larger window on the card template, which would have accounted for more variability between the samples and would have provided more useful results. However, these anomalies make up very few samples in each group of ten replicates, and so have not been considered to be significantly influential to the outcome of the results, as the movement was clearly visible on the images taken at  $4\times$  magnification, and the majority of the images at  $20\times$  magnification. An example of the visible change of particulate soiling at  $20\times$  magnification can be seen in [Figure 2 \(A,B\)](#), the front of one replicate from the test group R 1 min A, shown before and after the application of acetone vapour.

As the observations of the changes to the soiling made by visual analysis were largely subjective, ImageJ 'particle analysis' software was used to generate quantitative data on the changes occurring after the application of solvent vapour. ImageJ calculated data for the size of the area of silk analysed, the number of particulates, the average size of the particulates, and the percentage of the silk covered. The count of particulates and the percentage of the silk covered were considered the most relevant data to be analysed. The software recorded changes of particulate matter ranging in size from  $80 \mu\text{m}^{-2}$  to the largest particulates, approximately  $200 \mu\text{m}$ .



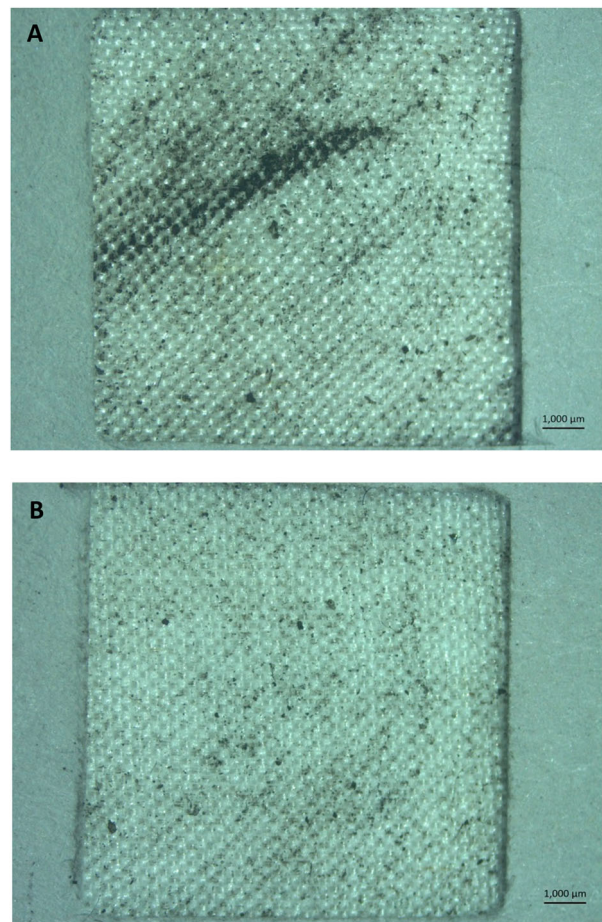
**Figure 2.** (A) R 1 min A before solvent application; (B) R 1 min A after solvent application.

The images at 4× magnification were analysed as they gave a better indication of the percentage of the surface of the silk fabric that was covered with the particulate soiling. The images at 20× magnification were not evenly in focus and therefore were problematic to process and get useable results with ImageJ.

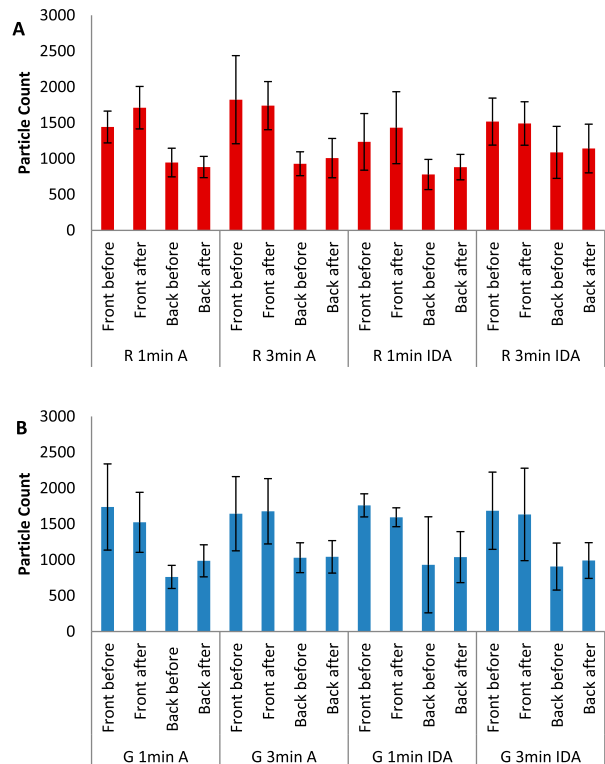
Although each of the replicates received the same amount of soiling, the mixture was unavoidably applied unevenly, which combined with the small 10×10 mm window used to photograph each sample, resulted in a large variance in the particle count between the replicates, subsequently resulting in a large variance in the data.

An example of the uneven application of the soiling mixture on two replicates can be seen in Figure 3(A,B) from the group R 1 min A, which were analysed with ImageJ and had initial average particle counts of 1750 and 1474, respectively.

The mean value of the particulate count of each replicate from each test group was calculated. The averages of the particle count for the front and back of the test groups before and after the application of solvent vapour through the two barrier layers are shown in Figure 4(A,B). A 2 sample *t*-test was carried out to determine if two groups were significantly different, 95% confidence. There was no significant



**Figure 3.** (A) Example of a replicate with an uneven application of the soiling material; (B) example of a replicate with even soiling. This example is indicative of the majority of the silk sample replicates.



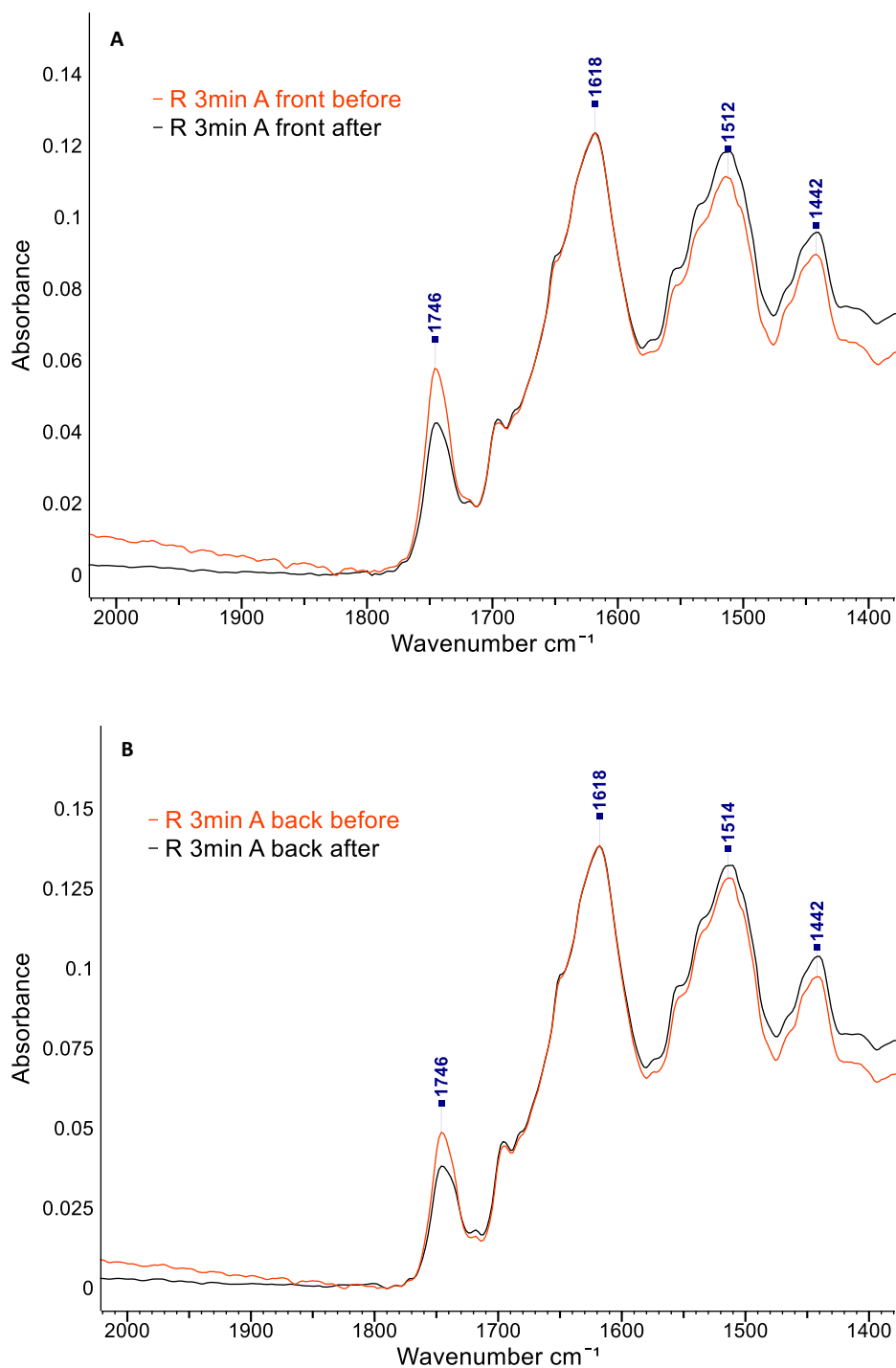
**Figure 4.** (A) Particle count for the samples with the Reemay® barrier layer; (B) particle count for the samples with Gore-Tex® barrier layer.

difference between the before and after treatment for the Gore-Tex® or Reemay® in the majority of the cases, with the exception of the back of the Gore-Tex® samples which were treated for 1 min with acetone, the front of the Gore-Tex® samples treated for 1 min with IDA, and the front of the Reemay® samples treated for 1 min with acetone. It is unlikely that the groups that demonstrated a significant difference did so for any reason other than the variance in the data gathered by ImageJ as a result of the uneven application of the soiling material and the small size of the

card template. However, the movement of solids in soiling after solvent vapour treatment was imaged showing detail of their behaviour through a textile, something which has not previously been available in the textile conservation published literature.

#### FTIR-ATR

Figure 5(A,B) shows comparisons of the spectra (2000–1400  $\text{cm}^{-1}$ ) from the front and back of the soiled samples before and after the application of the



**Figure 5.** (A) Spectra (averaged from 10 replicates) before and after the application of acetone to the front of samples through Reemay® for 3 min; (B) spectra (averaged from 10 replicates) before and after the application of acetone to the back of samples through Reemay® for 3 min.



solvent vapour (test R 3 min A). There is a visible difference in the carbonyl group (C=O) peak at around  $1744\text{--}46\text{ cm}^{-1}$  after treatment on both the front and back. As this particular peak is characteristic for the presence of oil, the decrease in IR absorption indicates that there is a decrease in the amount of the oil in the soiling material after the application of the solvent vapour, given that peak height is relative to the concentration of the molecule (Derrick, Stulik, and Landry 1999).

Table 1 shows the average peak height ratios  $I_{1744\text{--}46}/I_{1618}$  for the eight conditions before and after solvent application for both the front and back of the samples. The spectra were baseline corrected automatically and not manually. The Amide II peak has altered slightly in height, however, it can be seen that this also occurred at the trough between the 1618 and  $1512\text{ cm}^{-1}$  as well as between the 1512 and  $1422\text{ cm}^{-1}$ . Manual baseline correction in these areas would lead to this being resolved. However, as there is no such trough to peak alteration in the data at  $1888\text{--}1700\text{ cm}^{-1}$  or from  $1700$  to  $1618\text{ cm}^{-1}$ , the areas were used in the calculation.

The results of these ratios show a downward trend in the intensity of the oil peak after solvent application. To determine if the differences were significantly different *t*-tests were carried out on the before and after ratios. Where there was a significant difference is indicated in Table 1. Samples treated with acetone through the Gore-Tex® membrane, for both 1 and 3 min, showed a highly significant difference between the before and after treatment for both the front and back of the sample and that the application of the solvent vapour for the two different lengths of time made no difference to the movement of the oil. The 1 min acetone treatment through the Reemay® membrane showed a significant difference on the front between the before and after but there was no significant difference on the back. When treated for 3 min there was a very significant difference on the front

but no significant difference on the back. Samples treated with IDA through Gore-Tex® membranes for 1 min showed a highly significant difference on the front but no significant difference on the back, and when treated for 3 min showed a significant difference on the front but not on the back. Samples treated with IDA through Reemay® for 1 min showed a significant difference on both the back and front of the samples with *P* values of 0.044 and 0.043 respectively showing they were only just significant. After 3 min of IDA through the Reemay® membrane there was no significant difference on either the back or the front. The application of acetone has produced overall a greater change in the movement of oil on the front and back of the samples through both Gore-Tex® and Reemay® barrier layers than the application of IDA. Based on the calculated differences the order of greatest change to least when the affect to both front and back is considered is G 3 min A > G 1 min A > R 3 min A > R 1 min A > G 1 min IDA and G 3 min IDA > R 1 min IDA > R 3 min IDA.

The results show that the application of solvent vapour by a poultice method produces more than just a surface change; in fact the method allows the vapour to penetrate far enough into the fibres to cause a change in the level of oil throughout the entire textile. This was shown by FTIR analysis of both the front and back of the silk samples. The methodology allowed for the quantitative monitoring of the change/removal of oil through the depth of a textile.

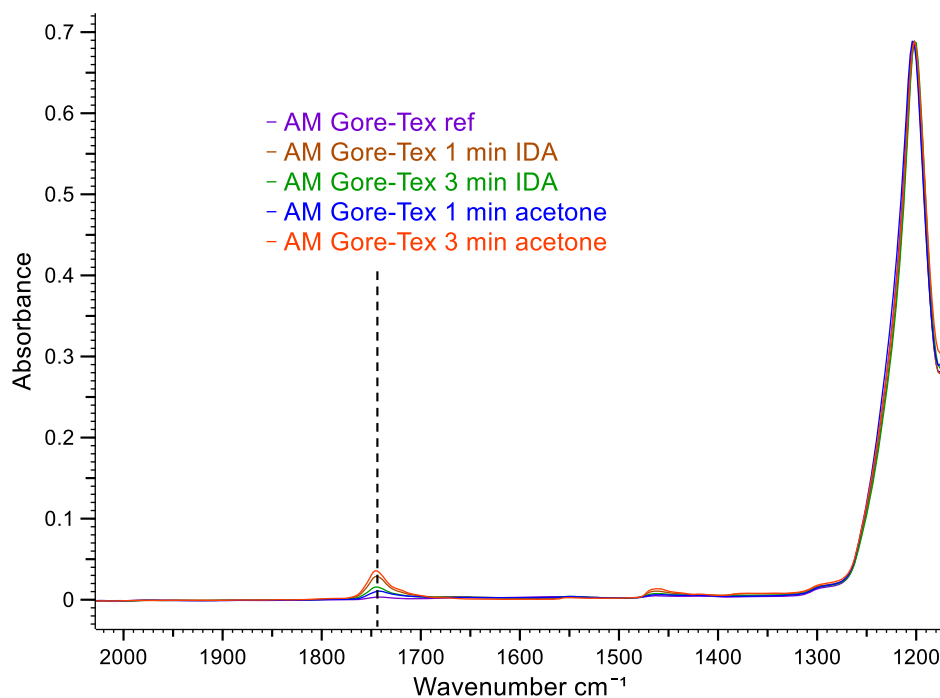
The barrier membranes have characteristic differences which impact on solvent vapour treatments. These differences, in the behaviour of the two membranes, independent of the actual solvent used, is also of interest, where a greater change of the oil peak was observed with the use of Gore-Tex®. Unlike the porous structure of Gore-Tex®, Reemay® is an irregular structure; the density of the material is not consistent which is a result of the method of production and the random way in which the threads are laid before being heat sealed. This uneven structure could be a factor in why a greater change of the carbonyl group was seen the Gore-Tex test groups. The Reemay® used in the experimentation could have, by chance, included a large area of more densely laid threads, which would have likely prohibited a larger amount of the vapour to pass through the material, as Reemay® is not designed to act as a semi-permeable membrane and so the movement of vapour is likely to be less controlled than in Gore-Tex® which has a pore structure.

Figure 6 shows the spectra of all of the PTFE sides of the Gore-Tex® pieces used, all of which show a slight peak at  $1744\text{--}46\text{ cm}^{-1}$ , indicating the presence of oil on the membrane, as shown by the dashed line. The spectra of the Reemay® pieces did not allow for this peak to be detected as a large absorbance band due to the polyester that dominated this area of the spectra.

**Table 1.** Shows the  $I_{1744\text{--}46}/I_{1618}$  ratios and SDs.

Front treatment	Before	After	<i>p</i> < .05
G 1 min A	0.44 (0.08)	0.29 (0.04)*	.001
G 3 min A	0.46 (0.07)	0.25 (0.05)*	.000
G 1 min IDA	0.45 (0.06)	0.35 (0.06)*	.001
G 3 min IDA	0.46 (0.08)	0.37 (0.07)*	.040
R 1 min A	0.42 (0.06)	0.34 (0.08)	.021
R 3 min A	0.47 (0.1)	0.35 (0.05)*	.004
R 1 min IDA	0.42 (0.07)	0.36 (0.04)*	.044
R 3 min IDA	0.43 (0.07)	0.39 (0.05)	.542
Back treatment	Before	After	<i>p</i>
G 1 min A	0.35 (0.06)	0.23 (0.03)*	.000
G 3 min A	0.37 (0.09)	0.22 (0.05)*	.000
G 1 min IDA	0.35 (0.04)	0.32 (0.05)	.161
G 3 min IDA	0.37 (0.06)	0.33 (0.05)	.086
R 1 min A	0.32 (0.07)	0.26 (0.04)	.070
R 3 min A	0.36 (0.06)	0.28 (0.04)*	.005
R 1 min IDA	0.35 (0.07)	0.30 (0.04)*	.043
R 3 min IDA	0.35 (0.08)	0.33 (0.06)	.538

\*Indicates a significant difference between before and after solvent treatment (*p* < .05).



**Figure 6.** Spectra of the Gore-Tex<sup>®</sup> pieces used in the experimentation, noting the presence of a small peak at  $1746\text{ cm}^{-1}$ , indicating the oil from the soiled samples.

The visible movement of the oil and particulate soiling is a result of the ability of the solvents used to partially solubilise the components of the soiling mixture (Mills and White 2011). IDA and acetone are solvents of high and medium polarity respectively (Museums and Galleries Commission 1992; Horie 2011), while the soiling mixture was made from oil, dust, and dirt of mixed polarity, which cannot conclusively be determined due to the unknown exact composition of the particulate matter. While the solubility of a substance generally follows the rule 'like dissolves like' (Mills and White 2011), the partial solubility of a substance in a solvent is possible due to the ability of the solvent to 'swell' the solute (Burke 1984). It is this swelling that has likely resulted in the movement of the components of the soiling mixture. Although not investigated in this research, the volatility of the solvent used for re-activation may have an effect on the movement of the soiling. This has been surmised given the more volatile nature of acetone than IDA (Horie 2011), however, the confirmation of this would require further research.

#### **Implications for textile conservation**

Surface and ingrained soiling in textile artefacts is an acknowledged and well-documented concern within the textile conservation profession. The presence of complex soiling can catalyse or accelerate deterioration of textile fibres due to chemical change of the soiling and subsequent molecular change in the fibre that can occur with the addition of environmental factors such as heat, moisture, humidity, and light (Tímár-Balázs and Eastop 1998; Lloyd and Lithgow 2011).

Particulate soiling such as dust and dirt granules can add to this type of chemical degradation over a long period of time, but the immediate concern with the accumulation of dust is the hard, sharp edges which can be a source of constant friction against the fibres, and with the natural swelling of the capillaries as a result of fluctuating RH levels, can cause mechanical damage, weakness and brittleness, and can eventually lead to the breaking of the thread (Flury-Lemberg 1988).

Given the risk of the embrittlement of textiles caused by the mechanical damage of dust and dirt particles, the movement of the particulate soiling caused by the application of solvent vapour as seen in the experimentation can be predicted to exacerbate this type of damage further. The results of the experimentation have emphasised the value in the categorisation of soiling on an historic object, for the undesirable reason of the partial solubilisation of complex soiling by solvent vapours after only 1 min of exposure, but also for the movement of oil as seen with the analysis using FTIR-ATR. This oil movement raises interesting questions regarding the potential increase of adherence of adhesive films on historic textiles. The adhesives used in textile conservation are largely soluble in water, making adherence to a greasy textile problematic (Karsten and Vuori 2015); a factor that could be made worse by the oily components of soiling combining with the adhesive upon partial solubilisation with solvent vapour. However, the use of solvent vapour, particularly acetone, could provide a better support for an area of damage, if the poultice method is used to decrease the oil prior to the

application of the adhesive film. The intentional movement of oily ingrained soiling would require careful ethical considerations, balancing the immediate support of the area of damage with the future chemical and mechanical stability of the fibres, and this area requires further research.

## Conclusions

The quantitative and qualitative methods applied to the experiment have successfully examined and answered the research question: the application of solvent vapour of acetone and IDA in a poultice method results in a change in complex particulate soiling found on silk fabric. The analysis of the large particulates using microscopy showed the movement of solids through a textile and although ImageJ did not provide quantitatively significant results (the complexity of the soiling mixture and uneven application of the soiling contributed to the uncertainty of the results) using the parameters set within this trial it demonstrated its possible future usefulness for this purpose. Analysis with FTIR-ATR provided numerical data which enabled quantitative analysis of the change/removal of oil through the depth of a textile; this quantitative monitoring has not been reported previously.

The variable of time of exposure produced some variance within the data, however the majority of the tests with an exposure time of 3 min did not produce a greater change in the movement of the soiling mixture when compared to the tests with an exposure time of 1 min, and so it can be assumed that time of exposure to solvent vapour within this time scale does not have a large effect on the movement of oil. The type of solvent and barrier layer, did however, show significant differences in the data gathered before and after, with the trials using Gore-Tex® producing a larger change in the oil, and acetone producing an overall greater effect than IDA. As this experiment did not include the addition of a layer of adhesive between the barrier membrane and soiled textile, further research would be necessary to find out if the presence of adhesive would inhibit the effect of the solvent vapour on the movement or change in a complex soiling mixture.

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