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A preliminary investigation into the effect of solvent vapour on ingrained textile soiling

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Abstract

This research paper aimed to investigate the effects of solvent vapour on ingrained soiling when applied to a textile in a poultice method for the re-activation of an adhesive film.

The research was based around a tailored and focused methodology in which replicates of silk fabric were artificially soiled with a mixture of organic and inorganic particulate soiling and oil. Six variables were included in the experiment which were predicted to influence any change in the level of soiling. The vapour of acetone and IDA was applied to the replicates by way of dampened blotting paper, through two barrier layers, Gore-Tex® and Reemay®, for two exposure times, one minute and three minutes. The replicates were analysed with light microscopy, with the addition of ImageJ image analysis software, and FTIR-ATR, with the addition of calculated absorbance ratios. The results of the tests showed movement of the particulate soiling and a decrease in oil, on the front and back of all replicates, indicating that solvent vapour can partially solubilise textile soiling. Further analysis is required to determine the statistical significance of the results.

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Abbreviations

AIC: American Institute for Conservation of Historic and Artistic Works CCI: Canadian Conservation Institute CTC: Centre for Textile Conservation FTIR-ATR: Fourier transform infrared spectroscopy attenuated total reflectance ICOM: International Council of Museums IDA: Industrial denatured alcohol IIC: The International Institute for Conservation of Historic and Artistic Works IMS: industrial methylated spirits IR: Infrared JAIC: Journal of the American Institute for Conservation NATTC: North American Textile Conservation PTFE: polytetrafluoroethylene RH: Relative humidity TCC: Textile Conservation Centre

TSG: Textile Specialty Group

1. Introduction

1.1. Introduction

The application of an adhesive treatment to a textile is a complex process in which the conservator must balance the ethical considerations of reversibility/re-treatability and minimal intervention with the type and condition of the object, the strength of the bond required, and the thickness of the adhesive film and supporting fabric. The experience and knowledge of the conservator is an integral component to the success of such a complex treatment, as the decisions made on the activation method, adhesive and substrate choice, and the length of activation time required to produce a satisfactory bond largely dictate the success of the treatment.¹

In lieu of this level of in-depth knowledge, research into past adhesive treatments can prove useful for the creation of treatment parameters. The author undertook such research for this dissertation, as well as her recent treatment of an early 19th century silk shoe (Glasgow Museum no. E.1945.11.e.2/CTC 374.2) which formed the basis of the research question presented in this dissertation. Object treatment based dissertations written by former students at the Textile Conservation Centre (TCC)² provide a valuable source of documentation on the application and solvent re-activation of adhesive films, with experimentation focused on various barrier layers, solvent application methods, support materials and the exposure time of the adhesive film to the solvent, and helped

¹ Irene Karsten and Jan Vuori, "Textiles" in *Adhesive Compendium for Conservation*, edited by Jane L. Down, 149. (Ottawa: Canadian Conservation Institute, 2015). ² The Textile Conservation Centre (TCC) closed in 2009 and re-opened in 2011 as the Centre for to form the underlying knowledge of the variables of adhesive re-activation implemented in this study.^{3, 4, 5, 6, 7}

As mentioned above, the basis of the research question for this dissertation was formed during the conservation of an early 19th century silk shoe, which was soiled with ingrained soiling and was unable to be cleaned due to the material composition. To find a textile that is of a mixed composition, that cannot be cleaned and requires an adhesive support treatment is not uncommon, and these two important aspects of the conservator's decision making process are briefly discussed below.

1.2. <u>Cleaning historic textiles</u>

The conservation of textile artefacts will likely begin with cleaning, a process that aims to remove unwanted dust and dirt. The conservator's options for cleaning include mechanical cleaning utilising brushes, vacuums and chemical sponges to remove or loosen larger surface soiling, or wet or solvent cleaning, the aim of which is to solubilise and remove smaller molecular soiling that cannot be removed by mechanical means.⁸ Dirt is removed as part of the conservation process as it can be visually distracting; the colours and patterns of a textile can be muted by large amounts of particulate soiling, and dirty textiles on display can seem uncared for.⁹

³ Cordelia Rogerson, "The examination and conservation of a painted cotton Scottish Reform Banner (1832): Two adhesive treatments, Masters dissertation, Courtauld Institute of Art, University of London, 1997.

⁴ Susan M. Stanton, "The conservation treatment of an embroidered panel dating from the 1930's using a combination of adhesive and stitching techniques," Masters dissertation, Courtauld Institute of Art, University of London, January 1999.

⁵ Elisabet Cerdà i Durà, "Conservation strategy for an English 17th century raised work box, the 'Mercy Jewel Cabinet'," Masters dissertation, Courtauld Institute of Art, University of London, January 1999.

⁶ Helen Bacchus, "From private meditation to public access: the conservation of a rare chinese embroidered thangka," Masters dissertation, Courtauld Institute of Art, University of London, January 1999.

⁷ Penny Hughes, "The Characterisation and conservation of an unusual raised work picture dated 1649 TCC 2550," Masters dissertation, Courtauld Institute of Art, University of London, January 2000.

⁸ Foekje Boersma, *Unravelling Textiles: A Handbook for the Preservation of Textile Collections,* (London: Archetype Publications Ltd., 2007), 135-136.

⁹ Helen Lloyd and Katy Lithgow, "Physical agents of deterioration" in *The National Trust Manual of Housekeeping*, (Swindon: The National Trust, 2011), 63.

As well as the undesirable visual effects soiling can have on a textile, dust and dirt can have long-term degradative effects. Large particles of dust can have sharp edges, causing or exacerbating mechanical damage, while elements of the surrounding environment such as humidity and light levels can react with the various components that make up dust and cause chemical degradation. Dust and dirt can attract insects and microbial organisms, while greasy or oily stains can result in the accumulation of even larger amounts of dust and dirt.¹⁰ As such, the removal of soiling through various cleaning methods is significant to the long-term stability, preservation, and visual aesthetics of a textile artefact.

However, the cleaning process is complicated by a number of factors that play an integral role in influencing the way in which a treatment is formulated. An object's condition, material composition, size, structure, and origin all influence the way in which it is cleaned, as unconsidered cleaning can result in damage, disfiguration, or the loss of important historical information.^{11, 12} When considering these factors and the complexity of many historic textile objects, it is clear that the removal of all types of soiling is not always feasible.

1.3. Supporting historic textiles

Supporting or protecting areas of weakness or damage on a textile object can be accomplished two ways: with support stitching or an adhesive treatment. Both methods aim to strengthen the weakened area by attaching a stronger support fabric, thus reducing the risk of further damage. A stitched treatment does so with a needle and thread, and an adhesive treatment with a thin film of the chosen adhesive, which is usually allowed to dry on the support fabric, and is re-activated once positioned on the object with either heat or organic solvent. As with the cleaning of historic textiles there are a variety of considerations that can influence the conservator's decision on the most appropriate support method for the object. An adhesive support treatment can be

¹¹ Dinah D. Eastop and Mary M. Brooks, "To Clean or Not to Clean: The Value of Soils and Creases" in *ICOM Committee for Conservation, 11th Triennial Meeting, Edinburgh, 1-6 September 1996: Preprints,* ed. Janet Bridgland, 687, (London: James and James, 1996).

¹⁰ Helen Lloyd and Katy Lithgow, "Physical agents of deterioration", 63-64.

¹² Virginia Greene, "Using case studies to examine the decision-making process for cleaning ethnographic objects" *Journal of the American Institute for Conservation* vol. 45, no. 3 (2006): 185.

utilised on textiles that are too brittle to withstand the action of stitching, that have painted components, or that prove to be technically too challenging to stitch into, for example, a three dimensional object. Re-activating the adhesive film by heat or solvent vapour again has variables for the conservator to consider. Solvent re-activation is generally 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.¹³

With the understanding that the condition, material composition, and structure of an object can be an influential factor on the level of cleaning undertaken, as well a factor on what type of support can be used, a hypothesis can be made that historic textiles which are supported with an adhesive support treatment may have some level of soiling retained in the fibres. This, combined with the use of solvents in the re-activation of the film has formed the research question of this dissertation:

Is there a change of the soiling found in textiles with the application of solvent vapour, as applied for the re-activation of an adhesive treatment?

In order to answer the research question, a set of aims and objectives has been formed.

1.4. Aims and Objectives

<u>Aims</u>

- To investigate the potential effect that solvent vapour has on ingrained soiling in textiles
- To determine if there is a difference in the effect of the solvent vapour if different types of barrier layers are used
- To determine if the effect of the solvent vapour on the soiling changes with varying lengths of exposure

¹³ Lynda Hillyer, Zenzie Tinker, and Poppy Singer. "Evaluating the Use of Adhesives in Textile Conservation. Part 1: An Overview and Survey of Current Use." *The Conservator* 21 (1997): 37.

Objectives

- To determine sample preparation and an analytical methodology which will measure these changes in a robust and repeatable manner
- To artificially soil silk samples with a mixture created to replicate the type of soiling that could be found on an historic textile
- To apply the vapour of two solvents commonly used in adhesive re-activation treatments through two different barrier layers, for two different lengths of time
- To analyse the samples before and after the solvent vapour application with the aim of recording both visual and measureable changes, using analytical equipment

1.5. <u>Overview</u>

This dissertation has been organised into five chapters:

Chapter two provides a review of published and unpublished literature on the solvent re-activation of adhesive films and the potential disfiguring and damaging effects recorded by the authors, with a particular focus on the documentation of unusual or unexpected results. Chapter three outlines the methodology taken to design the experiment and methods of analysis which aimed at answering the research question, and Chapter four discusses the results and findings of the experiment. Chapter five concludes the research and provides areas for further research.

2. <u>Chapter: Literature review: the potential damaging effects of solvents in the</u> <u>re-activation of adhesives in textile conservation</u>

2.1. Introduction

When comparing the long-standing tradition of the use of adhesives in the conservation and restoration of objects such as canvas paintings, a process which dates to approximately the 17th century¹⁴, or paper, with the use of starch paste dating several hundred years¹⁵, the use of adhesives in textile conservation seems remarkably new, having only been introduced to the profession in the 1950's.¹⁶ Although this introduction came with some distain,¹⁷ an increase in the scholarly literature and conference papers regarding the use of adhesives for the consolidation and support of textile artefacts was seen from the 1960's to the late 1990's. ¹⁸, ¹⁹, ²⁰, ²¹, ²², ²³, ²⁴ Despite this growing body of literature, a survey undertaken by Hillyer, Tinker, and Singer in 1997 recorded the infrequent use of adhesive treatments in the UK, Europe, and North America, particularly regarding solvent re-activated treatments, with – out of

¹⁴ Westby Percival-Prescott, "The lining cycle: causes of physical deterioration in oil paintings on canvas: lining from the 17th century to the present day," in *Lining Paintings: Papers from the Greenwich Conference on comparative lining techniques*, ed. Caroline Villers, 1-15 (London: Archetype Publications Ltd., 2003).

¹⁵ V.D. Daniels, "A Study of the Properties of Aged Starch Paste (*Furu-Nori*)" in *IIC The Conservation of Far Eastern Art*, ed. John S. Mills, Perry Smith and Kazuo Yamasaki, 5-10, (London: The International Institute for Conservation of Historic and Artistic Works, 1988).
¹⁶ Lynda Hillyer, Zenzie Tinker, Poppy Singer, "Evaluating the use of adhesives in textile conservation: Part I an overview and surveys of current use" *The Conservator* no. 21 (1997): 37.
¹⁷ H. J. Plenderleith, *The Conservation of Antiquities and Works of Art: Treatment, Repair, and Restoration*, (London: Oxford University Press, 1956), 106.

¹⁸ J. Lodewijks, "The use of synthetic material for the conservation and restoration of ancient textiles" in *IIC 1964 Delft Conference on the Conservation of Textiles, collected preprints 2nd edition, 1965*, (London: International Institute for Conservation, 1965), 79-85.

¹⁹ H. J. Plenderleith and A. E. A. Werner, *The Conservation of Antiquities and Works of Art: Treatment, Repair, and Restoration, second edition,* (London: Oxford University Press, 1971), 116.

 ²⁰ Judith Hofenk-de Graaff, Hydroxyl propyl cellulose, a multipurpose conservation material.
 ICOM-CC 6th triennial meeting preprints vol. III, Ottawa, 1981. 81/14/9-1 – 81/14/9-7.
 ²¹ Shelia Landi, *The Textile Conservator's Manual: second edition*, (Oxford: Butterworth Heinemann, 1992), 40-42.

²² United Kingdom Institute for Conservation, *Lining and Backing: The Support of Paintings, Paper and Textiles* (Hampshire: The United Kingdom Institute for Conservation of Historic and Artistic Works, 1995),

²³ United Kingdom Institute for Conservation, Textile Section, *Starch and Other Carbohydrate Adhesives for Use in Textile Conservation*, (London: UKIC, 1995).

²⁴ Ágnes Tímár-Balázsy, and Dinah Eastop, *Chemical Principles of Textile Conservation* (Oxford: Elsevier, 1998), 304-331.

80 respondents – only 13% using solvents for thermoplastic adhesive re-activation, and 9% for carbohydrate adhesive re-activation (all of which from the UK), and 4% and 7% of the respondents using a combination of solvent and low pressure table reactivation, from the UK and North America respectively.

According to the survey undertaken, the respondents quoted ageing, flexibility, reversibility, health and safety, and the working properties of the adhesive – namely the re-activation temperature – as concerns regarding the use of adhesives.²⁵ The potential degradative effects of a heat sealing treatment are a documented concern amongst authors of adhesive based literature^{26, 27}, with the use of solvents often suggested as an alternative to heat sealing when the textile is extremely fragile, however, the concern with the use of solvents is largely (and understandably) focused on the health implications to the textile conservator, and not necessarily the object.^{28, 29} The practice of adhesive treatments saw rigorous evaluation and testing in the 1980's and 1990's, the most notable being the testing performed by the Canadian Conservation Institute (CCI) on selected polyvinyl acetate and acrylic adhesives, testing which begun in 1983³⁰ with the final report delivered in 2015.³¹ From the 1990's to the early 2000's the re-evaluation of adhesive treatments on objects after years of display and storage emerged indicating the failures and successes of the treatments; highlighting areas of required research and the processes necessary for the refinement of techniques,

 ²⁵ Hillyer, Tinker, Singer, "Evaluating the use of adhesives in textile conservation", 41-44.
 ²⁶ Michaela Keyserlingk, "The use of adhesives in textile conservation" in *ICOM 9th Triennial Meeting, Dresden, German Democratic Republic 26-31 August 1990 Preprints Volume I*, ed. Kirsten Grimstad, 308 (Paris: ICOM Committee for Conservation, 1990).

²⁷ Carole Dignard, "Choosing Adhesives and/or Consolidants for Conservation Treatments" in *Adhesive Compendium for Conservation*, ed. Jane L. Down, 117 (Ottawa: The Canadian Conservation Institute, 2015).

 ²⁸ Hillyer, Tinker, Singer, "Evaluating the use of adhesives in textile conservation", 43-44.
 ²⁹ Boris Pretzel, "Evaluating the use of adhesives in textile conservation Part II: Tests and evaluation matrix" *The Conservator* 21, vol. 1 (1997): 54-55.

³⁰ Jane L. Down, "Adhesive Testing at the Canadian Conservation Institute, Past and Future" in *Adhesives and Consolidants IIC Preprints of the Contributions to the Paris Congress, 2-8 September 1984*, ed. N.S. Brommelle, Elizabeth M. Pye, Perry Smith, and Garry Thompson, 18-21 (London: The International Institute for Conservation of Historic and Artistic Works, 1984).

³¹ Jane L. Down, "The evaluation of selected poly(vinyl acetate) and acrylic adhesives: A final research update," *Studies in Conservation* vol. 60, no. 1 (2015): 33-54.

ultimately advancing the profession by creating a method of support treatment that has been used to present day.³²

This review of the literature on the potential disfiguring or unwanted effects of solvent re-activated adhesives looks primarily at case studies documenting testing and treatments where the outcomes have been considered surprising or undesirable. This research does not aim to simply point out what was 'wrong' with the conservation, but highlight the gaps in the knowledge of the use of solvent vapour which the methodology and results chapters of this dissertation aim to begin to fill, and much like the testing and evaluation taken by conservators in the 1980's-2000's, this research aims to enhance the existing body of knowledge surrounding the use of solvent re-activated adhesive treatments.

2.2. Literature discussing the potential for staining on testing materials

Within the body of literature on the use of adhesives in textile conservation there are only a select few written works that mention the potential disfiguring effects of the use of solvents for the re-activation of adhesive films. The few that do mention the potential negative outcomes discuss such information in very brief terms, using somewhat ambiguous language. The majority of the literature discussed in this review dates from the latter half of the 1990's to the early 2000's, in which the application of the solvent to the adhesive film occasionally differed from what is documented in current conservation literature, and the method known to the author, which is the application of the vapour of the chosen solvent through a barrier layer.

Lyndall Bond, in her discussion of the conservation of an embroidered picture, tests several methods of applying Klucel G® dissolved in industrial methylated spirits (IMS) to test fabric. She noted in her experimentation "when the adhesive was applied by brush directly onto the test piece it caused staining".³³ Bond's method of direct application of the adhesive to the test piece is quite different from the focus of this

³² Lynda Hillyer, "Advances in adhesive techniques-the conservation of two Coptic tunics at the Victoria and Albert Museum" in *Textile Conservation: Advance in Practice*, ed. Frances Lennard and Patricia Ewer, 181-188 (Oxford: Butterworth Heinemann, 2010).

³³ Bond, "The Practical Conservation of a Painted and Embroidered Silk Picture", 49.

research paper, however, what is of note in this writing is the lack of information of the "test piece". Is the reader to assume that the test piece is representative of the object in terms of condition, and what exactly does "staining" actually imply, for example, is the "staining" visible adhesive or the movement of dye or soiling? Bond's use of the word 'staining' to reference the appearance of the test piece after adhesive testing is one of the ambiguous phrases used throughout the literature discussed here. Although the word 'staining' is not in essence ambiguous, the use of it (and words such as blotchy, ringing and darkening, used in the discussed literature to follow) to describe clean test fabric raises questions around what is actually meant by the authors' description, and how this disfiguring result found in testing transfers to the results of the treatment of an historic object.

These questions raised by Bond's testing are echoed in the analysis of two unpublished TCC dissertations: Cordelia Rogerson's and Celia Medina's solvent re-activated adhesive testing for their partial fulfillment of the MA programme.^{34, 35} Although the research projects were completed six years apart, both authors examine the different working properties of solvent re-activated adhesive films, utilising both liquid solvent and solvent vapour. What is found in both dissertations is that the use of solvent vapour produced less "darkening" and "ringing" on the test fabric, when compared to the results of the use of liquid solvent.^{36, 37} Again the reader is presented with unclear terms describing the appearance of the test fabrics, with only an explanation from Rogerson, who suggests that the disfiguring appearance is the adhesive being driven into the fabric.³⁸ However, of particular note is a statement made in Medina's research, that solvents "can be applied to soiled textiles" and they "will not risk moving soil further into the textile as a starch paste (or wet adhesive) would".³⁹ Supposing Rogerson is

³⁴ Cordelia Rogerson, "The examination and conservation of a painted cotton Scottish Reform Banner (1832): Two adhesive treatments," Masters dissertation, University of Southampton, 1997.

 ³⁵ Celia Medina, "The application of solvent reactivation of adhesives in textile conservation: an analysis of practice and research," Masters dissertation, University of Southampton, April 2003.
 ³⁶ Rogerson, "The examination and conservation of a painted cotton Scottish Reform Banner", 55-56.

 ³⁷ Medina, *The application of solvent reactivation of adhesives in textile conservation*, 52.
 ³⁸ Rogerson, "The examination and conservation of a painted cotton Scottish Reform Banner", 55-56.

³⁹ Medina, *The application of solvent reactivation of adhesives in textile conservation*, 10.

correct in her suggestion that the change in appearance of the clean test fabric is caused by the adhesive being driven into the fabric, one could presume that the adhesive would, in some way, effect the soiling, causing the statement provided by Medina to be questioned.

2.3. <u>Literature discussing the potential for staining from adhesive treatments</u> <u>in conservation practice</u>

Considering the potential for solvents used in the re-activation of adhesive treatments to 'stain' or 'darken' a new clean sample of fabric, it is not surprising to find conservators voicing concerns over the potential for a visual change to occur on a soiled historic object with the use of the same types of support treatments. Although within the body of published literature these authors are few, their concerns and observations are especially valid within this particular research paper, based on the knowledge of the characterisation and solubility of ingrained soiling in organic solvents. The precautionary methods taken by conservators to avoid adverse effects and unwanted staining caused by solvent application include the avoidance of humidity for adhesive re-activation and in its place the application of the vapour of a solvent of medium polarity⁴⁰, the use of a poultice method, avoiding the use of liquid solvent⁴², and the prevention of ringing on the textile by the complete avoidance of solvent, instead using a heat sealing treatment.⁴³ These published works indicate that there has been, since the late 1990's, a concern over the effect of solvents used in adhesive re-activation.

⁴⁰ Annie Lord and Howard Sutcliffe, "Combining cold lining and solvent reactivation for the treatment of an embroidered silk picture a case study," *The Conservator* 24, vol.1, (2000): 89-95, accessed 20 June 2017, <u>http://dx.doi.org/10.1080/01410096.2000.9995155</u>

⁴¹ Penny Hughes, "The Characterisation and conservation of an unusual raised work picture dated 1649 TCC 2550". Postgraduate diploma, Courtauld Institute of Art, University of London, January 2000.

⁴² Kate Gill, and Foekje Boersma. "Solvent reactivation of hydroxypropyl cellulose (Klucel G®) in textile conservation: recent developments." *The Conservator* 21, vol. 1 (1997): 12-20, accessed 22 June 2017, <u>http://dx.doi.org/10.1080/01410096.1997.9995111</u>

⁴³ Heidi Cutts, Lynne Harrison, Catherine Higgitt and Pippa Cruickshank, (2010), "The image revealed: study and conservation of a mid-nineteenth-century Ethiopian church painting" in *The British Museum Technical Research Bulletin* vol. 4, 1-17.

The practice of lining and supporting works of art on paper employs many techniques similar to those found in textile conservation, particularly the use of heat and solvents to re-activate adhesive coated paper and tissue supports.⁴⁴ Whilst a review of the literature concerning the adhesive support methods used on paper falls outside the scope of this research, Alan Donnithorne's article "Paper Lining: An Overview" is worth noting in this research as he explicitly outlines the potential issues conservators may face when using adhesive supports, and states that the use of solvents risks "staining from [the] migration of water-soluble or solvent-soluble discolouration products (sometimes referred to as water stains)".⁴⁵ As with case studies presented in textile conservation literature, the re-activation of adhesives on paper can be accomplished with liquid solvent, solvent vapour, and humidification.⁴⁶ Although the specific type of re-activation which risks "water stains" is not explicitly specified in Donnithorne's article, the open acknowledgement of the risks involved with the use of solvents is worth noting given the similarities in materials and degradation products in both paper and textile objects.⁴⁷

A poultice method for the slow and controlled introduction of solvent vapour, avoiding staining as a result of liquid solvent, is a considered approach to an adhesive treatment, particularly on very soiled textiles that cannot be cleaned, however, during the review of adhesive literature for this research paper, a case study of particular interest caused the author to question the reliability of the poultice method, specifically with the use of acetone. In the conservation of a Korean painted silk banner by Mika Takami, an adhesive treatment was applied and re-activated with acetone through Gore-Tex® for a total of ten minutes of exposure time, and five minutes held under weight. The removal of the Gore-Tex® layer revealed that the paint layer on which the adhesive coated silk crepeline was being re-activated had penetrated into the Gore-Tex®, having been carried by the vapour as it evaporated, a result Takami equated to the solubility of the

⁴⁴ Alan Donnithorne, "Paper Lining: An Overview" in *Lining and Backing: The Support of Paintings, Paper and Textiles,* (Hampshire: The United Kingdom Institute for Conservation of Historic and Artistic Works, 1995), 16.

⁴⁵ Donnithorne, "Paper Lining: An Overview", 17.

⁴⁶ Donnithorne, 20.

⁴⁷ Joanna M. Kosek, "Washing Paper in Conservation" in *Paper and Water: A Guide for Conservators*, ed. Gerhard Banik and Irene Brückle, 313-314 (Oxford: Butterworth Heinemann, 2011).

drying medium in the oil paint in acetone.⁴⁸ This research undertaken by Takami suggests the need for (as much as possible) the understanding of the type of soiling, medium, or decorative surface of the textile artefact before applying a solvent reactivated adhesive treatment, as based on Takami's results, the solubilisation of a substance is possible given sufficient exposure to a solvent.

In the survey circulated as part of Medina's 2003 dissertation, seven out of eighteen respondents replied acknowledging their concerns over the possible cleaning effects and potential solubility of textile components during solvent re-activation of an adhesive film.⁴⁹ The movement of the paint during the adhesive re-activation on the Korean banner suggests that the reservations held by the conservators who answered the survey may in fact be valid.

2.4. Conclusion

The use of adhesives on a textile artefact is a complex part of the conservator's decision making process, which is made more ethically challenging given the potential disfiguring and damaging effects of the use of solvent vapour in the treatment. The solubilisation and partial solubilisation of degradation products and decorative elements of the object have been documented in Donnithorne and Takami's research, and it is the aim of this dissertation to expand upon these findings, quantitatively measuring change produced by the application of solvent vapour to soiled textile samples.

⁴⁸ Mika Takami, "The conservation of a Korean painted silk 'tiger' banner: solvent reactivation of acrylic adhesive," Postgraduate diploma, Coutauld Institute of Art, University of London, May 2000, 106.

⁴⁹ Medina, "The application of solvent reactivation of adhesives textile conservation," 60.

3. Methodology and materials

3.1. Introduction

This chapter will introduce the methods used to answer the research question:

Is there a change of the soiling found in textiles with the application of solvent vapour, as applied for the re-activation of an adhesive treatment?

An experiment was designed to replicate the process of applying solvent vapour for adhesive re-activation on artificially soiled silk, and included six different variables that were predicted to influence any perceivable or measureable changes in the level of soiling:

- Two different solvents commonly used for adhesive re-activation in textile conservation were applied to the soiled samples
- Two different barrier layers were used for both solvents
- The solvent vapour was applied through each barrier layer for two different amounts of time

The replicates of silk fabric were artificially soiled with a combination of oil and organic and inorganic particulate matter, and were humidified to aid in the adherence of the soiling to the silk fibres. The samples were analysed using light microscopy before and after the application of solvent vapour at two different magnifications, at which point they were photographed and the images evaluated with the scientific image analysis software programme ImageJ⁵⁰ 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 (from here on referred to as FTIR-ATR). A series of ratios using the absorbance values of the changing carbonyl peak 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.

3.2. Characterisation of soiling on historic textiles

⁵⁰ "Welcome", ImageJ, accessed 16 August 2017, <u>https://imagej.net/Welcome</u>

Ingrained soiling found on textile objects may be deposited from handling, use, display, storage, or excavation, and as such the soiling is a complex mixture of organic and inorganic matter, often including salts, oils, waxes, clay, soot, and skin.⁵¹ Although the exact composition of soiling found on textiles is almost always unknown, an informed conservator can often make an educated guess by determining the possible biography of the object, meaning, how the object could have been used or displayed from the time it was created to the present day.⁵² Historic objects that have likely been used or stored within an urban setting will generally be soiled with particulate matter that is a combination of larger particulates in the form of heterogeneous dust, comprised of fragments of skin, textile fibres, oils, grease, and inorganic salts, and fine particulate matter in the form of 'black soil': dark disfiguring soiling from a number of different sources that is usually grouped together under the heading 'soot'. Although this type of black soiling is difficult to accurately identify, it often is a result of the burning of oil, coal, candles, and tobacco.⁵³ Sooty particulate soiling can be a combination of organic and inorganic carbon, which when formed as a by product of decomposition brought on by high temperatures, has a consistency similar to lubricating grease. This, as well as oils, greases, and fats from cooking facilities and human sebum, can explain the often oily texture of tapestries, furniture, household textiles, and items of clothing that would have been subjected to a close proximity to sources of heat and human activity during use.^{54, 55} Although dusty soiling is usually considered to be surface soiling that does not adhere as well to a textile due to the large particle size, dust can be attracted to oils and greasy soiling, and become well adhered to the textile.⁵⁶ Many oily types of soiling have a lower surface tension, and therefore

⁵² Dinah Eastop, "The Biography of Objects: A Tool for Analysing an Object's Significance" in *ICCROM International Workshop on Flexible Materials in Asian Collections: Exchange of Approaches to Conservation, Presentation and Use 7th-25th April 2003*, (Kuala Lumpur: Department of Museums and Antiquities, 2003), 100-113.

⁵¹ Ágnes Tímár-Balázsy and Dinah Eastop. *Chemical Principles of Textile Conservation,* (Oxford: Elsevier, 1998), 157-160.

⁵³ Josep Grau-Bové and Matija Strlič, "Fine particulate matter in indoor cultural heritage: a literature review" *Heritage Science* 1:8 (2013): 3.

 ⁵⁴ Fenella G. France, Suzanne Thomassen-Krauss, Alberto Nuñez, and William N. Marmer,
 "Analysis of soiling and trace contaminants of the Star-Spangled Banner", in *Strengthening the Bond: Science and Textiles, North American Textile Conservation Conference 2002: Preprints* (Philadelphia: The North American Textile Conservation Conference, 2002), 155-164.
 ⁵⁵ Tímár-Balázsy and Easto,. *Chemical Principles of Textile Conservation*, 158-159.

⁵⁶ Tímár-Balázsy and Eastop, 158-159.

can act as a carrier for larger particulate soiling, drawing it into the fibres of the object.⁵⁷

This type of complex ingrained soiling is of particular interest in this research paper. As was outlined in Chapter 1 of this dissertation, this type of soiling is often very difficult to remove without highly interventive measures such as wet or solvent cleaning; measures which are not always possible given the composite nature of many textile objects. The experimentation, which is outlined and explained in the following sections, aimed to create a complex soiling mixture representative of the soiling distinctive to many historic objects.

⁵⁷ France, et. al, "Analysis of soiling and trace contaminants of the Star-Spangled Banner", 164.

3.3. Sample preparation

3.3.1 <u>Artificial soiling mixture</u>

Based on what can be understood of the complexity of the characterisation of soiling on historic objects, a mixture comprising of organic and inorganic matter was created to artificially soil the textile samples to be tested.

The soiling mixture comprised of:

- A Midelney subsoil, comprising of 53% clay and a 0.41% iron oxide content.⁵⁸
 This particular soil was chosen because of its high inorganic content and low iron content.
- Soot, which was formed of a mixture of soot from two different locations in Scotland: the west coast and the highlands. The highland soot came from a wood burning fire, and the west coast soot a coal, wood, and fuel burning fire. Soot was chosen as a component for the soiling mixture as it provided a carbon content and a slightly greasy texture.
- Dust, from three different sources. The dust mixture was a combination of the contents from four museum vacuums located at the Centre for Textile
 Conservation and two household vacuums. Dust was chosen for a complex composition of organic and inorganic compounds and large particle size. The dust was collected and sieved through 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.⁵⁹

⁵⁸ Ahmad Bakhsh, *Studies on the Chemistry and Behaviour of zinc in soil*. PhD thesis, University of Glasgow, 1988, 52-54.

⁵⁹ Tímár-Balázsy and Eastop. *Chemical Principles of Textile Conservation*, 160

The experimental design was finalised by carrying out a set of preparatory tests aimed at determining the parameters necessary to best answer the research question. Various ratios of the soiling components were applied to seven silk samples which were humidified at three different RH levels, and analysed with FTIR 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 FTIR and microscopy were finalised, and are discussed below.

3.3.2 <u>Application of soiling mixture</u>

Eighty replicate samples of silk measuring 50 mm x 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. The use of eighty replicates ensured that multiple samples would be tested for each experimental variable, with the aim of achieving statistically viable results. 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.⁶⁰ The medium weight habotai silk was chosen as it has a slightly more open weave structure. Silk threads are smooth with few twists, and in comparison to other more textured fibres such as wool or cotton the morphology of silk is less conducive to the retention of particulate soiling.⁶¹ However, the open weave structure of the silk 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.

⁶⁰ B. P. Saville, *Physical testing of textiles*, (Cambridge: Woodhead Publishing Limited, 1999), 132-134.

⁶¹ Tímár-Balázsy and Eastop. Chemical Principles of Textile Conservation, 160.

The eighty cut 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 grams of mixture was used to soil the samples, and comprised of

- 30% soil
- 30% soot
- 20% dust
- 20% oil

The four soiling components were combined in a Pyrex 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 (polyester sheeting), and sealed with brown packing tape (see figure 1). The samples were humidified to ensure that the soiling mixture became cemented to the threads. The effect of humidity on soiled textiles has been well documented in 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 at 80% RH. ^{62, 63, 64} 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

⁶² Raj Kumar, A. M. Dave, H. C. Srivastava, "Effect of Humidity on Soiling Behavior of Textiles" *Textile Research Journal* vol. 54 issue 9 (1984): 585-589.

⁶³ Peter Brimblecombe, David Thickett, Young Hun Yoon, "The cementation of coarse dust to indoor surfaces" *Journal of Cultural Heritage* 10: (2009) 410-414.

⁶⁴ Dafni Kyropoulou, "Scanning electron microscopy with energy dispersive X-ray spectroscopy: an analytical technique to examine the distribution of dust in books" *Journal of the Institute of Conservation*, vol. 36 no. 2, (2013): 173-185.

remained at this level \pm 5% over the remaining two days of humidification. The temperature of the chamber was recorded as 22°C \pm 3°C; the environment within the chamber was monitored with a Hanwell monitor.



Figure 1: humidification of the soiled samples (each measuring 50mm x 50mm) to ensure cementation of the soiling mixture

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, as an object would very likely be vacuumed to remove any loose particulate soiling before implementing more interventive treatment (see figure 2). The eighty samples were numbered with a 6B graphite pencil in the bottom right corner to ease identification.



Figure 2: vacuuming the samples to remove any loosely adhered particulate soiling

The eighty samples were randomly separated into eight groups using a random number generator.⁶⁵ Each group contained ten replicates; this allowed for statistically viable results to be gained taking into account the possibility for anomalies. Each group was assigned a barrier layer, time length of application of the solvent vapour, and a solvent. The test groups are 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
- R_3 min_IDA

⁶⁵ "Custom random number generator", Math Goodies, accessed 22 July, 2017, <u>http://www.mathgoodies.com/calculators/random no custom.html</u>.

3.4. Organic solvents used in experimentation

Acetone and industrial denatured alcohol (IDA) were chosen as the solvents to be used in the experiment because they are regularly used in textile conservation practice, as documented in the adhesive re-activation case studies presented and discussed in Chapter 2. These solvents are the two used in the taught adhesive block at the CTC⁶⁶, which is the course that formed the authors experience with adhesive treatments, and was is part the inspiration for this dissertation research.

3.5. Barrier layers

Two different materials were used as a barrier layer between the soiled silk sample and the solvent dampened blotting paper.

3.3.3 <u>Gore-Tex®</u>

Gore-Tex® 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-8 µm in diameter, permeable to vapour but not to liquid.^{67, 68} Gore-Tex® was chosen for this experiment as it is commonly used in the re-activation of adhesives in conservation practice, as documented in the literature discussed in Chapter 2 of this dissertation, and it has been used by the author during conservation treatments.

3.3.4 <u>Reemay®</u>

Reemay® is a randomly spun bonded polyester fabric (Preservation Equipment P492-2014, 34 gsm). The use of Reemay® as a barrier layer in adhesive re-activation is not

⁶⁸ I. Tarsiche, E. Hopîrtean, and D. Ciurchea, "Least-squares analysis of ammonia diffusion through PTFE membranes" *Measurement Science and Technology* 8 (1997): 1367. Accessed 12 August 2017. <u>http://iopscience.iop.org.ezproxy.lib.gla.ac.uk/article/10.1088/0957-0233/8/11/026/meta;jsessionid=DA8775019E0B541BB5E5B14CF2F06334.c1.iopscience.cld.i op.org.</u>

⁶⁶ CTC, "Principles and Practice: Advanced Skills Adhesive Practical: Workshop notes" *Principles and Practice* adhesive workshop notes (Glasgow: Centre for Textile Conservation and Technical Art History, 2016), 1-25.

⁶⁷ M.J. Smith, A. Kerr, and M.J. Cowling, "Effects of marine biofouling on gas sensor membrane materials" *Journal of Environmental Monitoring* vol. 9 (2007): 1381.

found in conservation literature, instead it is most often used as a material for packing objects and creating protective covers,⁶⁹ and is used in interventive conservation treatments as a support aid for delicate objects during wet cleaning treatments,⁷⁰ and as a backing material in leather conservation.⁷¹

However, Reemay® was chosen for this experiment as the author recently used it as a barrier layer for the application of a solvent re-activated adhesive overlay during the conservation of a 19th century silk shoe (Glasgow Museum no. E.1945.11.e.2/CTC 374.2).

3.6. 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 reactivation of an adhesive film on a flat textile, as taught at the CTC.⁷² 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 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.⁷³ The dampened blotting paper was then placed over the

⁶⁹ Nancy Kerr, Linda Capjack and Robert Fedosejevs, "Ability of textile covers to protect artifacts from ultraviolet radiation" *Journal of the American Institute for Conservation* vol 39, no. 3, article 3, (2000): no page numbers, accessed 4 August 2017, http://cool.conservation-us.org/jaic/articles/jaic39-03-003.html.

⁷⁰ Susan Anne Mathisen, "The use of Reemay[™] in wet cleaning" in *AIC Textile Specialty Group Postprints 23rd Annual Meeting St. Paul, Minnesota June 1995,* ed. Patricia Ewer and Beth McLaughlin, 64-68 (Washington: TSG, 1995).

⁷¹ Marion Kite, Roy Thompson and Aline Angus, "Materials and techniques: past and present" in *Conservation of Leather and related materials* ed. Marion Kite and Roy Thomson, 121-129. (Oxford: Butterworth Heinemann, 2006).

⁷² CTC, "Principles and Practice: Advanced Skills Adhesive Practical: Workshop notes" *Principles and Practice* adhesive workshop notes (Glasgow: Centre for Textile Conservation and Technical Art History, 2016), 1-25.

⁷³ The appropriate health and safety measures were taken to ensure the safety of all in the lab, see Appendix A

selected membrane, and was weighted with a glass weight, as is commonly done in textile conservation practice for adhesive reactivation. The glass weight measured 155 x 95 x 5 mm, and weighed 219 g.

The solvent dampened blotting paper was held for two time intervals, one minute and three minutes. The time of re-activation with solvent vapour is a factor that is generally determined by the conservator through testing, as the thickness of the object and support fabric as well as thickness of the adhesive film can influence the time needed for a strong bond to form.⁷⁴ The time intervals for this experiment were chosen as the author has found through her own testing of adhesives that one to three minute application times of solvent vapour 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; the change or movement in the level of soiling following any differentiation in the length of solvent exposure from what is recorded in this research paper can be predicted from the results gained from this experiment.

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.⁷⁵

⁷⁴ Irene F. Karsten and Jane Down, "The effect of adhesive concentration, reactivation time, and pressure on the peel strength of heat and solvent-reactivated Lascaux 360/498 HV bonds to silk" *ICOM Committee for Conservation 14th Triennial Meeting The Hague 12-16 September 2005: Preprints,* ed. Isabelle Verger (London: James & James, 2005), 927-935.

⁷⁵ Karsten and Down, "The effect of adhesive concentration, reactivation time, and pressure on the peel strength of heat and solvent-reactivated Lascaux 360/498 HV bonds to silk" 927.

3.7. Analysis of the samples

3.3.5 <u>Microscopy</u>

The samples were photographed under magnification using a Zeiss Stemi 2000-C stereomicroscope and Zen software, at four times magnification and twenty 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 x 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. This was 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.

3.3.6 <u>Image J</u>

ImageJ is an open access image processing software that was designed by the National Institute of Health for precise biomedical and scientific image analysis.⁷⁶ The 'particle analysis' function automatically counts the portions of an image that have been segmented from the background.⁷⁷ Although used mainly in medical and scientific industries, the software was considered useful for this research as it has been implemented in dust monitoring in the heritage sector,⁷⁸ and used for research at the CTC. 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 vapours, with the aim of calculating an average

http://www.nature.com/nmeth/journal/v9/n7/full/nmeth.2089.html

⁷⁶ Caroline A. Schneider, Wayne S. Rasband and Kevin W. Eliciri, "NIH Image to ImageJ: 25 years of image analysis" *Nature Methods*, 9 (2012): 671-675.

 ⁷⁷ "Particle Analysis", ImageJ, accessed 13August 2017, <u>https://imagej.net/Particle Analysis</u>
 ⁷⁸ Barry Knight, "Measuring particulates in historic buildings: A comparison of methodologies" in *IAP Copenhagen 2001, 4th meeting of the Indoor Air Pollution Working Group, presentation abstracts,* ed. Morten Ryhl-Svendsen, 57-67 (Copenhagen: The National Museum of Denmark, 2002).

number of particles and the percentage of the surface covered between the ten replicates of each of the eight experimental groups.

The images at four times magnification were analysed as they gave a good indication of the percentage of the surface of the silk fabric that was covered with the particulate soiling. The images of the samples were made 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 3). A scale was set to measure 171 pixels/1000 μ m, and particles measuring less than 80 μ m⁻² were filtered out as these were mostly introduced by the thresholding procedure.



Figure 3: thresehold and black and white image of one replicate during processing with ImageJ

3.3.7 <u>Fourier transform infrared spectroscopy (FTIR)</u>

Fourier transform infrared spectroscopy (FTIR) is an analytical technique commonly employed by heritage scientists to identify materials. The instrument shines a beam of infrared light onto the material sample causing molecules to vibrate. A spectrum is produced that charts the percentage of light absorbed by the sample at the exact wavelength of infrared light that has caused the molecules to vibrate. Characteristic molecular bonds will absorb light and vibrate at a particular region of the infrared light wavelength, and so their presence in the sample material will be known by the peaks and troughs produced on the spectrum.⁷⁹

The reference soiling materials and samples were analysed on a Perkin Elmer Spectrum One FTIR Spectrometer with a Universal Sampling Attenuated Total Reflectance accessory, and Spectrum software version 5.0.1. A diamond/thallium bromoiodide (C/KRS-5) ATR crystal with a penetration depth of up to 2µm was used, and an initial background scan of the uncovered crystal was taken. The spectra of the samples were taken in transmittance mode at a wavelength region of 4000-400 cm⁻¹, and an average of 16 scans under a clamping force of approximately 50 N, at a resolution of 8 cm⁻¹.⁸⁰ 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.

FTIR was chosen as the best possible analytical method to analyse the movement of the soiling on the silk samples as the beam of infrared light only penetrates approximately 2 μ m into the surface of the sample. This low penetration allows for any slight movement of the soiling material to be detected, and is seen as a change in the percentage of light absorbed on the spectrum; a factor that is particularly relevant for the movement of any oil as this is less likely to be detected by visual analysis. Figure 4 shows the spectrum of the olive oil used in the artificial soiling and a reference of the silk fabric used for the substrate of the samples. The peak present in the oil spectrum at 1744 cm⁻¹ is indicative

⁷⁹ Michele R. Derrick, Dusan Stulik, James M. Landry, *Scientific Tools for Conservation: Infrared Spectroscopy in Conservation Science*, (Los Angeles: J. Paul Getty Trust, 1999), 24-25.

⁸⁰ Margaret Smith, "Technical Art History: Making and Meaning, analytical information for MLitt dissertations" *Technical Art History* analytical techniques handout (Glasgow: Centre for Textile Conservation and Technical Art History, 2017), 1.
of carbonyl (C=O) group stretching, and is a characteristic peak for the existence of oil.⁸¹ As seen in the overlaid spectra, the carbonyl peak is absent from the spectrum of the silk control, making this peak ideal for the identification and monitoring of oil as part of the soiling mixture on the silk samples, before and after the application of solvent vapour.



Figure 4: spectra of the olive oil used in the creation of the artificial soiling material, and the silk used to create the samples. Note the absence of the characteristic carbonyl peak at 1744 cm-1 in the silk spectrum

The ten replicate samples from each of the allocated experimental groups were analysed 10 mm from the bottom left corner along the bottom edge, in transmittance mode, 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 spectra were analysed using Bio-Rad Laboratories KnowItAll® software, Windows version 10.0.15063. Each spectrum was viewed as absorbance mode, baseline corrected to zero, and the ten replicates were overlaid and averaged to produce one spectrum representative of the group (see figure 5).

⁸¹ George Socrates, *Infrared Characteristic Group Frequencies: Tables and Charts, second edition,* (Chichester, John Wiley & Sons Ltd, 1994), 80.



Figure 5: the ten replicates of the group G_1min_A overlaid and baseline corrected before being averaged

3.3.8 <u>Analysis by absorbance ratios</u>

The measurement of the absorbance value of a peak of unknown concentration against the absorbance value of a characteristic peak of known concentration or consistent intensity, which exhibits minimal interference from other aspects of the spectrum, can provide numerical data relating the intensity of the unknown peak to the sample as a whole.⁸² The ratio method of analysis was used in this research to compare the spectra of the test groups before solvent application to the spectra taken after solvent application, with the attempt to provide more accurate analysis of the changes occurring in the oil of the soiling mixture. This comparison between two peaks is possible due to the fact that the intensity of a peak on an IR spectrum has a direct relationship to the concentration of the molecule within the sample.⁸³

The intensity (I) of the characteristic oil peak (1744 cm⁻¹) was compared against the characteristic silk peak (1618 cm⁻¹) as the intensity of the silk peak is consistent

⁸² Paul Garside and Paul Wyeth, "Identification of Cellulosic Fibres by FTIR Spectroscopy: thread and single fibre analysis by attenuated total reflectance" *Studies in Conservation* vol.48, no. 4 (2003): 269-275.

⁸³ Derrick et al., *Infrared Spectroscopy in Conservation Science*, 123.

between the before and after spectra of each test group and is not affected by other material components within the sample. 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 were:

 R_1 = I_{1744}/I_{1618} before solvent application

 $R_2 = I_{1744}/I_{1618}$ after solvent application

Application of the absorbance ratios facilitated the compilation of semi quantitative data allowing for direct comparison of the results produced by the experimental variables.

3.8. Conclusion

The design of the experiment and analytical methods of light microscopy and ATR-FTIR used for this research paper were tailored specifically to achieve quantitative and qualitative results regarding the potential effects of solvent vapour on a mixture of organic and inorganic soiling material which are potentially found ingrained in historic textiles. The inclusion of the variables of solvent type, length of exposure to the solvent vapour, and type of barrier layer were incorporated into the experiment as aspects of the conservators' decision making process when considering the solvent re-activation of an adhesive treatment.

4. Results and discussion

4.1. Introduction

The execution of the experiment utilising the variables and analytical techniques outlined in the previous chapter have produced results which reveal the movement of the oil and particulate elements of the soiling material with the application of solvent vapour. This chapter illustrates and discusses the results, details of the successes and short comings of the methodological methods, highlighting areas that require further quantitative analysis, and aspects of the process that were unexpected at the beginning stage of the experimentation. The chapter concludes with the implications these results have on the preservation and chemical stability for a historic textile, and considerations for the conservator undertaking a solvent re-activated adhesive treatment.

4.2. The results of analysis using microscopy

Movement of large particulate soiling was observed during the application of the solvent vapour in the experimental process, as the barrier layer and silicone release paper used for each sample in each test group had noticeable amounts of dark particulate soiling on both surfaces after the removal of the solvent dampened blotting paper.

Visual analysis of the microscopy images of the ten replicate samples in each test group confirmed that movement of particulate soiling measuring approximately 50-200 μ m occurred on all of the replicates. This movement was clearly visible on the images taken at four times magnification, and the majority of the images at twenty times magnification. An example of the visible change of particulate soiling at twenty times magnification can be seen in figures 6 and 7, the front of one replicate from the test group R_1min_A, shown before and after the application of acetone vapour. This visible change appears to be lateral movement of the particulates; it is unclear from the images how much of the particulate soiling is moving vertically through the interstices of the silk. This result is similar to what was seen in all of the images at both four and twenty times magnification; the level of soiling did not drastically change, but the particulates move across the surface of the silk fabric. A small number of the images have been considered anomalies due to the fact that the paper window was not well aligned for the photography after the application of solvent vapour, and as a result the before and after images are not exact representations of each other and an accurate comparison cannot be made; a result exacerbated by the high magnification and resulting small visible area of the sample. 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 experimentation. The conclusion gained from the visual examination of the images at four times and twenty times magnification was that each test group displayed movement of the larger particulate soiling, however differentiation of the amount of soiling moved between each test group could not be conclusively determined from the visual examination.



Figure 6: replicate of the group R_1min_A before solvent application



Figure 7: replicate of the group R_1min_A after solvent application

As the observations of the changes to the soiling were largely subjective, ImageJ 'particle analysis' software was used to generate quantitative data on the changes of the particulate soiling occurring after the application of solvent vapour. ImageJ calculated figures 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. For this particular research paper the count of particulates and the percentage of the silk covered were considered the most relevant data to be analysed. What was discovered was that while the eye could detect change and movement of the larger particulates of soiling, the software recorded changes of particulate matter ranging in size from 80 μ m⁻² to the largest particulates, approximately 200 μ m. The software was used only on the images taken at four times magnification, as it was found that the images taken at twenty times magnification were not suitable for analysis as they were not entirely in even focus, however, they were visually analysed for comparison against the data gathered from ImageJ.

The data collected from each replicate of each test group was entered into an Excel spreadsheet and the mean values were calculated. The averages of the particle count and percentage of the silk covered 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 figures 8 and 9. The mean values depicted in the bar charts and the data collected from each replicate and used to calculate the mean values can be seen in Appendix B.



Figure 8: Average particle count of the front and back of the test groups, before and after the application of solvent vapour



Figure 9: average percentage of the surface area covered by particulate soiling, showing the front and back of the test groups, before and after the application of solvent vapour

Figure 8 shows the mean values of the particle count, and figure 9 the mean values for the average percentage covered for each test group, the results obtained from ImageJ. Standard deviation bars have been added to figure 8, however the standard deviation was greater than the mean values calculated for the percentage of silk covered, and as such could not be added to the figure 9. The standard deviation for each group displayed in figure 8 is large, particularly for the front of the silk samples. This is due to the large variance in the particle count data calculated for each replicate, a factor likely caused by an uneven application of the soiling mixture over the surface of each of the ten replicates during sample preparation. Although each of the replicates received the same amount of soiling, the mixture was unavoidably applied unevenly, which combined with the small 10 mm x 10 mm window used to photograph each sample, has resulted in a large variance in the particle count between the replicates. The experiment

would have benefitted 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.

An example of the uneven application of the soiling mixture on two replicates can be seen in figures 10 and 11 from the group R_1min_A, which were analysed with ImageJ and had initial average particle counts of 1750 and 1474 respectively.

This large variance in the standard deviation indicates that any differences between the mean numbers of particulate soiling recorded before and after the application of solvent vapour are not likely significant, due to the large variance in numerical data and the large overlap of the standard deviation bars.⁸⁴

Despite the fact that these results are not quantifiable, the experiment taken was a preliminary investigation and has highlighted the effect of solvent vapour on particulate soiling; a result that could be explored further in future research.

⁸⁴ "Graphing Resource: Using Error Bars in your Graphs", LabWrite Resources, North Carolina State University, <u>https://projects.ncsu.edu/labwrite/res/gt/gt-stat-home.html</u>



Figure 10: example of a replicate with an uneven application of the soiling material



Figure 11: example of a replicate with even soiling. This example is indicative of the majority of the silk sample replicates

A comparison of the results from the different test groups displayed in figures 8 and 9 indicate that there is no clear pattern to the amount or directional movement of the particulate soiling with the variables of length of exposure to the solvent vapour, type of solvent, and type of barrier layer. While an initial visual analysis of figures 8 and 9 suggest that there could be a difference in the particle count and percentage of the silk covered between these variables, it can be surmised that any result is a product of the large variance in the data gathered from each replicate by ImageJ.

4.3. Conclusion

Despite the large variance in data, the methods of using visual examination and automated quantitative analysis of the images at four and twenty times magnification have answered the research question: the application of the vapour of acetone and IDA through Gore-Tex® and Reemay® for the length of exposure of one and three minutes has resulted in movement of the particulate soiling.

Although the results gained from the analysis of the microscopy images cannot conclusively determine the amount of movement particulate soiling undergoes with the application of solvent vapour, and further testing would be needed to understand the significance of the results, the tests have shown the potential for the creation of more robust results in the analysis of particulate soiling on textiles with the application of ImageJ, much in the same manner that the software has been employed in the heritage sector for dust monitoring.

4.4. The results of FTIR-ATR analysis

The comparison of the overlaid spectra from the soiled samples before and after the application of solvent vapour show that there is a visible difference in the carbonyl group peak at 1744-1746 cm⁻¹, on both the front and back of the test samples, an example of which is seen in figures 12 and 13; the averaged front and back spectra from the test group R_3min_A, shown between in detail between 2000-1450 cm⁻¹. As this particular peak is characteristic for the presence of oil-as shown in the spectrum of oil and silk (see figure 4)-the change in absorption indicates that there is a change 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.⁸⁵ This result is seen in all of the test groups, with the variables of the time of exposure, barrier layer used, and specific solvent used causing small variances in the amount of change seen.

⁸⁵ Derrick et al., Infrared Spectroscopy in Conservation Science, 123.



Figure 12: detail of the front of the averaged spectra before and after the application of acetone to the samples through Reemay® for three minutes, shown from 2000 cm-1 to 1450 cm-1



Figure 13: detail of the back of the averaged spectra before and after the application of acetone to the samples through Reemay® for three minutes, shown from 2000 cm-1 to 1450 cm-1

While visual analysis of the spectra concluded that the amount of oil in the soiling material changed with the application of solvent vapour, the very slight differences in the change between the peak heights between the test groups meant that definitive comparisons between each group could not be made, and conclusions on the effect of the variables used in the experiment could not be drawn. The spectra were further analysed by the set of ratios calculated between the intensities of characteristic oil peak (1744 cm⁻¹), and the characteristic silk peak (1618 cm⁻¹), before and after the application of solvent vapour, for each test group. The averages of the ratios R₁ and R₂ can be seen in Table 1, and the mean values for the front and back of all test groups can be seen in figure 23. The results of these ratios show a downward trend in the intensity of the oil after solvent application.

Table 1: calculated averages of the ratios R_1 (I_{1744}/I_{1618} before solvent application) and R_2 (I_{1744}/I_{1618} after solvent application) for each test group

Front of silk samples

	R1	R2	
G_1min_IDA	0.45	0.35	
G_3min_IDA	0.46	0.37	
G_1min_A	0.44	0.29	
G_3min_A	0.46	0.25	
R_1min_IDA	0.42	0.36	
R_3min_IDA	0.43	0.39	
R_1min_A	0.42	0.34	
R_3min_A	0.47	0.35	

Back of silk samples

	R1	R2	
G_1min_IDA	0.35	0.32	
G_3min_IDA	0.37	0.33	
G_1min_A	0.35	0.23	
G_3min_A	0.37	0.22	
R_1min_IDA	0.35	0.30	
R_3min_IDA	0.35	0.33	
R_1min_A	0.32	0.26	
R_3min_A	0.36	0.28	

An example of the comparison between the effect of the two solvents can be seen in figures 14 and 15: details of the spectra which display the change in the carbonyl peak with the application of acetone and IDA vapour applied to the soiled samples through Gore-Tex® for one minute, and figures 16 and 17, showing details of the spectra of the soiled samples before and after the application of the two solvents through Gore-Tex®, for three minutes. Visual analysis of the spectra confirms the change in the intensity of the carbonyl peak that occurred with the use of acetone is greater than that with the use of IDA. The effect of the variation of length of exposure between tests using the same barrier layer and solvent, seen between figure 14 and 16, and 15 and 17, is slightly more challenging to compare with only a visual examination of the spectra, however this variable can be assessed with the results of the calculated ratios as seen in Table 1. The differences between R_1 and R_2 show that, for the example spectra given in figures 14/15 and 16/17, as well as the results of the test groups illustrated in Appendix C, the variation between three minutes of solvent exposure vary only slightly from the results of one minute of solvent exposure, and the application of acetone has overall produced a greater change of the intensity of the oil peak on the front and back of the samples in the test groups than the application of IDA, through both Gore-Tex® and Reemay® barrier layers.



Figure 14: detail of the averaged before and after spectra of acetone applied through Gore-Tex® *for one minute, shown from 2000cm-1 to 1450 cm-1*



Figure 15: detail of the before and after spectra of IDA applied through Gore-Tex® for one minute. A comparison of 14 and 15 show that the acetone has produced a larger change in the peak height



Figure 16: detail of the before and after spectra of acetone applied through Gore-Tex® *for three minutes*



Figure 17: detail of the before and after spectra of IDA applied through Gore-Tex® *for three minutes*

The visual analysis of the spectra, and comparison of the differences between R_1 and R_2 , from the front and back of the soiled samples show a change in the carbonyl peak

intensity on both sides of the samples, an example of such change is seen in figure 18 and 19: the front and back spectra of the test group R_1min_IDA. This change is seen in all test groups, through both Gore-Tex® and Reemay® barrier layers, and indicates that the application of solvent vapour by a poultice method produces more than just a surface change; this method allows the vapour to penetrate far enough into the fibres to cause a change in the level of oil throughout the entire thread, a result that was unexpected at the beginning stage of the experimentation.



Figure 18: detail of the before and after spectra of IDA applied through Reemay® for one minute



Figure 19: detail of the before and after spectra of IDA applied through Reemay® for one minute

Of particular interest in this research paper is the difference in peak height between the tests with a Gore-Tex® barrier layer and a Reemay® barrier layer, where a greater

change in the carbonyl peak, and therefore larger movement of the oil in the soiling mixture, was observed with the use of Gore-Tex® with both acetone and IDA. This result was unexpected as Gore-Tex® is marketed as a semi-permeable membrane and is often used in textile conservation, whilst a survey of the literature on the re-activation of adhesive treatments has shown that Reemay® is not such a commonly chosen material, and more often used for packing objects, making protective covers, and is used as a backing material in leather conservation.^{86, 87}

This large change in carbonyl peak of the spectra of the Gore-Tex® test groups could be explained by an observation that was made during the application of the solvent vapour. The PTFE side of the Gore-Tex® became very slightly oily in appearance with the application of IDA for both the one and three minute tests, but became noticeably oily during the tests with acetone, particularly the three minute test, where the exact shape of the blotter paper was noticeable on the Gore-Tex® after the application of the acetone vapour to only three replicates (see figure 20).



⁸⁶ Nancy Kerr, Linda Capjack and Robert Fedosejevs, "Ability of textile covers to protect artifacts from ultraviolet radiation" *Journal of the American Institute for Conservation* vol 39, no. 3, article 3, (2000): no page numbers, http://cool.conservation-us.org/jaic/articles/jaic39-03-003.html.
⁸⁷ Mary-Lou E. Florian, "The mechanisms of deterioration in leather" in *Conservation of Leather and related materials* edited by Marion Kite and Roy Thomson, 36-57. (Oxford: Butterworth Heinemann, 2006).

Figure 20: image of the piece of Gore-Tex \mathbb{B} used to apply acetone for three minutes. Note the oily square shaped stain in the centre of the Gore-Tex \mathbb{B}

This unexpected and noticeable change to the surface of the Gore-Tex® is due to the interaction between the acetone vapour and the PTFE layer. While the pores of the Gore-Tex® are able to maintain shape and integrity with the application of vapour from water and a number of organic solvents, acetone permanently alters the structure of the PTFE material, enlarging the pores.⁸⁸ This enlargement of the pores has led to a greater amount of acetone vapour to permeate the fibres, which subsequently has had a larger solubilising effect on the oil. Figure 21 shows the spectra of all of the PTFE side of the Gore-Tex® pieces used in the experimentation, all of which show a slight peak at 1744cm⁻¹, as indicated by the dashed line, indicative of oil. In contrast, no such characteristic peak is seen on the spectra of the Reemay® pieces used in the experimentation (figures 21 and 22).

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 permeable membrane.

⁸⁸ Personal communication, Dr. Mahesh Uttamlal, Instrumentation, Control and Analytical Sciences, Glasgow Caledonian University, 10 August 2017.



Figure 21: the spectra of the Gore-Tex® pieces used in the experimentation, noting the presence of a small peak at 1744 cm-1, indicating the oil from the soiled samples



Figure 22: the spectra of the Reemay® pieces used in the experimentation, which do not show the characteristic oil peak seen on the Gore-Tex® pieces

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.⁸⁹ IDA and acetone are solvents of polar and medium polarity^{90, 91}, respectively, while the soiling mixture is made of non-polar oil⁹² and 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"⁹³, the partially solubility of a substance in a solvent is possible due to the ability of the solvent to "swell" the solute,⁹⁴ and 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 reactivation may have an effect on the movement of the soiling. This has been surmised given the more volatile nature of acetone than IDA⁹⁵, however the confirmation of this would require further research.

Overall, the application of the vapour of acetone and IDA has caused a change in the oil of the soiling mixture applied to the silk replicates, the results of which are seen in figure 23, the mean values of the absorbance ratios for the front and back of all test groups. The addition of the standard deviation bars indicates that the change seen on the front of the samples is likely significant, due to the small variation in the data and lack of overlap of the standard deviation bars.⁹⁶ Further statistical analysis would be necessary to determine the significance of the change on the back of the samples, as a larger variance in data is seen.

⁸⁹ Mills and White, *The Organic Chemistry of Museum Objects*, 69.

⁹⁰ Museums and Galleries Commission, *Science for Conservators: Volume 2 Cleaning*, (Oxon: Routledge, 1992), 66-68.

⁹¹ Velson Horie, *Materials for Conservation: Organic Consolidants, Adhesives and Coatings, second edition,* (Oxon: Routledge, 2011), 67-70.

⁹² Mills and White, *The Organic Chemistry of Museum Objects*, 31-33.

⁹³ Mills and White, 69.

⁹⁴ John Burke, "Solubility Parameters: Theory and Application" *AIC The Book and Paper Group Annual, volume three* (1984): no page numbers. Accessed 13 August 2017. http://cool.conservation-us.org/coolaic/sg/bpg/annual/v03/bp03-04.html

⁹⁵ Horie, *Materials for Conservation*, 344-348.

⁹⁶ "Graphing Resource: Using Error Bars in your Graphs", LabWrite Resources, North Carolina State University, accessed 10 August 2017. <u>https://projects.ncsu.edu/labwrite/res/gt/gt-stat-home.html</u>



Figure 23: the mean values of the peak ratios for all of the test groups, showing the change between the front and the back of the samples before and after solvent vapour application

4.5. 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 the 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.^{97, 98} 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 are the hard, sharp edges which can be a source of constant friction against the fibres with the natural swelling of the capillaries as a result of fluctuating RH levels, causing mechanical damage, weakness and brittleness, and can eventually lead to the breaking of the thread.⁹⁹

Given the relatively short-term 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 one minute 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 an greasy textile problematic.¹⁰⁰ However, the use of solvent vapour, particularly acetone, could provide a stronger bond and therefore a better support for an area of damage, if the same decrease in oil results with the added layers of the adhesive film and support material. The intentional movement of oily ingrained soiling would require careful ethical

⁹⁷ Helen Lloyd and Katy Lithgow, "Physical agents of deterioration" in *The National Trust Manual of Housekeeping*, 55-67.

⁹⁸ Tímár-Balázsy and Eastop. *Chemical Principles of Textile Conservation*, 158-159.

⁹⁹ Mechthild Flury-Lemberg, *Textile Conservation and Research*, (Bern: Abegg-Stiftung, 1988),23.

¹⁰⁰ Irene F. Karsten and Jan Vuori, "Textiles" in *Adhesive Compendium for Conservation*, ed. Jane L. Down, 150-151, (Ottawa: Canadian Conservation Institute, 2015).

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.

4.6. Conclusion

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 results in changes to the amount of oil and particulate soiling present on silk samples, with the variables of time of exposure, barrier layer, and solvent type producing small differences in the results. This preliminary research merits further investigation into the significance of these measured changes, as a means of informing the decision making process for adhesive re-activation in the textile conservation profession, and furthering the knowledge on the potential damaging effects of solvents on historic textiles.

5. <u>Conclusion</u>

5.1. Summary of the research

This research paper aimed to investigate the effects of solvent vapour on ingrained soiling when applied to a textile in a poultice method for the re-activation of an adhesive film.

The reasoning for this research was based upon a lack of published literature focused on this very question, and the authors concern for the potential for solvent vapour to imbed already ingrained soiling farther into the textile fibres, exacerbating chemical and mechanical degradation.

To answer the research question, a tailored and focused methodology was created. An experiment was designed in a replicable manner which aimed to replicate the solvent re-activation of an adhesive via a poultice method on a soiled textile. The success of this experiment centred largely around the recreation of a type of complex soiling similar to what could be found on an historic textile. The characterisation of historic soiling was accomplished by a review of conservation and scientific journals, and the resulting mixture included organic and inorganic particulate soiling and oil, and was applied to replicates of silk habotai.

As the re-activation of an adhesive film is a process that is often influenced by the type and condition of the object, the strength of bond required, the thickness of the adhesive film and supporting fabric, and the conservators' own experience¹⁰¹, a number of variables were included in the experimental design. These variables were decided upon after a survey of literature of published and unpublished sources on adhesive re-activation by solvent vapour and by drawing upon the authors' own experience, and included the use of two different solvents, acetone and IDA; two different barrier layers, Gore-Tex® and Reemay®; and two different lengths of exposure to the vapour, one minute and three minutes.

Quantitative and qualitative methods of analysis were used to monitor the movement of the particulate soiling and oil. Light microscopy with the addition of ImageJ image analysis software was used to specifically monitor the visible particulate soiling, while

¹⁰¹ Karsten and Vuori, "Textiles" in *Adhesive Compendium for Conservation*, 149.

FTIR-ATR with the addition of calculated absorbance ratios was used to monitor the level of oil.

5.2. Results of the experimentation

Change and movement of the particulate soiling and oil was documented in all of the test groups.

- The particulate soiling was observed moving laterally across the silk replicates, and changes in the number of particulates and the percentage of the silk covered were recorded. The data calculated by ImageJ varied largely, meaning that the results cannot be considered statistically viable, however overall the results from the visual analysis of the images taken with microscopy and data collected by ImageJ confirmed changes in the level of particulate soiling, on the front and back of the samples from each test group.
- The level of oil monitored on the front and back of the test groups decreased with the application of solvent vapour, a result which was confirmed by visual analysis of the spectra and calculated absorbance ratios, however, further research is necessary to apply quantitative analysis to the absorbance ratios to determine the significance of the change in the oil.

Analysis of the results from each test group have suggested that there are slight variances between the application of the experimental variables.

- Acetone vapour produced a greater change in the level of particulate soiling and level of oil than the application of IDA vapour.
- The use of the two barrier layers and two lengths of exposure to the solvent vapour did not produce large differences in the data collected before and after the application of solvent vapour, however, it was noted in the results of FTIR-ATR analysis that the Reemay® barrier layer lessened the effect of the solvents, indicating it acted as the more effective barrier layer over the semi-permeable membrane, Gore-Tex®.

5.3. Further research

This research taken was a preliminary investigation into the effect of solvent vapour on soiled textiles, and the results showing movement of the soiling of the silk replicates warrant further research in order to fully understand the potential degradative factors of a solvent re-activated adhesive treatment.

- The results produced by FTIR-ATR require further quantitative analysis to determine the significance of the changes in the soiling material; a factor crucial to the understanding of the potential degradation of the soiled textile artefact. The data collected by ImageJ also requires further analysis to determine significance, however the images of the soiled replicates must be re-taken and re-analysed to capture a larger area of the silk surface to reduce the variance in data.
- Further analysis of the properties of the solvents used in adhesive re-activation are needed to understand why the soiling is affected by the vapour, specifically if solvent volatility has a significant effect. A clearer understanding of the properties of the solvents commonly used could aid the decision making process taken by the conservator when determining the best treatment for the object in question.
- The replication of the experiment with the addition of an adhesive coated support fabric is necessary to determine if the solvent vapour can permeate through the adhesive film to cause the same or less level of movement and change to the textile soiling. This result could aid in the decision to solvent reactivate an adhesive film, depending on the level of soiling in the textile, and could also become a factor in determining the potential for the creation of strong adhesive bonds on very greasy textiles.
- The experiment could be replicated to test the effectiveness of other barrier layers used in textile conservation, specifically Sympatex® (hydrophilic polyester membrane¹⁰²), a semi-permeable membrane used as an alternative for Gore-Tex®

¹⁰² "Care Instructions," Sympatex, accessed 17 August 2017, <u>http://www.sympatex.com/en/information/235/care</u>

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Appendix A: Risk assessment and COSHH forms
School/Service/Unit Unit Safety Coordinator/Supervisor: Centre for Textile Conservation and Technical Art History Unit Safety Coordinator/Supervisor: Describe the activity Cleaning FTIR equipment or work process. (iii. how kingh worken this is carried out and quantify substance(s) involved in the process. Public Name the substance(s) involved in the process. Methanol = 1-2 mult ref2 ust Public (Attach data sheels to this assessment) Methanol = 1-2 mult ref2 ust Public GHP Classification (state the category of danger)** Image: Classification (state the category of danger)** Image: Classification (state the category of danger)** Image: Classification (state the category of danger)** Image: Classification (state the category of danger)** Image: Classification (state the category of danger)** Image: Classification (state the category of danger)** Image: Classification (state the category of danger)** Image: Classification (state the category of danger)** Image: Classification (state the category of danger)** Image: Classification (state the category of danger)** Image: Classification (state the category of danger)** Image: Classification (state) Image: Classification (state the category of classification (state the category of classification (state) Image: Classification (state) Image: Classification	of Glasgow	COSHH Risk Ass	essment		Ref No:
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Long-term exposure level (8)	hrTWA):	Short-term exposure le	vel (15 mins):
- ,			
Vethanol – 200ppm		Methanol - 250ppmpm	
Solvent will be used in ver	v small quantities and		9 C
herefore present a much I	ower risk than the WELs	-	
above suggest (see descri	ption of activity)		
What are the risks to Health	from the process /tasks? ()	ook at the H and P and as an th	no data abaati
That are the hole to Health	nom the process reases! (A	ook at the H and P codes on th	e data sheet)
/lethanol – may damage to a	airways and internal organs	and central nervous sys	tem.
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Specific target organ toxicity,	single exposure (Category 1- T	arget Organs -;Optic nerve)	
specific target organ toxicity,	repeated exposure (Category	1- Target Organs - Kidney, Liver, splee	n, Blood)
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What Persona	al Protective	Equipment is used ?	(state type and	standaro)	
	NE TRACCION					
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Respirator	Decant solven extraction	t into smaller containers u	inder Eye v	vear	Plastic goggles	
Gloves	Wear nitrile glo	ves	Over		Labcoat	í.
Eootwear	Wear closed to	shoes	Other			
Assessment	of the process	s/task	Other		an Marthall Strange	
Only use sma Carry out task Always replac Use a suitable	Il quantities o ks under extra ce lids on solv e carrier if tra	of chemicals. action as outlined abo rent bottles. nsporting large bottle	ove. es of solvents	2	đ	
Now rate the	overall rating	with the controls you	ı have listed		all the Carl	
High		Med	ium]	Low	\checkmark
list the new o	ontrols that r	eed to be in place to	give adequa	te cont	ol (consider the need fo	or monitoring as well as
Final check: H yes	las the risk ra	ting been reduced to Note any monitori	o as low as p ng that may	ossible be need	with the new contro	ls?
Emergency P	lans and proc	edures (this will apply to	o carcinogens,	mutagens	s or similar health risks)	an ang ang ang ang ang ang ang ang ang a
Are plans in p	lace to deal v	vith spillages or eme	rgencies?		Yes 🗸 M	N/a
Refer to any f	irst aid emerg	ency that should be	noted in this	assess	ment	
Call Poison C	entre or doct	or if exposed.				
Skin Contact - Ingestion - Rin Inhalation -Mo	Wash off imn se mouth with ve to fresh air	nediately with plenty of water.	f water.			
Has waste dis	posal been c	onsidered and estab	lished		Yes 🗸 N	l/a
Use non-chlor Loa USE/DISPO	inated waste SAL of METH	disposal ลุ <i>No</i> L			A	
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Assessed by: Keira Miller & Aisling Macken Approval by:	Date: (.). 2017 Date:	F	Review Date:			
Kana Than as		1.6 1-	,			

Sign off Sheet of Understanding

I have been taken through the COSHH assessment for the listed task/process and understand the safety controls and responsibilities to ensure I work safely.

NAME	Signature	DATE
KEIRA MILLER	K.C. Miller	1.6.17
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ootwear Assessment o	of the process/task		Other	State of the second		22022
List the summ Use small wear the	ary of controls alread quanthes of appropriate	ly in place and identify accord and PPE and work	iDA under a	extraction.		
Now rate the	overall rating with t	he controls you have	e listed			/
High		Medium		Low		
ist the new o	controls that need to	be in place to give	adequate con	trol (consider the need	d for monitoring	as well as
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Assessed by: Asling Marker	Date: Assy Mark	Review Date:	
Karetha	p 23.6.	17	

Sign off Sheet of Understanding

I have been taken through the COSHH assessment for the listed task/process and understand the safety controls and responsibilities to ensure I work safely.

NAME	Signature	DATE

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Appendix B: raw data from ImageJ analysis

Test	Average particle count		Average surfact covered (%)	ce area
Gore-Tex 1	Front before	Front after	Front before	Front after
minute acetone	1736.7	1523.1	2.99	2.38
	Back before	Back after	Back before	Back after
	761.7	985.9	1.22	1.65
Gore-Tex 3	Front before	Front after	Front before	Front after
minutes acetone	1643	1676.9	2.56	2.44
	Back before	Back after	Back before	Back after
	1029.5	1041.8	1.70	1.52
Gore-Tex 1	Front before	Front after	Front before	Front after
minute IDA	1759.3	1593.6	2.77	2.35
	Back before	Back after	Back before	Back after
	930.3	1037.7	1.49	1.56
Gore-Tex 3	Front before	Front after	Front before	Front after
minutes IDA	1684.9	1633.3	2.59	2.40
	Back before	Back after	Back before	Back after
	906.8	990.2	1.56	1.49
Reemay 1	Front before	Front after	Front before	Front after
minute acetone	1441.8	1710.9	2.52	2.88
	Back before	Back after	Back before	Back after
	946	883.6	1.82	1.47
Reemay 3	Front before	Front after	Front before	Front after
minutes acetone	1822.6	1739.6	3.0	2.71
	Back before	Back after	Back before	Back after
	929.1	1007.7	1.44	1.48
Reemay 1	Front before	Front after	Front before	Front after
minute IDA	1234.5	1431.9	1.91	2.01
	Back before	Back after	Back before	Back after
	779.4	882.1	1.19	1.27
Reemay 3	Front before	Front after	Front before	Front after
minutes IDA	1517.7	1490.7	2.17	2.07
	Back before	Back after	Back before	Back after
	1087.9	1141.6	1.81	1.71

Mean values of particle count and percentage of the silk covered

Data gathered for each replicate used in the calculation of the mean values

Gore-Tex 1 minute acetone

	Particle		Average particle	% Area
Sample	count	Total area	size	covered
01_back before	621	961586.734	1548.449	0.934
04_back before	567	904941.206	1596.016	0.87
15_back before	1120	1863423.168	1663.771	1.747
28_back before	767	1312590.827	1711.331	1.227
40_back before	739	1048586.339	1418.926	0.983
46_back before	803	1556975.821	1938.949	1.492
52_back before	552	979224.465	1773.957	0.917
53_back before	925	1384562.025	1496.824	1.313
54_back before	785	1451942.13	1849.608	1.364
72_back before	738	1467796.274	1988.884	1.356
Average	761.7	1293162.899	1698.6715	1.2203

Sample	Particle Count	Total area	Average particle size	% Area covered
01_back_after	843	1398005.017	1658.369	1.346
04_back_after	849	1403454.882	1653.068	1.399
15_back_after	1190	2115768.26	1777.957	2.094
28_back_after	1235	2444213.238	1979.12	2.354
40_back_after	942	1265424.759	1343.338	1.227
46_back_after	1227	2594431.235	2114.451	2.495
52_back_after	1248	2204749.633	1766.626	2.131
53_back_after	1007	1316158.009	1307.009	1.272
54_back_after	576	1088651.077	1890.019	1.021
72_back_after	742	1175914.914	1584.791	1.136
Average	985.9	1700677.102	1707.4748	1.6475

Sample	Particle count	Total area	Average particle size	% Area covered
01_front before	2287	4962016.12	2169.662	4.723
04_front before	1237	1953527.543	1579.246	1.829
15_front before	2302	3946426.205	1714.347	3.814
28_front before	1088	1907880.817	1753.567	1.807
40_front before	1778	2355991.542	1325.08	2.277
46_front before	2828	5918152.974	2092.699	5.54
52_front before	1351	2380103.049	1761.734	2.237
53_front before	2221	4173437.706	1879.08	3.928

54_front before	1040	1688565.195	1623.62	1.61
72_front before	1235	2267043.196	1835.663	2.106
Average	1736.7	3155314.435	1773.4698	2.9871

	Particle		Average particle	% Area
Sample	count	Total area	size	covered
01_front_after	1851	3467465.973	1873.293	3.317
04_front_after	1086	1883769.323	1734.594	1.781
15_front_after	1084	1400878.581	1292.323	1.356
28_front_after	1024	1555555.55	1519.097	1.468
40_front_after	1496	1883174.787	1258.807	1.811
46_front_after	1490	2301492.929	1544.626	2.133
52_front_after	2448	4464592.407	1823.771	4.221
53_front_after	1578	2546967.887	1614.048	2.433
54_front_after	1319	2143248.772	1624.904	2.023
72_front_after	1855	3419639.298	1843.471	3.191
Average	1523.1	2506678.551	1612.8934	2.3734

Gore-Tex 3 minutes acetone

	Particle		Average	% Area
Sample	count	Total area	size	covered
05_back before	1013	1419143.872	1400.932	1.376
07_back before	980	2012088.784	2053.152	1.893
10_back before	1392	2276060.235	1635.101	2.167
33_back before	1273	2238175.445	1758.19	2.167
37_back before	698	1443519.618	2068.08	1.354
50_back before	971	1773087.592	1826.043	1.636
64_back before	1066	1546307.304	1450.57	1.455
65_back before	693	1045877.922	1509.203	0.969
70_back before	1128	2054465.583	1821.335	1.936
77_back before	1081	2100079.266	1942.719	2.023
Average	1029.5	1790880.562	1746.5325	1.6976

Sample	Particle count	Total area	Average particle size	% Area
05_back_after	839	1092482.493	1302.125	1.046
07_back_after	877	1428689.384	1629.064	1.335
10_back_after	1353	1924461.617	1422.366	1.832
33_back_after	1388	2227705.112	1604.975	2.079
37_back_after	797	1166435.457	1463.533	1.111
50_back_after	829	1123992.6	1355.841	1.077
64_back_after	1299	1855727.307	1428.581	1.788
65_back_after	1193	1822202.401	1527.412	1.738
70_back_after	963	1733353.149	1799.951	1.687
77_back_after	880	1583267.271	1799.167	1.505
Average	1041.8	1595831.679	1533.3015	1.5198

Sample	Particle count	Total area	Average particle size	% Area covered
05_front before	815	977275.726	1199.111	0.93
07_front before	1232	2454683.579	1992.438	2.337
10_front before	1667	2425848.848	1455.218	2.282
33_front before	1693	2887435.585	1705.514	2.781
37_front before	1601	2635982.295	1646.46	2.492
50_front before	2511	4493427.133	1789.497	4.265
64_front before	1539	2101334.38	1365.389	2.014
65_front before	1296	2508059.188	1935.231	2.425
70_front before	1479	2517604.7	1702.234	2.322

77_front before	2597	3948474.035	1520.398	3.753
Average	1643	2695012.547	1631.149	2.5601

Sample	Particle count	Total area	Average particle size	% Area covered
05_front_after	1598	2036860.875	1274.631	1.945
07_front_after	928	1956665.34	2108.476	1.825
10_front_after	1864	2374554.098	1273.902	2.256
33_front_after	2058	2898896.808	1408.599	2.719
37_front_after	1617	2888063.149	1786.063	2.757
50_front_after	1308	1900284.048	1452.817	1.785
64_front_after	2382	3323919.928	1395.432	3.207
65_front_after	1692	2970868.012	1755.832	2.717
70_front_after	1073	1883802.345	1755.641	1.786
77_front_after	2249	3737878.151	1662.018	3.437
Average	1676.9	2597179.275	1587.3411	2.4434

Gore-Tex 1minute IDA

	Deutiale		Average	0/ Аног
C	Particle	T - 4 - 1	particle	% Area
Sample	count	l otal area	size	coverea
13_back before	921	1521304.004	1651.796	1.451
14_back before	1071	2030387.099	1895.786	1.894
16_back before	873	1372968.688	1572.702	1.283
18_back before	949	1247192.49	1314.218	1.203
21_back before	882	1301096.573	1475.166	1.262
43_back before	1181	1891300.04	1601.439	1.8
62_back before	1101	2010404.279	1825.98	1.842
67_back before	711	1131060.904	1590.803	1.068
68_back before	985	1863258.031	1891.633	1.724
75_back before	629	1518991.936	2414.932	1.418
Average	930.3	1588796.404	1723.4455	1.4945

	Particle		Average particle	% Area
Sample	count	Total area	size	covered
13_back_after	913	1373827.453	1504.74	1.273
14_back_after	915	1558792.442	1703.598	1.501
16_back_after	1238	1984839.475	1603.263	1.896
18_back_after	1151	1622836.566	1409.936	1.574
21_back_after	1130	1684964.984	1491.119	1.62
43_back_after	875	1483947.679	1695.94	1.434
62_back_after	1170	1868707.884	1597.186	1.812
67_back_after	1120	1699431.886	1517.35	1.626
68_back_after	997	1753996.564	1759.274	1.636
75_back_after	868	1329832.208	1532.065	1.253
Average	1037.7	1636117.714	1581.4471	1.5625

Sample	Particle count	Total area	Average particle size	% Area covered
13_front before	2304	3728993.257	1618.487	3.55
14_front before	1016	1923503.769	1893.212	1.84
16_front before	1445	2522294.888	1745.533	2.422
18_front before	3193	4396617.766	1376.955	4.186
21_front before	1086	1711256.432	1575.743	1.659
43_front before	1497	2328643.151	1555.54	2.194
62_front before	1720	3404016.375	1979.079	3.176
67_front before	980	1780915.583	1817.261	1.653
68_front before	2228	3616792.177	1623.336	3.38

75_front before	2124	3922843.173	1846.913	3.674
Average	1759.3	2933587.657	1703.2059	2.7734

Sample	Particle count	Total area	Average particle size	% Area covered
13_front_after	1522	1920564.132	1261.869	1.853
14_front_after	1364	2271634.297	1665.421	2.13
16_front_after	1598	2272228.825	1421.92	2.12
18_front_after	1615	2000924.822	1238.963	1.882
21_front_after	2247	3102094.065	1380.549	3.055
43_front_after	2060	3875809.226	1881.461	3.695
62_front_after	1762	3512848.455	1993.671	3.306
67_front_after	991	1651473.111	1666.471	1.541
68_front_after	1179	1641200.947	1392.028	1.589
75_front_after	1598	2512716.345	1572.413	2.364
Average	1593.6	2476149.423	1547.4766	2.3535

<u>Gore-Tex 3 minutes IDA</u>

			Average	
	Particle	m - 1	particle	% Area
Sample	count	Total area	size	covered
08_back before	609	939350.428	1542.447	0.894
09_back before	776	1417709.404	1826.945	1.312
24_back before	1764	3972957.291	2252.243	3.703
27_back before	728	1168752.14	1605.429	1.126
30_back before	657	912854.701	1389.429	0.839
45_back before	1043	1854871.797	1778.401	1.723
49_back before	688	1500615.379	2181.127	1.378
60_back before	847	1476034.19	1742.661	1.417
66_back before	804	1119076.929	1391.887	1.061
80_back before	1152	2346564.099	2036.948	2.173
Average	906.8	1670878.636	1774.7517	1.5626

Sample	Particle count	Total area	Average particle size	% Area covered
08_back_after	1054	1683623.931	1597.366	1.575
09_back_after	793	1420957.263	1791.875	1.313
24_back_after	846	1347965.812	1593.34	1.25
27_back_after	786	1414632.48	1799.787	1.321
30_back_after	957	1303726.498	1362.306	1.211
45_back_after	1148	1856752.136	1617.38	1.716
49_back_after	980	1614666.664	1647.619	1.492
60_back_after	1636	2957880.336	1807.995	2.757
66_back_after	981	1399316.242	1426.418	1.311
80_back_after	721	1070188.036	1484.311	0.998
Average	990.2	1606970.94	1612.8397	1.4944

Sample	Particle count	Total area	Average particle size	% Area covered
08_front before	1558	2111452.991	1355.233	1.999
09_front before	1987	3877367.524	1951.368	3.577
24_front before	1558	2703145.294	1735.01	2.55
27_front before	819	1770974.357	2162.362	1.645
30_front before	1538	2045196.586	1329.777	1.878
45_front before	2972	4599042.74	1547.457	4.353
49_front before	1419	1949504.273	1373.858	1.808
60_front before	1883	3317948.714	1762.055	3.087
66_front before	1234	1840615.391	1491.585	1.676

80_front before	1881	3714564.1	1974.782	3.343
Average	1684.9	2792981.197	1668.3487	2.5916

Sample	Particle count	Total area	Average particle size	% Area covered
08_front_after	1080	1424854.7	1319.31	1.322
09_front_after	1084	1474119.657	1359.889	1.372
24_front_after	1644	2519794.878	1532.722	2.333
27_front_after	1479	2625982.906	1775.512	2.431
30_front_after	1323	1876820.516	1418.61	1.732
45_front_after	3478	6147042.736	1767.407	5.559
49_front_after	1616	2490290.597	1541.021	2.321
60_front_after	1580	2633025.652	1666.472	2.425
66_front_after	1495	2169025.639	1450.853	2.012
80_front_after	1554	2636205.125	1696.4	2.508
Average	1633.3	2599716.241	1552.8196	2.4015

Reemay 1 minute acetone

	Particle		Average particle	% Area
Sample	count	Total area	size	covered
06_back before	1000	1567145.298	1567.145	1.436
11_back before	715	1346461.533	1883.163	1.22
17_back before	902	1593162.39	1766.255	1.453
36_back before	902	1410290.596	1563.515	1.319
39_back before	679	1019282.052	1501.152	0.947
48_back before	1346	2826324.785	2099.796	2.621
56_back before	1013	3622940.234	3576.446	3.356
61_back before	896	2690188.023	3002.442	2.507
69_back before	838	1426529.915	1702.303	1.277
76_back before	1169	2277094.015	1947.899	2.088
Average	946	1977941.884	2061.0116	1.8224

Sample	Particle count	Total area	Average particle size	% Area covered
06_back_after	978	1316820.513	1346.442	1.221
11_back_after	1003	1709025.641	1703.914	1.551
17_back_after	925	1441435.896	1558.309	1.35
36_back_after	949	1389606.834	1464.285	1.352
39_back_after	1082	1619418.807	1496.69	1.507
48_back_after	796	1276239.319	1603.316	1.192
56_back_after	859	2626700.855	3057.859	2.432
61_back_after	806	2298666.697	2851.944	2.11
69_back_after	544	852991.452	1567.999	0.81
76_back_after	894	1264957.264	1414.941	1.168
Average	883.6	1579586.328	1806.5699	1.4693

Sample	Particle count	Total area	Average particle size	% Area covered
06_front before	1750	2529606.841	1445.49	2.354
11_front before	1283	2377743.591	1853.269	2.156
17_front before	1294	2391213.674	1847.924	2.23
36_front before	1873	3177641.029	1696.552	2.959
39_front before	1239	1896991.452	1531.067	1.733
48_front before	1253	2622324.789	2092.837	2.428
56_front before	1474	5784820.462	3924.573	5.174
61_front before	1385	2366324.79	1708.538	2.154
69_front before	1300	2226153.848	1712.426	2.045

76_front before	1567	2117811.964	1351.507	1.988
Average	1441.8	2749063.244	1916.4183	2.5221

	Particle		Average particle	% Area
Sample	count	Total area	size	covered
06_front_after	1886	2632923.078	1396.036	2.393
11_front_after	1673	2920444.444	1745.633	2.681
17_front_after	1452	2587179.49	1781.804	2.384
36_front_after	2160	3264068.373	1511.143	3.012
39_front_after	1901	2865811.961	1507.529	2.663
48_front_after	1854	3383829.063	1825.151	3.181
56_front_after	1684	6719623.862	3990.275	6.047
61_front_after	1078	2023794.865	1877.361	1.932
69_front_after	1583	2541025.643	1605.196	2.258
76_front_after	1838	2487111.114	1353.162	2.242
Average	1710.9	3142581.189	1859.329	2.8793

Reemay 3 minutes acetone

	Particle		Average particle	% Area
Sample	count	Total area	size	covered
02_back before	1051	1842974.363	1753.544	1.67
12_back before	800	1410598.284	1763.248	1.269
29_back before	992	1891076.926	1906.328	1.702
31_back before	920	1676205.129	1821.962	1.541
32_back before	753	1077333.331	1430.722	0.983
51_back before	717	1590495.728	2218.265	1.436
57_back before	824	1354632.477	1643.971	1.225
58_back before	1031	1692376.067	1641.49	1.549
71_back before	1271	1976512.823	1555.085	1.743
78_back before	932	1417606.84	1521.037	1.313
Average	929.1	1592981.197	1725.5652	1.4431

	Particle		Average particle	% Area
Sample	count	Total area	size	covered
02_back_after	824	1269504.272	1540.661	1.171
12_back_after	518	1275623.929	2462.594	1.131
29_back_after	1223	1854871.789	1516.657	1.67
31_back_after	1041	1726495.727	1658.497	1.559
32_back_after	1395	2217846.156	1589.854	1.969
51_back_after	728	1580854.697	2171.504	1.42
57_back_after	898	1137675.214	1266.899	1.044
58_back_after	1154	2056615.39	1782.162	1.892
71_back_after	1313	1961128.208	1493.624	1.78
78_back_after	983	1255897.438	1277.617	1.125
Average	1007.7	1633651.282	1676.0069	1.4761

Sample	Particle count	Total area	Average particle size	% Area covered
02_front before	2801	5282598.298	1885.969	4.908
12_front before	1366	3422666.657	2505.612	3.021
29_front before	1632	2827282.056	1732.403	2.578
31_front before	930	2225948.72	2393.493	1.996
32_front before	2078	2878222.225	1385.093	2.556
51_front before	999	2065435.896	2067.503	1.868
57_front before	1657	3145572.647	1898.354	2.799
58_front before	2044	3182632.478	1557.061	2.87
71_front before	2320	3806769.228	1640.849	3.358

78_front before	2399	4440239.317	1850.871	3.931
Average	1822.6	3327736.752	1891.7208	2.9885

Sample	Particle count	Total area	Average particle size	% Area covered
02_front_after	1659	3250461.544	1959.29	2.897
12_front_after	1084	2243965.818	2070.079	2.022
29_front_after	1720	3235213.674	1880.938	2.855
31_front_after	1577	3655179.49	2317.806	3.279
32_front_after	2010	2600273.504	1293.668	2.324
51_front_after	1580	3200786.321	2025.814	2.866
57_front_after	1567	2516820.514	1606.139	2.231
58_front_after	1818	2706153.852	1488.533	2.436
71_front_after	2262	3871829.06	1711.684	3.445
78_front_after	2119	3041641.031	1435.413	2.71
Average	1739.6	3032232.481	1778.9364	2.7065

Reemay 1 minute IDA

	Doutialo		Average	0/ Аног
Sample	count	Total area	size	% Area covered
19_back before	643	978905.983	1522.404	0.893
20_back before	596	927589.743	1556.359	0.852
22_back before	743	1556102.567	2094.351	1.41
25_back before	723	973094.021	1345.912	0.896
26_back before	952	1756478.637	1845.041	1.657
44_back before	1090	1782495.728	1635.317	1.631
47_back before	1119	1806358.979	1614.262	1.653
59_back before	605	1151521.363	1903.341	1.055
63_back before	806	1275350.431	1582.321	1.129
73_back before	517	870222.219	1683.215	0.766
Average	779.4	1307811.967	1678.2523	1.1942

	Particle		Average particle	% Area
Sample	count	Total area	size	covered
19_back_after	1005	1387692.307	1380.788	1.302
20_back_after	792	1251623.934	1580.333	1.161
22_back_after	898	1909094.027	2125.94	1.77
25_back_after	1095	1479384.621	1351.036	1.318
26_back_after	901	1388991.456	1541.611	1.258
44_back_after	969	1629299.147	1681.423	1.478
47_back_after	938	1290461.537	1375.759	1.173
59_back_after	532	712478.632	1339.246	0.656
63_back_after	1044	1697025.643	1625.503	1.522
73_back_after	647	1192341.879	1842.878	1.073
Average	882.1	1393839.318	1584.4517	1.2711

Sample	Particle count	Total area	Average particle size	% Area covered
19_front before	1104	1822564.099	1650.873	1.764
20_front before	1081	1875350.424	1734.829	1.745
22_front before	640	1363111.104	2129.861	1.256
25_front before	2102	2963965.813	1410.069	2.739
26_front before	1297	2296581.189	1770.687	2.094
44_front before	1158	1636170.934	1412.928	1.51
47_front before	1272	2279282.052	1791.888	2.061
59_front before	907	1938256.407	2136.997	1.721
63_front before	1602	2416786.326	1508.606	2.172

73_front before	1182	2331213.676	1972.262	2.05
Average	1234.5	2092328.202	1751.9	1.9112

Sample	Particle count	Total area	Average particle size	% Area covered
19 front after	1878	2282188.039	1215.223	2.057
20_front_after	1481	2444410.248	1650.513	2.207
22_front_after	844	1645504.272	1949.65	1.478
25_front_after	2479	3087179.483	1245.333	2.83
26_front_after	1422	2456102.564	1727.217	2.266
44_front_after	1435	1869435.904	1302.743	1.723
47_front_after	1706	3004341.882	1761.044	2.8
59_front_after	1131	1851555.556	1637.096	1.661
63_front_after	1099	1658324.787	1508.94	1.488
73_front_after	844	1729948.716	2049.702	1.567
Average	1431.9	2202899.145	1604.7461	2.0077

Reemay 3 minutes IDA

·	Particle		Average particle	% Area
Sample	count	Total area	size	covered
03_back before	1734	3293252.63	1899.223	3.074
23_back before	1136	1942888.403	1710.289	1.771
34_back before	1638	2818850.245	1720.91	2.523
35_back before	943	1492151.426	1582.345	1.353
38_back before	670	1093225.263	1631.679	0.974
41_back before	1183	2254642.445	1905.869	2.066
42_back before	909	1907800.684	2098.791	1.772
55_back before	878	1549057.824	1764.303	1.388
74_back before	1123	2253616.489	2006.782	2.037
79_back before	665	1210560.513	1820.392	1.095
Average	1087.9	1981604.592	1814.0583	1.8053

Sample	Particle count	Total area	Average particle size	% Area covered
03_back_after	1010	1564002.59	1548.517	1.457
23_back_after	1435	2699736.669	1881.35	2.432
34_back_after	1843	3157347.55	1713.157	2.864
35_back_after	839	1102869.258	1314.504	1.014
38_back_after	1301	2034403.749	1563.723	1.833
41_back_after	1191	2004172.218	1682.764	1.859
42_back_after	935	1923942.403	2057.692	1.713
55_back_after	844	1333675.316	1580.184	1.202
74_back_after	1290	1986696.753	1540.075	1.776
79_back_after	728	1034540.539	1421.072	0.939
Average	1141.6	1884138.705	1630.3038	1.7089

Sample	Particle count	Total area	Average particle size	% Area covered
03_front before	1455	2430081.042	1670.159	2.223
23_front before	1725	3051400.43	1768.928	2.736
34_front before	2000	2907253.506	1453.627	2.589
35_front before	1606	2373961.213	1478.183	2.128
38_front before	1761	2506754.209	1423.483	2.251
41_front before	1315	2294039.192	1744.516	2
42_front before	1415	2796826.368	1976.556	2.512
55_front before	794	1486406.07	1872.048	1.313
74_front before	1405	2103211.24	1496.948	1.876

79_front before	1701	2309394.335	1357.669	2.084
Average	1517.7	2425932.761	1624.2117	2.1712

Sample	Particle count	Total area	Average particle size	% Area covered
03_front_after	1531	2570466.131	1678.946	2.286
23_front_after	1377	2084196.844	1513.578	1.936
34_front_after	1645	2424301.49	1473.74	2.181
35_front_after	1997	3055196.465	1529.893	2.726
38_front_after	1046	1543859.642	1475.965	1.346
41_front_after	1767	2956978.21	1673.446	2.678
42_front_after	1349	2067644.741	1532.724	1.876
55_front_after	1045	1460722.955	1397.821	1.321
74_front_after	1685	2679969.901	1590.487	2.399
79_front_after	1465	2202865.831	1503.663	1.962
Average	1490.7	2304620.221	1537.0263	2.0711



Appendix C: FTIR spectra of front and back of test groups, before and after solvent vapour application, showing overall spectra and a detail between 2000-1450 cm⁻¹




























