Benner, Julie (2012) *Investigating the potential of decamethylcyclopentasiloxane (D5) as an alternative solvent for textile conservation cleaning*. [MPhil.]

INVESTIGATING THE POTENTIAL OF DECAMETHYLCYCLOPENTASILOXANE (D5) AS AN ALTERNATIVE SOLVENT FOR TEXTILE CONSERVATION CLEANING

Submitted in partial fulfillment of the requirements for the
Degree of Master of Philosophy in Textile Conservation
in the School of Culture and Creative Arts,
University of Glasgow, 24 August 2012.

ABSTRACT
(236 Words)

Concerns about the health and environmental impacts of some solvents used in textile conservation have signaled the need for more environmentally friendly alternatives. At the same time, “green” cleaning solvents have begun to be developed by the professional dry cleaning industry. One of these alternative solvents, a cyclic silicon-based liquid, decamethylcyclopentasiloxane (D5), may have potential for use in textile conservation, however there previously have been no studies to show how its use may impact textile artifacts. In this study, the “green” profile of D5 was reviewed, along with a look at it structure, properties and potential for solubility. A series of experiments was performed to test the effects of D5 on textiles and to examine its soil removal performance. The samples used in testing were soiled and unsoiled new cotton and wool fabrics, some of which were artificially aged. Analysis of the effect of D5 on textile substrates employed Attentuated Total Reflectance- Fourier Transform Infrared (ATR-FTIR) spectroscopy, tensile strength tests, and scanning electron microscopy (SEM). Soil removal tests were analyzed using colorimetry and ATR-FTIR. In the results of the analysis, no appreciable difference in the condition and composition of treated and untreated samples could be detected. D5 was shown to have significant effect on nonpolar soiling. Assessment of the overall results suggests that there is potential for use of D5 within the textile conservation field, however limitations of the trials indicate a need for more research.

This paper is written in American English.

Word Count: 18,486

ACKNOWLEDGEMENTS

The author would like to acknowledge the help of several people, without which this dissertation would not be possible. First I would like to offer tremendous thanks to the Textile Conservation Centre Foundation, whose generous bursary funded my studies on the MPhil in Textile Conservation course, culminating in this research.

My gratitude goes out to my supervisor, course convener Frances Lennard, for helping to inspire me to explore this topic, shepherding me through various false starts, and for constant support and feedback in the conception and execution of the research and writing. Other principals at the Centre for Textile Conservation and Technical Art History who were unceasingly generous with their time in regard to experiment design and planning, securing resources and materials, and providing indispensable moral support, were tutors Sarah Foskett, Karen Thompson, and Dr. Anita Quye, who was additionally helpful on the topic of theory and spectral analysis.

Thanks are due to ICON intern Dr. Margaret Smith for her patient instruction on the use of analytical equipment, data collection, and sample preparation. Instruction on tensile testing was ably provided by Professor Liz Tanner of the University of Glasgow School of Engineering. Peter Chung, from the School of Geographical and Earth Sciences, generously offered time and expertise on the topic of SEM analysis.

Materials could not have been acquired under the time constraints of this study without the help of Ailsa Boyd in the History of Art and John Byrne from Materials Research Products, LTD.

Several figures from the dry cleaning industry made contributions to this project: Everett Childers gave me the inside scoop on the alternative solvents used in the dry cleaning trade and led me to all the right contacts. The experimental phase of the research would not have been possible without D5 solvent generously provided by GreenEarth® president Tim Maxwell, who, along with Karen Maxwell, were so enthusiastic and went above and beyond to make themselves available for questions. Thanks to Jim Douglas for putting me in touch with Doris Easley, and to Mrs. Easley, for imparting her experiences with GreenEarth® and her remarkable career.

I also was helped by many people in the Chicagoland area, including Joseph Barabe at the McCrone™ Group, who so graciously went out of his way to make the SEM analysis included in this study a reality. Acknowledgement is also due to Dr. Kathleen Martin for sharing her chemistry expertise.

Many thanks Holly Lundberg at the Chicago History Museum for encouraging me professionally and working with my schedule during the completion of this paper; and to Sandra Tilmon for her considerable contribution to my understanding of statistical analysis.

The remarkable support provided by my classmates on this project and throughout the course will not be forgotten. Nor will the help of Scott Lynch, who lent an understanding ear and provided varied assistance throughout the writing and editing of this paper. Finally, I would like to thank my parents for being so instrumental in the development of my passion for cultural heritage.

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LIST OF ABBREVIATIONS

ACRONYMS

AIC	American Institute for the Conservation of Historic and Artistic Works
ASCII	American Standard Code for Information Interchange
ATR-FTIR	Attenuated Total Reflectance Infrared Spectrometry
CAS Number	Chemical Abstracts Services Registry Number
CIELAB	*Commission internationale de l'éclairage* color space
EDS	Energy Dispersive X-ray Spectrometry
EHS	Enivironment, Health & Safety
EPA	Environmental Protection Agency
HSE	Health and Safety Executive
ICOM-CC	International Committee of Museums – Conservation Committee
ICON	Institute of Conservation
IR	Infrared
JCAMP	Joint Committee on Atomic and Molecular Physical Data (JCAMP)
LCA	Life Cycle Assessment
MCI	Museum Conservation Insitute
MSDS	Material Safety Data Sheet
OSHA	Occupational Safety and Health Administration
RH	Relative Humidity
SEI	Secondary Electron Imaging
SEM	Scanning Electron Microscopy
UV	Ultraviolet

SYMBOLS

fd	dispersion factor
fh	hydrogen bonding factor
fp	dipole factor
ΔE_{L*a*b*}	Change in CIELAB color value
ΔH_v	Heat of vaporization
δ	Hildebrand solubility parameter

UNITS

Mm	millimeter
Nm	nanometer
N	Newton
L	liter
mL	milliliter
kv	kilovolts

INTRODUCTION

Conservation treatment of textile objects in collections often involves the removal of soils and stains. In cases where soil removal is justified and necessary for the preservation or interpretation of an object, textile conservators have many techniques at their disposal. Along with surface cleaning, wet cleaning, and enzyme cleaning, cleaning with non-aqueous, organic solvents is a powerful tool for the trained conservator. Though effective as soil removers, organic solvents have been found to present health and environmental risks.

There is a need to find alternatives to organic solvents used in conservation treatments. At the same time, the professional dry cleaning industry has begun using new products and technologies in response to legal and market pressure to "go green". This has led to the development of alternative products and processes, including alternatives to conventional cleaning solvents.

Conservation is a relatively small field compared to the dry cleaning industry, which is estimated to be worth $20 billion a year.[1] While institutional policies and ethical guidelines might drive conservators to go "green", the major resources needed for developing and distributing new alternative products are in the hands of industry. For industry, the stakes are now high to drive innovation. It is for this reason research into the developments of the dry cleaning industry are thought particularly valuable at this time.

From the outset, the aim of this research was to examine whether the recent developments in green dry cleaning would provide useful tools for textile conservators, and a major objective of that aim was to find a suitable alternative solvent to use in experimental trials and analysis. The search for that solvent was, by its nature, not one of scholarship. It began, out of necessity, with a combing of the internet – company websites for dry cleaning chains, chemical suppliers, and equipment manufacturers, newspaper trend pieces, online trade publications, environmental agency publications – all to find out what products are out there, how they are supplied, and how they are used. A good candidate for testing would be one that could be used without special dry cleaning equipment, acquired easily, able to be sourced on both sides of the Atlantic, and would have a reasonably green pedigree. Consultation with a dry cleaning expert out of Oklahoma who maintains a website on the latest developments in dry cleaning helped to make sense of field of possible candidates, by providing options for acquiring possible test solvents and valuable information based on experience in the field.[2]

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[1] 'Industry Guides: Dry Cleaning Business', Fulcrum Inquiry, http://www.fulcrum.com/drycleaning_appraisal.htm, (accessed 19 April 2012).

[2] E. Childers,'Textile Cleaning', http://www.textilecleaning.com, (accessed 10 April 2012).

The candidate that was chosen as a result of this initial research is a cyclic silicone-based polymer with the common chemical name decamethylcyclopentasiloxane, or D5. D5 is used under the tradename GreenEarth® by independent dry cleaners and dry cleaning chains worldwide exclusively through licensing and support by the GreenEarth® company based in Kansas City, Missouri, founded in 1999. According to its website, GreenEarth® is the "world's largest brand of green dry cleaning."[3] At the time of this project, it was found to be the only commercial option for green dry cleaning in Scotland. With 1,600 dry cleaners using the product worldwide, it was though that if the trials produced favorable results then D5 would be available for further study and application by textile conservators. President Tim Maxwell of GreenEarth® provided 10 liters of GreenEarth® solvent free of charge for use in this study through UK chemical distributor Alex Reid. Experimental trials with D5 make up the primary focus of the research presented here.

This paper aims to place the results gathered from the D5 testing in context, with a brief look at solvent use in textile conservation and the developments in alternative dry cleaning processes. It will present some of the basic principles which govern solvent activity to provide a theoretical background from which to look at the properties and performance of D5 as observed in the testing phase of the experimentation. The experimental procedure is outlined, along with a presentation and discussion of results. Using this information, conclusions are made regarding D5's potential for further study and application as a textile conservation tool.

The following research questions were used as a focus for the examination of D5:

1. What makes D5 "green"?

2. How does treatment with D5 affect textiles? Does it cause undesirable change to their condition? Does it leave behind material which may damage textiles in the future?

3. Do its structure and properties predict anything about its performance as a solvent?

4. How effective is D5 at removing soils? What kinds of soils respond to treatment with D5?

5. What is it like to work with D5? How practical is it to use?

6. Are there potential advantages to the use of D5 in textile conservation?

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[3] 'Our Company, GreenEarth®Cleaning, http://www.greenearthcleaning.com/?page=AboutUs, (accessed 10 August 2012).

1. CHAPTER ONE: BACKGROUND

1.1. CONTEXT

1.1.1. CONCERNS ABOUT ORGANIC SOLVENTS

1.1.1.1. SOLVENTS IN TEXTILE CONSERVATION

Solvents encompass a broad range of substances which have the ability to dissolve compounds and carry them away from surfaces, making them essential for the removal of contaminants over a wide range of applications. Organic solvents are so-named because they are composed primarily of hydrocarbons, a composition which gives them special properties for solubilizing a variety of materials.

Along with wet cleaning, cleaning with organic solvents has been a treatment used by conservators from the beginning of modern textile conservation. *Workshop Notes* from the Textile Museum in Washington DC document the use of solvents to treat objects in the Museum's collection as far back as the early 1950's.[4] Organic solvents provide significant cleaning advantages, and may be chosen to address soils that cannot be removed with water, such as fats, oils and waxes; or when use of water might harm elements of the object: causing swelling or shrinkage of fibers, bleeding of dyes, or damage to finishes.[5] A list of solvents which are known to have been used by textile conservators is included in Appendix I.

At least as far back as the 1970's, conservators have been aware of the hazards of toxic health effects from the organic solvents in their toolkit[6] Though use of the most toxic solvents, such as benzene, have long been banned,[7], organic solvents still in use pose health and safety risks for those who are exposed to them, as well as to the eco-systems in which they are used and disposed.

As environmental concerns have grown in the latter half of the twentieth century, conservation has grown increasingly aware of the greater environmental implications of the use of organic solvents. These concerns have become a part of the growing trend toward "going green" gaining traction in the museum and heritage sector. Institutions continue to develop internal policies calling on greener practice, for everything from purchasing office supplies to air conditioning their buildings and stores.[8] The realm of conservation is not immune to these growing trends. It has been argued strongly that the

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[4] The Textile Museum Preparation Department, 'Principles of Practical Cleaning for Old and Fragile Textiles', In *Workshop Notes, Paper No. 14*, (Washington, D.C., 1956), 89-92.

[5] Á. Tímár-Balázsy and D. Eastop, *Chemical Principles of Textile Conservation*, (Oxford, 1998), 175.

[6] S.B. Landi, 'The Practice of Dry-cleaning in the United Kingdom.' *ICOM Committee for Conservation 6th Triennial Meeting, Ottawa, 21-25 September 1981*: Preprints, (Paris, 1978), 1-6.

[7] Tímár-Balázsy and Eastop , *Chemical Principles*, 179.

[8] S.S. Brophy and E. Wylie, *The Green Museum* (Lanham, Maryland, 2008), 10.

core values of heritage conservation resonate with those of environmental conservation.[9] Going green has even been posited as a way of raising the profile of conservation with the general public by capitalizing on the trend.[10]

Professional ethics in the field have begun to address concerns about the effects of conservation treatments on the environment. The importance of minimizing the environmental impact of the conservator's work has been written into the codes held by the major bodies governing conservation practice. Both the Institute for Conservation (ICON) in Great Britain and the American Institute for the Conservation of Historic and Artistic Works (AIC) explicitly invoke a conservator's responsibility to the environment and to health and safety in their guidelines for professional ethics (Appendix II). [11]

All organic solvents commonly used in textile conservation are thought be toxic to some degree,[12] and many are thought to have environmental implications beyond immediate exposure. Regulations governing the workplace, such as those ordered by the UK Health and Safety Executive (HSE)'s Control of Substances Hazardous to Health (COSHH), and the US Department of Labor's Occupational Health & Safety Administration (OSHA) require that solvent use be controlled, recorded, and performed under strict health and safety precautions. This means each solvent must be assessed by the conservator before use for its particular health and safety implications, ensuring for proper personal protection, ventilation and disposal. These practices are time-consuming and restrictive and must be weighed the needs of the object in the conservator's care.

In 2007, Capello et al. published a ranking of twenty-six organic solvents, many of which will be familiar to conservators, according to the hazards they present to the environment, health, and safety (EHS).[13]

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[9] E. Avrami, Heritage, Values and Sustainability', In *Conservation: Principles, Dilemmas and Uncomfortable Truths*, edited by A. Richmond, et al, eds., (Amsterdam, 2009), 181.

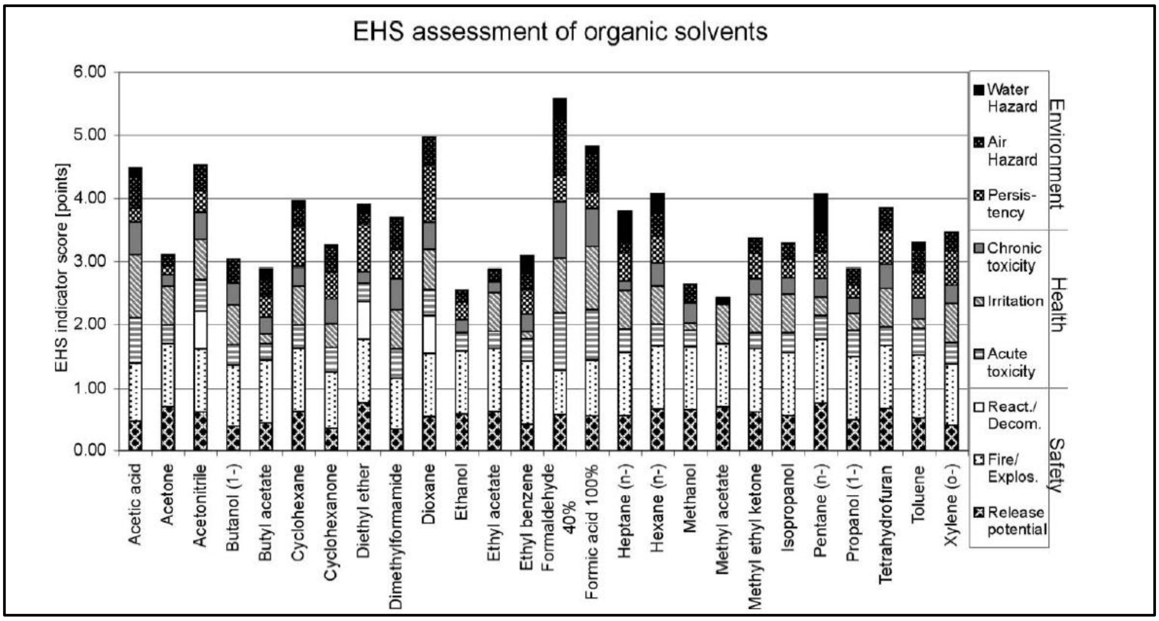
[10] M.de Silva and J. Henderson, 'Sustainable Practice in Conservation', *Journal of the Institute of Conservation* 34,1 (2011): 6

[11] 'Professional Guidelines: Article 9', The Institute of Conservation, http://www.icon.org.uk/index.php?option=com_content&task=view&id=121, (accessed 05 August 2012); 'Code of Ethics and Guidelines for Practice: Article VII', American Institute for the Conservation of Historic and Artistic Works, http://www.conservation-us.org/index.cfm?fuseaction=page.viewPage&pageID=858&nodeID=1, (accessed 05 August 2012).

[12] Tímár-Balázsy and Eastop, *Chemical Principles*, 179; M. Ballard and N.S. Baer, 'Professional Notes: Conservation I: Halogenated Hydrocarbon Dry Cleaning Solvents',*The International Journal of Museum Management and Curatorship* 8 (1989): 336; M. Pascoe, 'Toxic Hazards from Solvents in Conservation,' *The Conservator*, 4, 1 (1980): 25-28.

[13] C. Capello, et. al., 'What is a Green Solvent? A Comprehensive Framework for the Environmental Assessment of Solvents', *Green Chemistry* 9 (2007), 927-934.

FIGURE 1.1.: EHS ASSESSMENT OF ORGANIC SOLVENTS, REPRINTED FROM CAPELLO, ET AL., 'WHAT IS A GREEN SOLVENT? A COMPREHENSIVE FRAMEWORK FOR THE ENVIRONMENTAL ASSESSMENT OF SOLVENTS', *GREEN CHEMISTRY* 9 (2007): 930.



The study couples the assessment of immediate EHS risks with a life-cycle assessment, or LCA, which takes into account the broader environmental footprint of production and disposal of the solvents. This type of holistic evaluation resonates with the emerging field of green chemistry. Green chemistry proposes not only to deal with the hazardous effects of chemicals, but to examine their impact from production to disposal and propose substitutions when necessary.

Possible substitutes for organic solvents use in conservation need to be identified, and yet conservation research is just at the beginning to catch up with this need. Looking to the dry cleaning industry may provide new tools to solve this problem.

1.1.1.2. SOLVENTS IN INDUSTRY

Environmental legislation has begun to target organic solvents for the hazards that they present to health and the environment. The last few decades of the twentieth century has seen far-reaching international legislation restricting chlorinated solvents, particularly ozone-depleting chlorofluorocarbons, phasing out their use in both developed countries and the developing world by 2010.[14]

The most widely used solvent, in the dry cleaning industry, tetrachloroethylene, known to the trade as perchloroethylene or PERC, may not be far behind. PERC, another chlorinated solvent, has been the

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[14] J.B. Durkee, 'Cleaning with Solvents', In *Developments in Surface Contamination and Cleaning*, R. Kohli, et al., eds., (Norwich, N.Y., 2011), 763-765.

industry standard for decades. PERC has been shown to be toxic and carcinogenic, capable of causing cancer and neurological damage.[15]

The use of PERC is strictly regulated, but these restrictions may soon be growing more severe. In California, whose environmental laws are often ahead of the curve, yet predictive of future trends, the California Air Resources Board (CARB) has legislated to have PERC phased out of dry cleaners completely by 2013.[16] Both CARB and the New York State Environmental Protection Agency have encouraged the replacement of PERC by issuing statements approving alternative processes for dry cleaning (listed in Table 2.1), while acknowledging that the alternatives themselves are in need of further scrutiny. In anticipation of more widespread bans on the use of PERC, dry cleaners have begun to adopt new technologies from the growing field of green chemistry.

1.1.2. ALTERNATIVES TO ORGANIC SOLVENTS

Industry has begun to identify alternatives to its most harmful solvents, including ionic liquids, supercritical fluids, and cyclic siloxanes.[17] For the dry cleaning industry, the use of liquid carbon dioxide and cyclic siloxanes may be the most promising and marketable alternatives. Refined and synthetic hydrocarbons are also proposed as alternatives to traditional hydrocarbon solvents.

1.1.2.1. LIQUID AND SUPERCRITICAL CARBON DIOXIDE

At high temperature and pressures, carbon dioxide can behave like a liquid solvent – capable of removing soils and carrying them away from textiles without interacting with the textile. It is thought to be green because the only by-products are nontoxic gases and water. Recently, there has been research into the use of this technology in the conservation field, including the cleaning of textiles,[18] however the need for a high pressure system creates many practical barriers for use in conservation. Dry cleaners have the advantage of tumbling articles in a drum within a closed system machine, handling which would be inappropriate for the vast majority of artifacts.

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[15] United States Environmental Protection Agency 'Existing Chemicals: Fact Sheet on Perchloroethylene, also Known as Tetrachloroethylene', February 2012,US Environmental Protection Agency, http://epa.gov/oppt/existingchemicals/pubs/perchloroethylene_fact_sheet.html, (accessed 10 August 2012).

[16] California Envionmental Protection Agency Air Resources Board,'Fact Sheet 2008: Dry Cleaning Alternative Solvents: Health and Environmental Impacts', March, 2008, http://www.arb.ca.gov/toxics/dryclean/alternativesolvts_e.pdf (accessed 20 March 2012).

[17] J. M. Desimone, 'Practical Approaches to Green Solvents', *Science* 297 (2 August 2002), 799-803.

[18] H. Tello and A. Unger, 'Liquid and Supercritical Carbon Dioxide as a Cleaning and Decontamination Agent for Ethnographic Materials and Objects', A.E. Charola, et al., eds., *Pesticide Mitigation in Museum Collections: Science in Conservation: Proceedings from the MCI Workshop Series,* (Washington, D.C, 2010):.35-50; M. Sousa, et al., 'The Art of CO_2 for Art Conservation: A Green Approach to Antique Textile Cleaning,' *Green Chemistry* 9 (2007): 943-7.

1.1.2.2. REFINED AND SYNTHETIC HYDROCARBONS

Highly refined and synthetic hydrocarbons are also considered possible viable alternatives to PERC. These compounds are classified as VOC's. VOCS are known to cause immediate harmful effects to health. These hydrocarbons have not yet been approved by CARB, citing the need for more research. Stoddard solvent, often used in conservation applications, is among this class of solvents.[19]

1.1.2.3. CYCLOSILOXANES

Decamethylcyclopentasiloxane (D5) is among the class of silicone-based polymers known as amethylated cyclosiloxanes, which also include hexamethylcyclotrisiloxane (D3), octamethylcyclotetrasiloxane (D4), and dodecamethylhexasiloxane (D6). Cyclosiloxanes are classed as volatile methylsiloxanes (VMS). In the United States, VMS fluids are not considered as volatile organic compounds (VOCs),[20] indicating a lesser risk for harmful health effects from short-term immediate exposure. The EPA has not issued a formal risk assessment on the health effects of D5 as of this writing. The government of Canada has issued a statement that D5 is not considered to be a high priority for assessment of potential risks to human health.[21]

Studies have indicated, however, that exposure to D5 may cause harm to reproductive and immune functions.[22] D5 is known to be bioaccumulative and persistent in the environment, both in air and water, but the UK Environment Agency has assessed the risks from environmental exposure as minimal.[23] Use of D5 is widespread in cosmetics and pharmaceutical applications. In cosmetics, D5 is known as a cyclomethicone, a term which may indicate a pure cyclosiloxane, including D3, D4, and D6, or mixture of cyclic and linear siloxanes, including polydimethylsiloxane (PDMS).

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[19] California Environmental Protection Agency Air Resources Board, 'Fact Sheet 2008', 2.

[20] 'XIAMETER® Brand Cyclosiloxane Fluids', Silicones Simplified, Xiameter® from Dow Corning, https://www.xiameter.com/en/ExploreSilicones/Silicone-Fluids/Pages/cyclosiloxane-fluids.aspx, (Accessed 15 August 2012).

[21] Government of Canada, 'Siloxane D5 (Cyclopentasiloxane, decamethyl-)', *Chemical Substances,* 10 February 2012, http://www.chemicalsubstanceschimiques.gc.ca/challenge-defi/summary-sommaire/batch-lot-2/541-02-6-eng.php (accessed 25 July 2012).

[22] 'Cyclosiloxanes: Materials for the December 4-5, 2008 Meeting of the California Environmental Contaminant Biomonitoring Program (CECBP) Scientific Guidance Panel (SGP)', California Office of Environmental Health Hazard Assessment, (accessed 18 August 2012).

[23] D.N. Brooke, et al., 'Environmental Risk Assessment Report: Decamethylcyclopentasiloxane,' *Environment Agency,* 2009, http://publications.environment-agency.gov.uk/PDF/SCHO0309BPQX-E-E.pdf. (Accessed 15 August 2012)

D5 is available from a range of manufacturers. GreenEarth® sources D5 from Dow Corning®, Shin-Etsu, and GE® Silicones. Dow Corning has recently started marketing their silicones through the name Xiameter®.[24]

TABLE 1.1: LIST OF ALTERNATIVE DRY CLEANING METHODS

			Approval	
Method/Material	CAS	VOC	CARB	NY State EPA
GreenEarth® decamethylcyclopentasiloxane	541-02-6	no	**	✓
Rynex 3™ dipropylene glycol tert-butyl ether	132739-31-2	yes	**	✓
ExxonMobil DF-2000 synthetic hydrocarbon	64742-48-9	yes	**	✓
Stoddard Solvent	8052-41-3	yes	**	-
Sasol (LPA-142) highly refined hydrocarbon	64742-47-8	yes	-	✓
ChevronPhilips® EcoSolv®, highly refined hydrocarbon	68551-17-7	yes	**	✓
R.R. Streets Solvair ™ dupropylene glycol n-butyl ether (DPGnBE) (used with liquid carbon dioxide)	29911-28-2	yes	**	✓
SolvonK4™ dibutoxymethane	2568-90-3	no*	**	✓
1-Bromopropane n-propyl bromide	106-94-5	yes	**	-
Carbon dioxide	-	no	✓	✓
Professional wet cleaning	-	no	✓	✓
*below European directive standard, not assessed by CARB				
**under consideration, 2008				

(Adapted from ‘Fact Sheet 2008: Dry Cleaning Alternative Solvents: Health and Environmental Impacts.’ California Air Resources Board, March 2008, http://www.arb.ca.gov/toxics/dryclean/alternativesolvts_e.pdf, (accessed 20 March 2012); and ‘Approved Alternative Solvents for Dry Cleaning’, New York State Department of Environmental Conservation, 2012, http://www.dec.ny.gov/chemical/72273.html (accessed 20 February 2012)

1.1.3. Textile Conservation & Dry Cleaning: Collaboration and Considerations

Textile conservators have long utilized knowledge, products, and processes developed by the professional dry cleaning industry and adapted these for the needs of their collections. Published examples include the Textile Museum’s collaboration with the National Institute of Dry Cleaning in

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[24] ‘‘Xiameter ® Brand Silicones’, Xiameter from Dow Corning®, https://www.xiameter.com/en/ExploreSilicones/Pages/Discoverhome.aspx, (Accessed 15 August 2012).

the United States in the 1960's,[25] and the adaptation of a commercial dry cleaning machine by the Royal Scottish Museum (now National Museum of Scotland) as early as the 1970's.[26]

Textile conservators will occasionally elect to take large objects requiring solvent cleaning to a professional dry cleaning facility. It can be a challenging collaboration. For textile conservators, the first priority is the object in his or her care, while professional dry cleaners aim to clean textiles in a manner that is high-volume, efficient, cost-saving, and consistently effective at removing soils. This means that articles are processed in bulk, under high heat and agitation, and with additives, including water and detergent in the processing.

Success can be achieved, however, when the textile being cleaned is particularly robust, able to be protected in a way that will minimize handling and agitation, and processed without heat or additives.[27] With the disappearance of PERC on the horizon, it may be useful to find out what alternatives will be available in the future, in dry cleaning facilities as well as the lab.

1.1.4. SILICON-BASED SOLVENTS & TEXTILE CONSERVATION

1.1.4.1. HISTORY

Silicon-based solvents have been in use for decades in cosmetics and other applications, and were originally patented for use in textile cleaning in 1987,[28] twelve years before the founding of GreenEarth®. As far back as 1998, there has been awareness of the potential of silicon-based solvents for use in the textile conservation field. Tímár-Balázsy and Eastop include a silicon-based solvent by the trade name Volasil® in their discussion of solvent cleaning, however it is only briefly mentioned, and not expounded on in any detail.[29] Searches of the literature failed to reveal any published accounts of its use in conservation treatments. Volasil® is still on the market today, distributed through various companies marketing it for use in cosmetic applications. As it turns out, the name encompasses

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[25] L. Bellinger, 'Cleaning of Textiles', International Institute for Conservation of Historic and Artistic Works, *1964 Delft Conference on the Conservation of Textiles, Collected Preprints, 2nd ed.* (London: IIC, 1965), 92-93.

[26] L. Eaton and K. Moodie, 'The Development of Dry-cleaning at the Royal Museum of Scotland' in *Conservation Today: Preprints for the UKIC 30th Anniversary Conference 1988*, ed. V. Todd, (London: United Kingdom Institute of Conservation, 1988), 38-40.

[27] Canadian Conservation Institute, 'Conservation Notes 13/13: Commercial Dry Cleaning of Museum Textiles,' http://www.cci-icc.gc.ca/publications/notes/13-13_e.pdf (Accessed 12 April 2012); Keifer, Kathleen,"Conserve O Gram Number 16/2: Dry Cleaning Museum Textiles," *National Park Service, Department of the Interior.* September 2000, http://www.nps.gov/museum/publications/conserveogram/16-02.pdf (accessed 12 April 2012).

[28] K.A. Kasprzak, 'Methods for Cleaning Textiles with Cyclic Siloxanes', United States Patent 4,685,930. August 11, 1987.

[29] Tímár-Balázsy and Eastop, *Chemical Principles*, 184.

several formulations of cyclosiloxanes, including D4, D5 and D6. (See Appendix III for additional trade synonyms for D5).

1.1.4.2. CURRENT USE

In the course of this research, it was possible to find one source who has used D5 extensively to treat historic textiles. Doris Easley began a career in the dry cleaning business in California 1943, eventually serving as president of the state board of Fabricare and as president of the International Dry Cleaner's Council in 1987.[30] Since the early nineties, she has turned to specialized cleaning and restoration work. Coming from a dry cleaning background, Easley brings strength of knowledge of solvent-based textile cleaning methods to her work. This same strength may raise alarm bells with conservators keen to point out that the best treatment for an object may not be to clean it at all. Nevertheless, Easley's record of work with the Asian Art Museum San Francisco and the Los Angeles County Museum of Art, among other institutions, suggests that there may be a place for industry-acquired knowledge within the professional and ethical boundaries of the cultural heritage sector.

Easley was consulted in the course of this research, by phone and by email. She reports that using GreenEarth® D5 enables her to clean textiles in a way that is safer than traditional solvents, and without the drying effect on textiles that many solvents produce. She reports that though no solvent can work on every soil, D5 has been useful tool for many projects, including heavily soiled textiles. She notes that at times it is necessary to mix D5 with a non-ionic detergent for effective removal of some soils,[31] a mixture which known in the dry cleaning trade as a "charged" solvent.[32]

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[30] S. Havranek, 'From Rags to Riches: Dry-cleaning Vet Plies Specialized Trade: Restoring Vintage Clothes,' *Sacramento Business Journal*, September 24, 2004, 17-18.

[31] Personal communication with Doris Easley, email 03 August 2012 and phone 05 August 2012.

[32] A charged solvent is a solution made up of a solvent carrying a detergent, and in some a very minimal amount of water. Professional dry cleaning processes often use charged solvents. Tímár-Balázsy and Eastop, *Chemical Principles* 176.

1.2. THEORY

1.2.1. SOLVENTS AND SOLUBILITY

1.2.1.1. SOLVENTS DEFINED

A solvent is defined as a substance which acts to bring another substance, known as the solute, into a solution, or mixture. In broad terms, a solution can be thought of as 'any mixture of substances which is homogenous down to a molecular scale'.[33] Solutions as they will be discussed here, however, will focus on liquid and solid interactions.

1.2.1.2. INTERMOLECULAR FORCES

The forces responsible for cohering homogenous molecules into a solid or a liquid are the same types of bonds which attract heterogeneous molecules to one another. These intermolecular forces are not as strong as the primary ionic and covalent bonds which make up molecules, but can be very powerful in adhering one substance to another, as a soil to the surface of a textile. To dissolve a soil or stain, a solvent must be capable of either dispersing the molecules of the soil, or swelling them enough to allow them to be carried away from the substrate with agitation.

Three main types of intermolecular forces can exist between molecules are:

- **van der Waals**: movement of electrons around the atoms of a molecule causes shifts in electronegativity. These shifts result in fluctuating polar attractions between molecules.
- **Dipole-dipole/polar**: when one end of a molecule is significantly more electronegative than the other, this produces a pronounced dipole moment which allows attraction between the electronegative end of one molecule and the electropositive end of another.
- **Hydrogen bonding**: The strongest of the intermolecular forces occurs when hydrogen atoms in one molecule can create secondary bonds with the oxygen or nitrogen atom of an adjacent molecule.

1.2.1.3. SOLUBILITY PARAMETERS

There are various methods for quantifying the forces in substances which will allow a solvent-solute interaction. Borrowing from Torraca,[34] Burke thoroughly describes solubility parameters in the context of conservation materials, invoking both Hildebrand and Hansen solubility parameters as tools

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[33] Anne Moncrieff and Graham Weaver, *Science for Conservators, Vol. 2: Cleaning* (London: Routledge, 1992), 49.

[34] G. Torraca, *Solubility and Solvents for Conservation Problems, 2nd ed.* (Rome: International Centre for the Study of Preservation and Cultural Property), 1975.

which have been employed by conservators.[35] Conservators commonly employ solubility parameters to determine which solvents may work on a certain soil or stain. Conversely, these parameters may help identify the identity of a soil or stain according to the composition of the substance that will successfully dissolve it.

Hildebrand and Hansen solubility parameters are both based on the principle that the interaction of intermolecular forces between solvents and solutes determines their ability to come into solution. When the intermolecular forces are the same, then solubility will result. As Hansen states, the 'physical affinities of two materials are at a maximum when their energies match.'[36]

Hildebrand Solubility Parameters

Hildebrand solubility parameters compare the energies of materials by calculating an overall value for the energy of their intermolecular forces. The calculation is equal to the square root of the cohesive energy density of the substance. Cohesive energy density, in turn, is calculated from the known heat of vaporization and molar volume of the molecule at standard temperature and pressure.

EQUATION 1.1: HILDEBRAND SOLUBILITY PARAMETER (δ)

$$\delta = \sqrt{\frac{\Delta H_v - RT}{V_m}}$$

= cohesive energy density ΔH_v = heat of vaporization V_m = molar volume

R=gas constant T=temperature

Comparing the Hildebrand values of materials is helpful in determining their ability to come into solution. The *Journal of Cosmetic Science* has published the Hildebrand solubility parameter for 'Dimethicone D5' to be equal to 5.77.[37] This figure can be used to make predictions about which soils D5 may be able to dissolve when compared with known Hildebrand values for soils. Even better

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[35] J. Burke, 'Solubility Parameters: Theory and Application', *The Book and Paper Group Annual,* The American Institute for Conservation*,* 1984, http://cool.conservation-us.org/coolaic/sg/bpg/annual/v03/bp03-04.html, (accessed April 20, 2012).

[36] C.M. Hansen, 'Surface Characterization Using Hansen Solubility (Cohesion) Parameters', *Proceedings of the 28th Risø International Symposium on Materials Science: Interface Design of Polymer Matrix Composites – Mechanics, Chemistry, Modelling and Manufacturing*, (Roskilde, Denmark, 2007): 192

[37] C.D. Vaughn, 'Using Solubility Parameters in Cosmetics Formulation', *Journal of Cosmetic Science* 36 (September/October 1985): 329.

predictions about solubility performance are possible by using parameters which 'take into account the dispersive and polar character' of the intermolecular forces.[38]

Hansen Solubility Parameters

Hansen proposed that solubility parameters be based on the fractional proportions of the types of bonds that make up the overall energy of a substance. For each substance, that overall energy is made up of some percentage van der Waals, some percentage dipole, and some percentage hydrogen bonding. In the Hansen solubility parameters, the most affinity between substances will occur when the percentages for each match.

Hansen parameters are derived from empirical experimentation and are published for many different solvents and soils. (See Appendix I for fractional parameters of solvents used in textile conservation). They have been adopted with success by conservators, who may use the triangular axes of a Tea's graph to plot the intersection of the three values. An example is provided in Figure 1.2. On the graph, two organic solvents are plotted: the highly polar methanol and the highly nonpolar white spirit. Water is also included for comparison.

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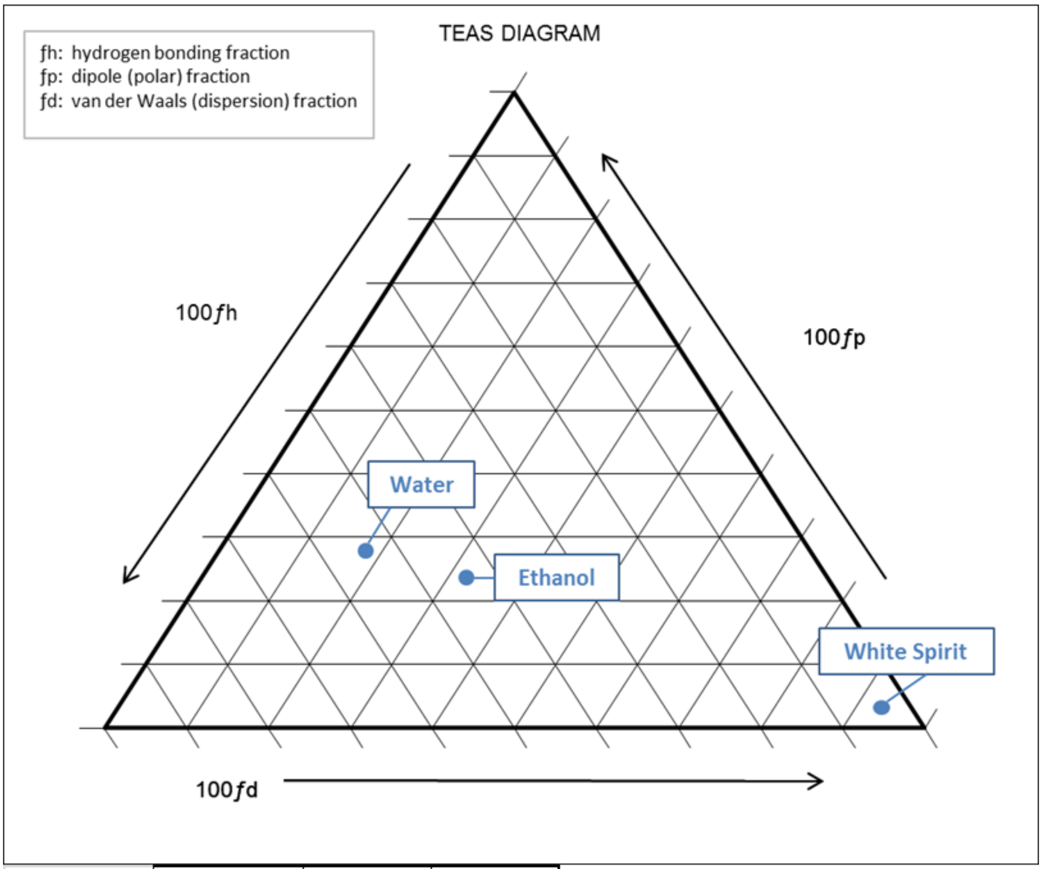
[38] C.M. Hansen, 'Surface Characterization', 192.

FIGURE 1.2: TEAS DIAGRAM SHOWING LOCATIONS OF THREE COMMON SOLVENTS, ADAPTED FROM TÍMÁR-BALÁZSY AND EASTOP, CHEMICAL PRINCIPLES OF TEXTILE CONSERVATION, 1998, 166. (TERNARY GRAPH FORMATTED WITH TRI-PLOT ©GRAHAM AND MIDGELY, 2000)



	100 ƒd	100 ƒp	100 ƒh
Water	18	28	54
White Spirit	90	4	6
Ethanol	36	18	46

No source was found in the research which gives the fractional parameters of D5. While the methods of determining Hildebrand solubility parameters are theoretical calculation based on easily gathered data, the methods of determining the fractional parameters for substances are based ‘on experimental observation and can be a “difficult and laborious undertaking’,[39] which is likely why the information is not yet available.

A look at the structure of the molecule can help to estimate what the relative proportions of the intermolecular forces might be for D5.

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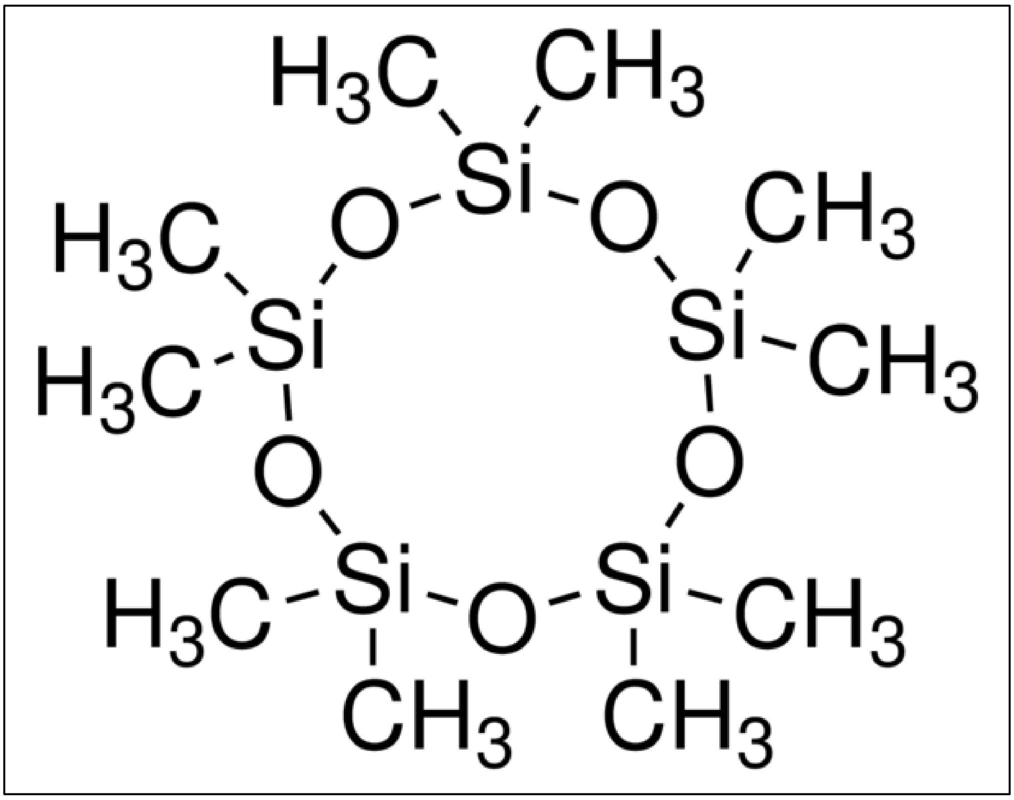
[39] D.M. Koenhen,and C.A. Smolders, ‘The Determination of Solubility Parameters of Solvents and Polymers by Means of Correlations with Other Physical Quantities’, *Journal of Applied Polymer Science*, 1975:1164

1.2.2. DECAMETHYLCYCLOPENTASILOXANE (D5)

1.2.2.1. STRUCTURE

D5 is a an organo-silicon, composed of a ring of oxygen and silicon atoms, with methyl groups branching from each silicon atom, as shown in Figure 1.3 Though in structure it is similar to a cyclic hydrocarbon, the presence of the silicon atoms distinguishes it from a classically organic hydrocarbon.

FIGURE 1.3: STRUCTURE OF DECAMETHYLCYCLOPENTASILOXANE, REPRINTED FROM 'DECAMETHYLCYCLOPENTASILOXANE,'SIGMA ALDRICH, HTTP://WWW.SIGMAALDRICH.COM/CATALOG/PRODUCT/ALDRICH/444278?LANG=EN&REGION=US (ACCESSED MAY 2012).



The chemically symmetrical cyclic structure of D5, with its branching nonpolar methyl groups, indicates that there is no strong dipole moment in the molecule. The presence of hydrogen and oxygen throughout the molecule suggest that hydrogen bonding is very likely to occur. It is reasonable to assume, therefore, that D5 creates secondary bonds with other molecules primarily through van der Waals (*ƒ*d) and hydrogen (*ƒ*h) forces, with little to no dipole interaction (*ƒ*p).This assumption would locate D5 toward the bottom right corner of the Tea’s diagram, closer to white spirit than water or methanol, both of which derive their intermolecular energy largely through hydrogen bonding.

1.2.2.2. PROPERTIES

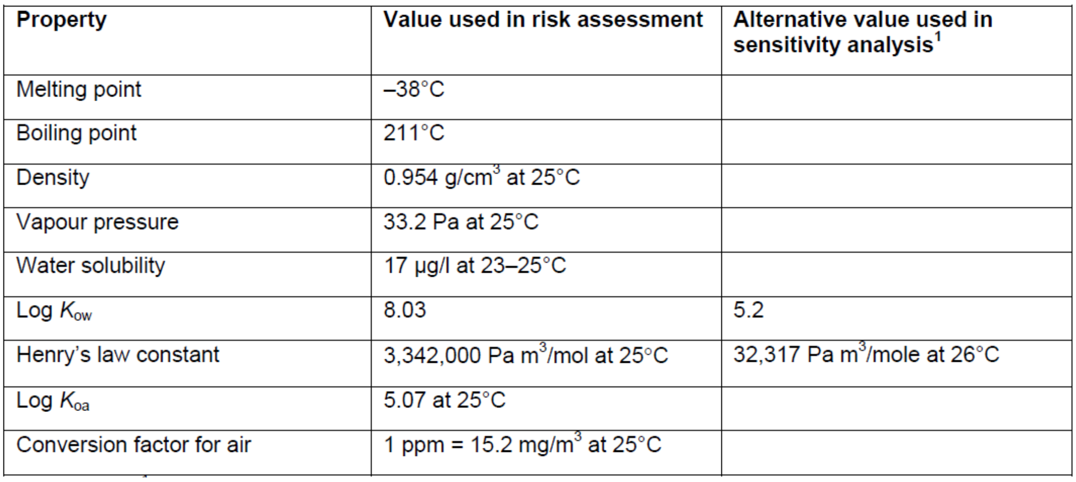
Like many organic solvents D5 is volatile compound. Volatile solvents have become indispensable tools for conservation cleaning because they are able to carry soils away from the surface of an object, and then evaporate away completely without leaving behind contaminant material. As a volatile, it is reasonable to expect that this is also true of D5.

Though a host of solvents will evaporate away without leaving behind harmful material, they may negatively affect textiles by dessicating the fibers.[40] In Easley's experience, D5 does not have this effect.

Organic solvents generally have a surface tension which is lower than that of water, therefore solvent solutions will wet out materials more rapidly than aqueous solutions. Quick wetting minimizes handling time for fragile objects, so the combination of low surface tension and the tendency to evaporate away from textiles makes solvents ideal for many cleaning applications.

The Environment Agency UK reports the surface tension of D5 as 18.9 mN/m at 20°C and 18.5 mN/m at 25°C,[41] w which is low compared to that of water at 73mN/m.[42]

TABLE 1.2: PHYSICAL PROPERTIES, REPRINTED FROM BROOKE, D.N., ET AL.,'ENVIRONMENTAL RISK ASSESSMENT REPORT: DECAMETHYLCYCLOPENTASILOXANE', *ENVIRONMENT AGENCY,* 2009. HTTP://PUBLICATIONS.ENVIRONMENT-AGENCY.GOV.UK/PDF/SCHO0309BPQX-E-E.PDF. (ACCESSED 15 AUGUST 2012)



Property	Value used in risk assessment	Alternative value used in sensitivity analysis[1]
Melting point	–38°C	
Boiling point	211°C	
Density	0.954 g/cm^3 at 25°C	
Vapour pressure	33.2 Pa at 25°C	
Water solubility	17 µg/l at 23–25°C	
Log K_{ow}	8.03	5.2
Henry's law constant	3,342,000 Pa m^3/mol at 25°C	32,317 Pa m^3/mole at 26°C
Log K_{oa}	5.07 at 25°C	
Conversion factor for air	1 ppm = 15.2 mg/m^3 at 25°C	

(See Appendix IV for additional properties of D5.)

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[40] Tímár-Balázsy and Eastop, *Chemical Principles*, 172.
[41] D.N. Brooke, et al., 'Environmental Risk Assessment Report', 20.
[42] Tímár-Balázsy and Eastop, *Chemical Principles* 192.

1.2.2.3. POTENTIAL FOR USE IN TEXTILE CONSERVATION

Though more study is needed, the research suggests that D5 may present fewer health and environmental risks than organic solvents if used in limited quantities. D5 also shares some of the properties of organic solvents which are used to advantage in conservation cleaning.

Its widespread use in the dry cleaning industry suggests that D5 is an effective soil remover. Dry cleaning conditions, however, are very different than those used in textile conservation, as previously discussed. Would D5 used on its own, without heat and the addition of detergents, have an effect on soils that my make it useful to conservators? In theory, D5 has the potential to be effective on nonpolar soils – would experimental evidence confirm this?

The experimental phase of the research assessed the performance of D5 in the conservation context. The testing was designed to show whether D5 would fit a conservators criteria for treatment: providing effective soil removal without leaving behind detectable contaminant material or causing appreciable damage to fibers.

There was one possibility of contamination which inspired the most concern when considering D5 as a solvent for historic objects. D5 contains silicon and oxygen, which are the elements that make up sand. There was some speculation that, if residue is left behind from treatment with D5, then over time the silicon and oxygen atoms in any remaining residue might react to form microscopic silica molecules which would have potential to cause physical degradation to textile fibers. The structure of the D5 molecule is described to be stable: with a zero reactivity level,[43] however, which suggests that it is nonreactive under normal conditions structure of the D5 molecule and its energetic disposition toward volatility suggest that this possibility is unlikely, however experimental testing would be a step toward confirming this assumption.

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[43] Cyclopentasiloxane; MSDS; GE® Silicones, November 06, 2003,*GE MSDS*, http://www.greenearthcleaning.com/?page=InfoFactsResearch (accessed 05 May 2012).

2. CHAPTER TWO: EXPERIMENTAL DESIGN

This chapter describes the experimental protocol which was used to find answers to the following of the research questions:

- What, if any, effect does treatment with D5 have on a textile?
- How effective is D5 at removing soiling from textiles?

In abstract terms, a soiled historic textile can be seen as a system. This system is composed of many factors, but its major factors can be broken down to two elements: the textile being soiled, and the substance doing the soiling. The term 'substrate" is used here as shorthand for the textile element of the system, while the term "soil" or "soiling" is used to describe the soiling substance.

In order to assess how treatment with D5 might affect these two factors. The experiment was divided into two separate stages. Stage 1 examines the D5's effect on the textile substrate, while Stage 2 assesses the effect of D5 treatment on the soiling. It was thought this sequence of testing stages would provide an effective experimental analogue to the major factors present in a practical solvent cleaning application.

In this chapter, the steps making up both stages of experimentation are outlined, including sample selection and preparation, the procedure followed for the application of the independent variables, and the subsequent analysis of the dependent variables.

2.1. STAGE 1: TEXTILE SUBSTRATE TESTING

In this stage, samples were subjected to a combination of independent variables (or corresponding controls) consisting of immersion treatment with D5 and artificial ageing. The effects of these independent variables were assessed using Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR), tensile strength testing, and Scanning Electron Microscopy (SEM).

2.1.1. SAMPLE SELECTION AND PREPARATION

2.1.1.1. FABRIC SELECTION

Sample specimens were taken from new woven fabrics for testing. New fabrics were chosen to eliminate the variability inherent to aged textiles. Though the ultimate aim of the project would be to gather enough information to assess the possible use of D5 on textile artifacts, an examination of D5's effect on new materials would provide the controls necessary to obtain more scientifically sound results. The information gathered in this study will provide the foundation for more research which can take into account the variability of aged and degraded textiles.

Only a limited range of fabric types could be tested due to the restrictions of time and resources. It was decided to use natural fibers, as they are materials very familiar to all textile conservators. Cotton and wool were selected to provide some variety to the range, to see the effects of treatment on both cellulosic and proteinaceous materials. .

2.1.1.2. SAMPLING METHOD

The sample specimens were cut from lengths of plain weave cotton lawn and wool delaine. Before sampling, the fabrics were pre-scoured in order to remove finishes or residues which might introduce unknown variables.

Rectangular pieces 150 mm long by 185 mm wide were cut from both fabric in the manner shown in Figure 2.11. Each piece was then divided in half in order to produce two samples measuring 150 mm long by 92.5 mm wide, one half which would be treated with D5 immersion, and one half which would remain a control.

Because the samples would ultimately be subjected to tensile testing, it was important to note where they were cut from the bolt of cloth. Ideal adherence to British Standards would prescribe that the samples be taken in a stepwise pattern from the original fabric.[44]

This is done to control for faults which might occur in any one section of the fabric from which the samples are being taken. These standards exist so that tensile strengths can be easily compared over multiple trials and experiments. This experiment was designed to be a single trial, not compared to previous studies, so a sampling pattern was designed for the project which differs from the British standard. This was done to maximize the resources at hand. The sampling pattern would still account for the difference in warp and weft, and ensure that specimens treated to opposing independent variables in direct comparison during analysis would come from adjacent areas on the original cloth.

Controlling for warp and weft in tensile tests is important because warp and weft can differ in a single fabric via the strength of their fibers, the spin of the fibers, the twist of the yarns, and the overall number of yarns used between warp and weft. For this reason, half the samples were taken from the fabric in the warp direction, and half from the weft direction. The orientation was noted for eventual comparison of the testing results.

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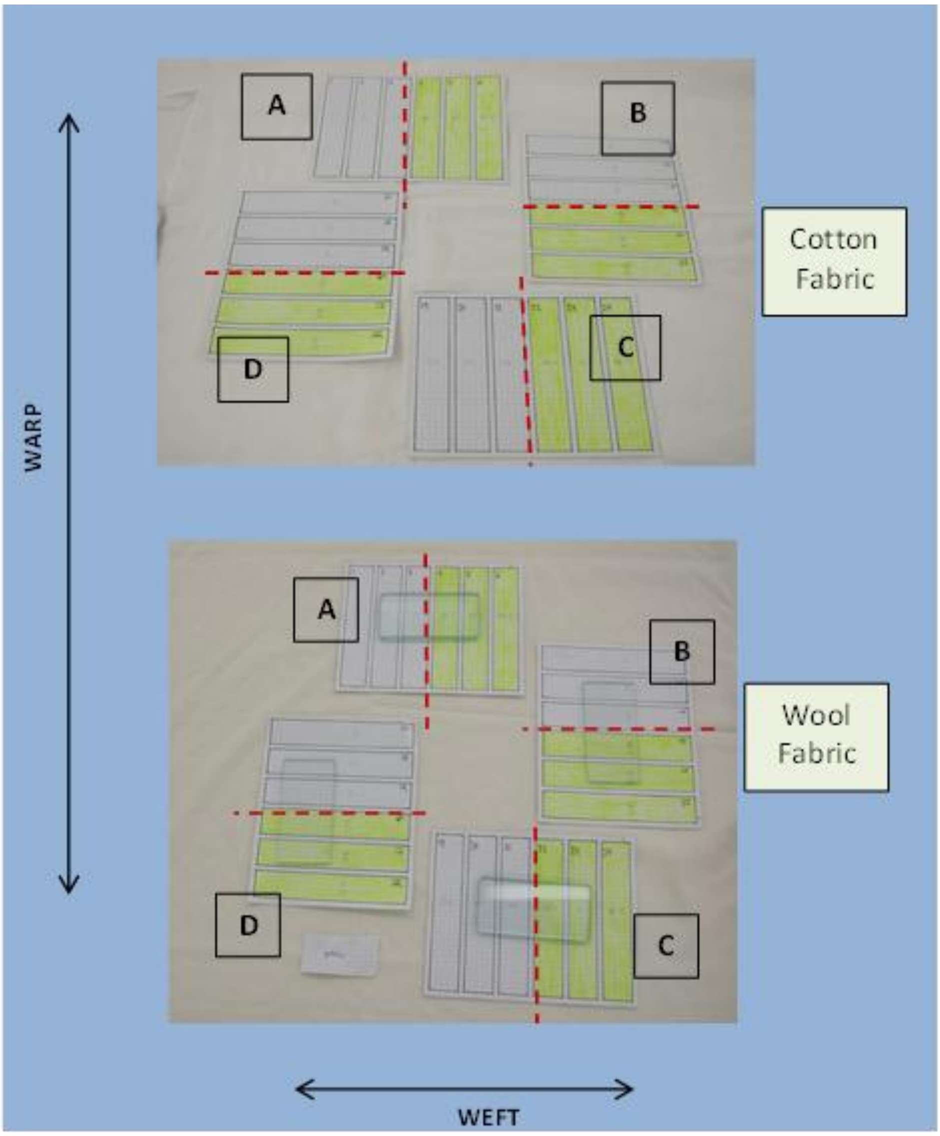
[44] British Standards Institution,'Textiles:Tensile Properties of fabrics, Part 1: Determination of Maximum Force and Elongation at Maximum Force Using the Strip Method',(London, 1999), BS ISO 13934-1:1999.

FIGURE 2.2: PAPER TEMPLATES MARK THE SAMPLING AREAS OF THE COTTON AND WOOL FABRIC. EACH PIECE WAS WAS DIVIDED ALONG THE DOTTED LINE INTO TWO SPECIMENS TO CREATE THE SAMPLE POPULATION FOR THE TEXTILE SUBSTRATE TESTING STAGE OF EXPERIMENTATION. ARROWS SHOW THE ORIENTATION OF WARP AND WEFT



Four rectangular areas were cut from the both cotton and wool fabrics and labeled A, B, C and D, as shown in Figure 2.2. The four lettered sections were then divided into the treatment samples (indicated by the yellow side of each template), and the control samples (the white side). Now divided, these would be immersed and analyzed with ATR-FTIR. Before tensile testing they would be cut into three strips as marked on the template.

The samples were labeled as shown in Table 2.1.1.

TABLE 2.1: DIVISION OF ORIGINAL SAMPLES INTO TESTING SPECIMENS

COTTON			WOOL		
Label			Label		
Cloth Position	Original Sample No.	Tensile Test Sample No.	Cloth Position	Original Sample No.	Tensile Test Sample No.
A		1	A		1
	1-3	2		1-3	2
		3			3
		4			4
	4-6	5		4-6	5
		6			6
B		7	B		7
	7-9	8		7-9	8
		9			9
		10			10
	10-12	11		10-12	11
		12			12
C		13	C		13
	13-15	14		13-15	14
		15			15
		16			16
	16-18	17		16-18	17
		18			18
D		19	D		19
	19-21	20		19-21	20
		21			21
		22			22
	22-24	23		22-24	23
		24			24

2.1.2. WORKING WITH D5

The D5 used in this experiment was provided by the GreenEarth® company, shipped through UK chemicals distributor Alex Reid (See Appendix VI, List of Suppliers). It was shipped in a quantity of 10 liters, housed in a plastic container, labeled “Green Earth Solvent.” It did not ship with a material data safety sheet (MSDS), however the GreenEarth® website provides MSDS’s from its three possible providers: GE™ Silicones, Dow Corning®, and Shin-Etsu. Alex Reid sources its D5 through Dow Corning®.[45] Each MSDS identifies the substance as decamethylcyclopentasiloxane, CAS number 541-02-6. The MSDS from Dow Corning reports a weight by volume of >60% w/v D5,[46] while that of GE™ is reported at >90%[47] and Shin-Etsu is reported at 100%.[48]

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[45] Personal email communication with Tim Maxwell, 21 August 2012.

46 ‘Fabric Cleaning Fluid: MSDS’, 04 April 2003, Dow Corning®, *Dow Corning MSDS*, http://www.greenearthcleaning.com/?page=InfoFactsResearch (accessed 05 May 2012), 1.

47 ‘GEC-5: MSDS’, 26 November 2003, *ShinEtsu MSDS*, http://www.greenearthcleaning.com/?page=InfoFactsResearch (accessed 05 May 2012), 1

FIGURE 2.3: MATERIALS ASSEMBLED IN FUME CUPBOARD IN PREPARATION FOR D5 IMMERSION TREATMENT.

2.1.2.1. HEALTH AND SAFETY

A COSHH form was completed as a part of the risk assessment for the trials (Appendix V). The MSDS sheets from the three suppliers were consulted in order to indicate the health and safety precautions that should be taken when decanting, using, and disposing of D5. (see Appendix IX). All handling of D5 was performed in a fume cupboard with the extraction on. Gloves and a lab coat were worn at all times.

2.1.2.2. OBSERVATIONS ON USE

D5 is described in the literature as odorless and colorless, and in practice this was observed to be true. The surface tension proved to be as expected, wetting out the samples quickly and thoroughly. There were a few spills when handling, (note the container label in Figure 2.1.2 is partially saturated with a D5 spill) which were small and easily controlled, but they required an appreciable amount of time before they were completely dry. Drying time was not strictly measured in the experiment, but the general impression from the treatment and the spills is that D5 takes several hours to up to a day evaporate from surfaces, even under fume extraction.

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48 'Cyclopentasiloxane: MSDS', 06 November 2003, *GE MSDS*, http://www.greenearthcleaning.com/?page=InfoFactsResearch (accessed 05 May 2012), 1.

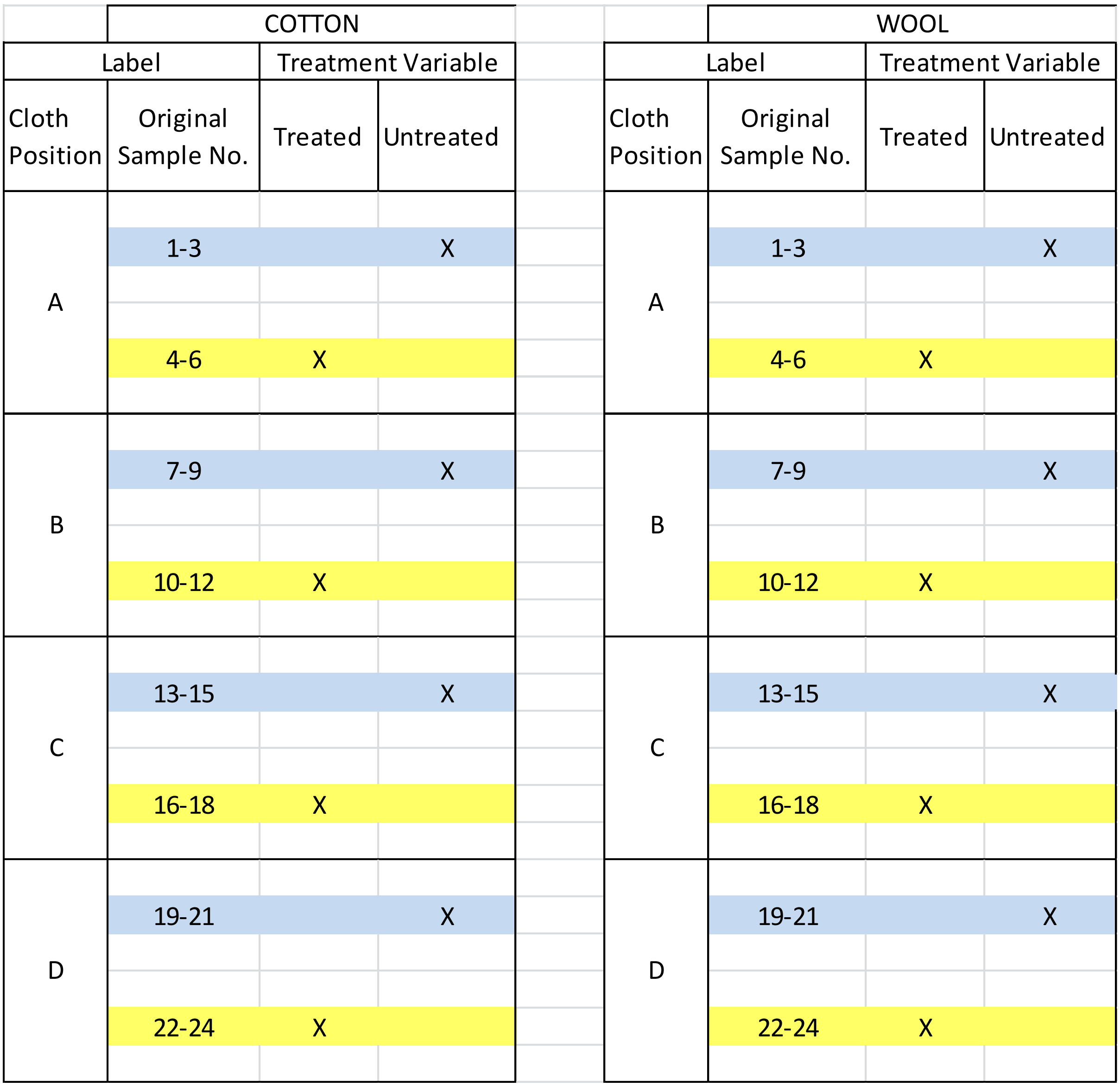
2.1.3. INDEPENDENT VARIABLES: SELECTION AND PROCEDURE

2.1.3.1. IMMERSION TREATMENT WITH D5

The immersion treatment was designed to be similar to conservation solvent cleaning in an open bath. At the same time, it was necessary to maximize time and resources, while controlling for the amount variables present.

Table 2.12 lists how the variable for D5 was applied across the sample population.

TABLE.2.2: SAMPLES TREATED WITH D5



	COTTON			
Label		Treatment Variable	
Cloth Position	Original Sample No.	Treated	Untreated
A	1-3		X
	4-6	X	
B	7-9		X
	10-12	X	
C	13-15		X
	16-18	X	
D	19-21		X
	22-24	X	

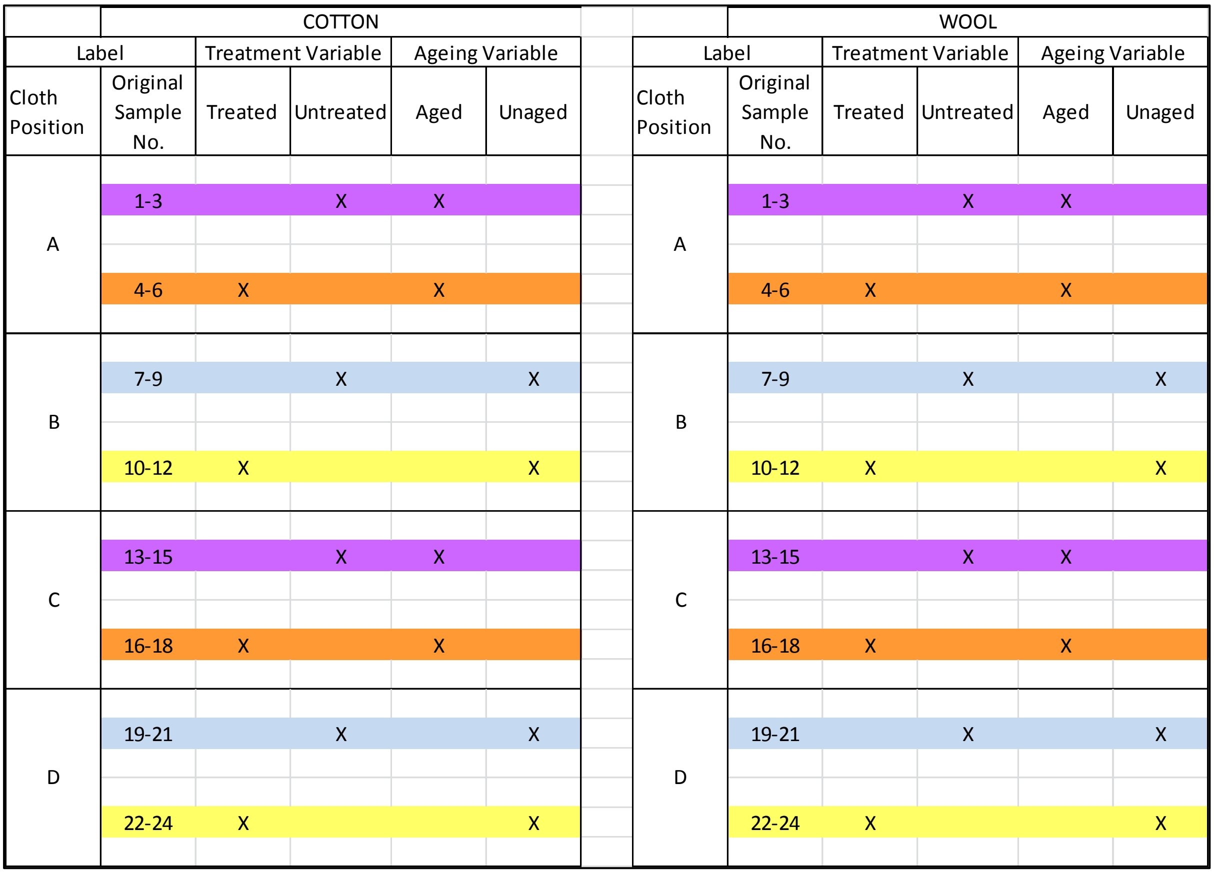
	WOOL		
Label		Treatment Variable	
Cloth Position	Original Sample No.	Treated	Untreated
A	1-3		X
	4-6	X	
B	7-9		X
	10-12	X	
C	13-15		X
	16-18	X	
D	19-21		X
	22-24	X	

Cotton samples A (4-6), B (10-12), C (16-18) and D (22-24) were placed in 1 Liter glass beakers and covered with 250 mL of undiluted D5 solvent, an amount sufficient to completely immerse each sample in the liquid. The samples were immersed for one hour. During this hour, gentle agitation was introduced every twenty minutes by stirring with a glass rod. After the hour, samples were removed from the beakers and blotted with paper towels. Then they were allowed to remain in the fume cupboard until dry. As noted, drying time was relatively lengthy for the samples, though this was not measured outright. After treatment, there appeared to be no difference between look and feel and there did not appear to be any dimensional change. The process was then repeated for the samples cut from wool: A(4-6), B (10-12), C (16-18) and D (22-24). These, too, appeared identical to untreated samples after the immersion process. No drying effect was detected in the feel of the samples.

2.1.3.2. ARTIFICAL AGEING

Artificial ageing was included as an independent variable in order to simulate the effects of the agents of deterioration that occur over an object's lifespan. As a tool which has been developed and used by conservators, it can involve exposure to high heat and relative humidity (RH), as well as ultraviolet (UV) light exposure, all environmental factors which have are known to catalyze degradation in artifacts over time. Artificial ageing by high temperature and RH were introduced to a selection of both treated and untreated samples, as shown in Table 2.13, after the completion of the immersion treatment .

TABLE 2.3



COTTON						WOOL					
Label		Treatment Variable		Ageing Variable		Label		Treatment Variable		Ageing Variable	
Cloth Position	Original Sample No.	Treated	Untreated	Aged	Unaged	Cloth Position	Original Sample No.	Treated	Untreated	Aged	Unaged
A	1-3		X	X		A	1-3		X	X	
	4-6	X		X			4-6	X		X	
B	7-9		X		X	B	7-9		X		X
	10-12	X			X		10-12	X			X
C	13-15		X	X		C	13-15		X	X	
	16-18	X		X			16-18	X		X	
D	19-21		X		X	D	19-21		X		X
	22-24	X			X		22-24	X			X

Untreated controls A(1-3) and C (13-15) and treated samples A(4-6) and C(16-18) of both cotton and wool were fastened to the upper rack of a laboratory oven with binder clips labeled with each sample number. The oven was set to maintain a continuous temperature of 60° C, a temperature well below the glass transition temperature (Tg) of wool and cotton,[49] but commonly used in Oddy tests.[50] A 250 mL beaker of deionized water was placed at the bottom of the oven to provide the water vapor necessary to create an elevated level of RH, though this level was expected to be variant throughout the duration of the accelerated ageing, and not strictly monitored.

The samples were left in the oven over seven days, which was the longest duration the experimental schedule would allow. Oven these seven days, the beaker was checked periodically and topped up with more deionized water as it got low.

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[49] Tg for cotton=160° C and Tg for wool=230° C, Tímár-Balázsy and Eastop, *Chemical Principles*, 16.

[50] J.A. Bamberger, et al., 'A Variant Oddy Test Procedure for Evaluating Materials Used in Storage and Display Cases', *Studies in Conservation* 44, no. 2 (1999): 87.

It was not possible to test the effects of accelerated ageing by UV exposure due to lack of resources. Though the duration of the RH aging was very short, far less than has been used in past studies,[51] it was thought that artificial ageing for even a short time may show some effect. If dramatic results were seen in treated samples with exposure over this short time, this would provide valuable information about D5 treatment.

All of the samples, aged and unaged, treated and untreated, were analyzed over a series of dependent variables in order to compare for differences between those that were treated and untreated.

2.1.4. ANALYSIS OF DEPENDENT VARIABLES

There were two criteria for the selection of analytical testing methods. The tests had to be practicable within the time and resources available. They also had to provide meaningful data on which a comparison of treated and untreated samples could be based. The techniques chosen included Fourier Transform Infrared Spectroscopy (FTIR) analysis, tensile strength testing, and Scanning Electron Microscopy (SEM).

2.1.4.1. ANALYSIS: ATR-FTIR

Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy (ATR-FTIR) was undertaken in order to determine if differences in molecular composition could be detected between the treated and untreated samples. It is a commonly-used method of analysis for organic materials in conservation science. The technique uses IR radiation to stimulate sample material and then measures the resulting response. IR radiation causes vibrations between the bonds of the molecules which make up the sample material. These vibrations can then be plotted onto a graphic spectrum. The objective of FTIR analysis in this experiment was to compare the spectra of treated samples against untreated controls to see if any differences could be detected that would indicate molecular change as a result of treatment.

The surface of each sample was analyzed in three “randomly” chosen locations[52]. For each testing location, contact was made between the sample and the Perkin Elmer® Universal Sampling Accessory.[53] Spectrum One software scanned each location 16 times and compiled these scans into an average spectrum by plotting % Transmittance against wavenumber cm-1, with wavenumber equaling the wavelength of IR exposure and % Transmittance equaling the resulting vibrations between the molecular bonds.

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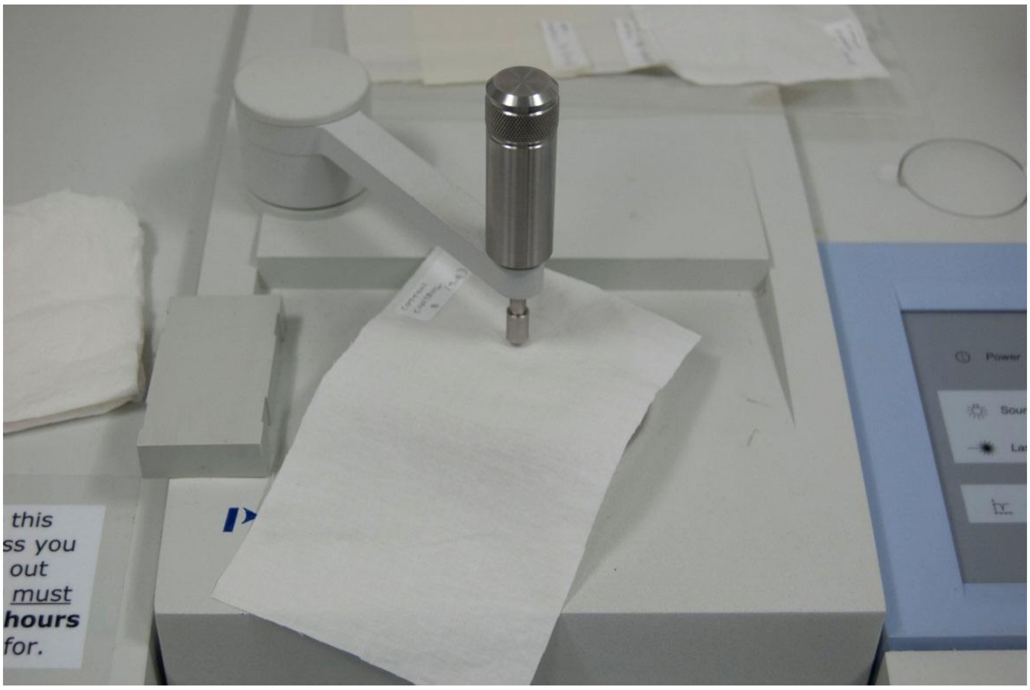
[51] N. Luxford and David Thickett,’Designing Accelerated Ageing Experiments to Study Silk Deterioration in Historic Houses’, *Journal of the Institute of Conservation* 34, no. 1 (2011): 115-127.

[52] Presented in quotes to indicate not true randomness, as the testing locations were chosen as randomly as possible while still facilitating ease of placement under the instrument probe.

[53] UATR DiComp™ crystal, composed of a diamond ATR with a zinc selenide focusing element.

FIGURE 2.4: FTIR ANALYSIS OF COTTON SAMPLE WITH UATR ACCESSORY



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2.1.4.2. ANALYSIS: TENSILE STRENGTH

Tensile strength testing was performed in order to detect whether a quantifiable difference could be observed in the physical properties of the samples. Tensile testing is capable of assessing:

a) fiber strength, measured by the amount of physical strain which can be applied before fiber breakage

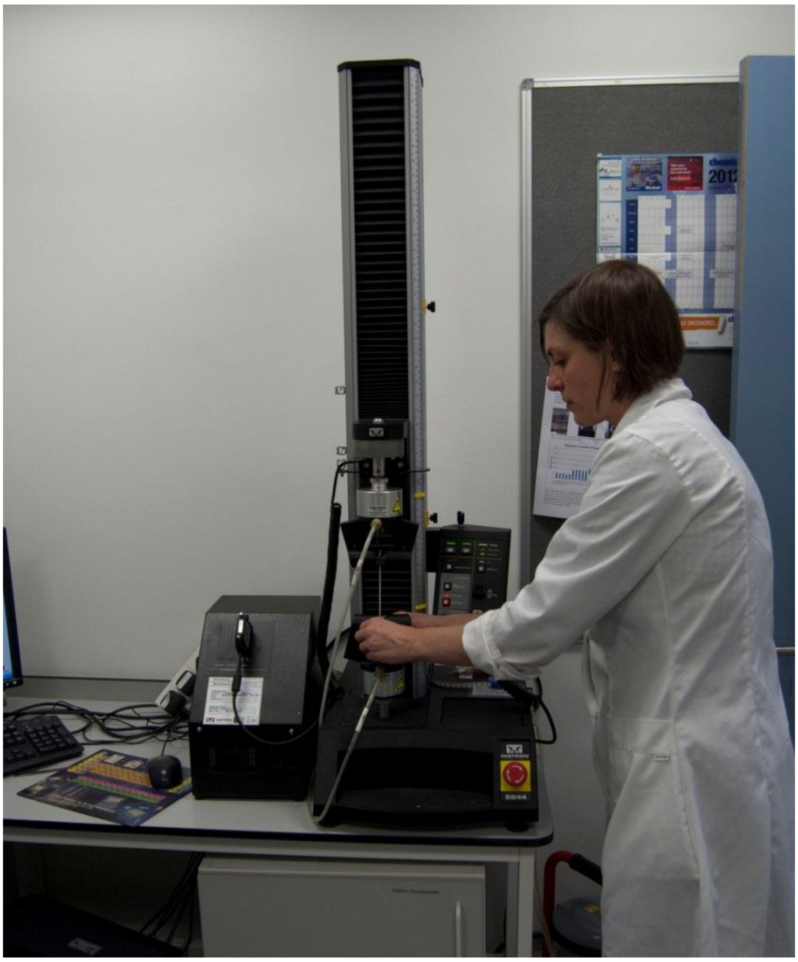
b) the amount of elongation exhibited by the fiber at the maximum strain load.

The tests were performed using an Instron$^{®}$ 5544 tensile testing machine running Bluehill$^{®}$ software to record results. For both cotton and wool, each sample A, B, C and D was cut into three test strip specimens, according to the templates shown in Figure 2.2. After the strips were cut from the original treatment samples, an excess of 10mm along the length remained to be retained for use as samples in SEM testing.

Each strip was placed vertically within the grips of the tensile tester at an initial length of 100 mm (the excess 40mm of length in each sample allowed area for contact with the grips), creating a testing area of 100mm x 25 mm. During the test, the Instron® apparatus fitted with a 1000N load cell stretched the fibers at a rate of 10mm per minute until the point of rupture.

The specimen length, sampling method, and test procedure was based on recommendations for industrial testing standards put forth by the British Standards Institute for determining the tensile properties of fabrics using the strip method.

FIGURE 2.5: PLACING THE SAMPLES BETWEEN THE GRIPS OF THE INSTRON(R) 5544 TENSILE TESTER



2.1.4.3. ANALYSIS: SEM

Treatment caused no macroscopic visible change to the samples. It was necessary to examine them at high magnification to determine if any microscopic differences could be detected between treated samples and untreated controls. This would help determine if any residues had deposited or if fibers appeared dessicated after treatment.

Scanning electron microscopy (SEM) was elected to provide elemental analysis of the samples in addition to high magnification imaging. Elemental analysis would provide information about the composition of the samples before and after treatment, while imaging would provide visual evidence of any change resulting from treatment.

Restraints on time and resources limited the number of specimens which could be analyzed in this trial, so it was decided to limit the field of testing to unaged samples. Four total specimens were tested: one each of untreated cotton, treated cotton, untreated wool, and treated wool.

Using a stereomicroscope, one yarn per sample, each under 1 mm in length, was taken and placed on a nickel stage. The stage was then placed in a Cressington 108/CarbonA carbon coating machine where the surface was sputter-coated with a relatively thin layer of carbon. After a baseline was run using a nickel standard, the stage with the carbon coated samples was placed in the vacuum chamber of the scanning electron microscope. (Model: JEOL® JSM-6480LV, running INCA software).

The samples were analyzed one at a time, each at low energy (25 kv) with a working distance of 10 nm. Seconday Electron Imaging (SEI) was used to take still images, captured at 500x magnification. Elemental analysis was performed with energy dispersive x-ray spectrometry (EDS), providing both graphic spectra and quantitative measurements of atomic composition.

2.2. STAGE 2: SOIL REMOVAL TESTING

In the second phase of the experiment, samples of standardized soiled fabrics were treated with D5. A total of two treatments was performed. Samples were assessed before and after treatement using a combination of colorimetry and ATR-FTIR spectroscopy.

2.2.1. TREATMENT ONE

2.2.1.1. SAMPLE SELECTION AND PREPARATION

In the soil removal stage of testing, samples were treated in an open immersion similar to the treatment of samples in the textile substrate stage. In this case, standardized soiled fabrics, acquired from Materials Research Products, LTD (see Appendix VI), were chosen to make up the sample population.

The obvious disadvantage to the use of standardized soiled fabric is that both the fabric itself and the soiling are new. By using new fabric, the tests would not be able to fully replicate the conditions of age and soiling in historic textiles. The standardized soiled samples were, however, thought to provide the following advantages:

- Soiling would be more uniform across samples than that applied by hand or in samples with “found” soiling.
- The composition of the soiling would be a known quantity

Both the cotton and wool were purchased soiled with a combination of olive oil and carbon black. This combination of soils was chosen from a host of soiling options and was selected for economy, availability across fabric types, and for the composition of the soiling itself. It was thought that carbon black and olive oil would allow for testing D5's effectiveness on two very different types of soiling in a single test. One square meter of each fabric was acquired for sampling along with a meter square of each cotton and wool fabric of the same fabric make, unsoiled. The soiled fabric was a dark gray in color, significantly darker than the corresponding unsoiled fabric, as shown in Figures 2.2.1 and 2.2.2..

FIGURE 2.6: COTTON SAMPLES OF THE SAME FABRICS, UNSOILED AND SOILED

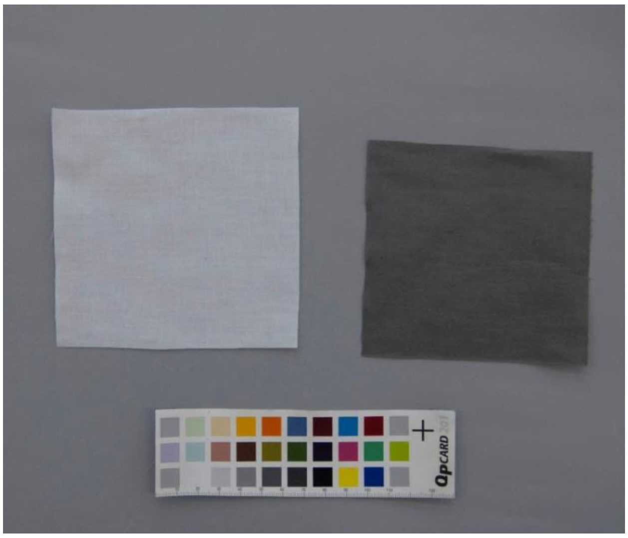
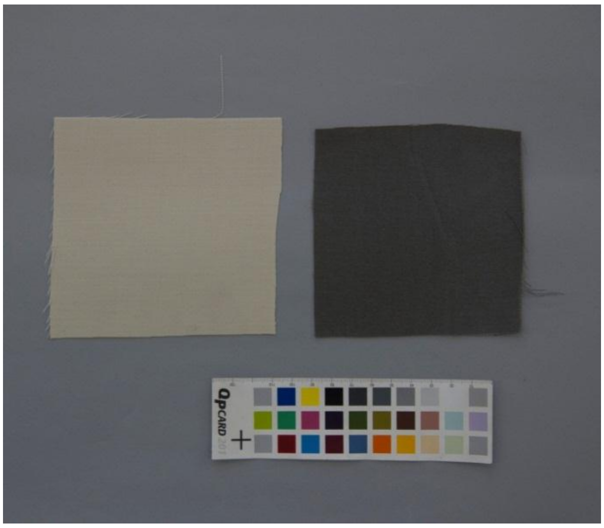


FIGURE 2.7: WOOL SAMPLES OF THE SAME FABRIC, UNSOILED AND SOILED

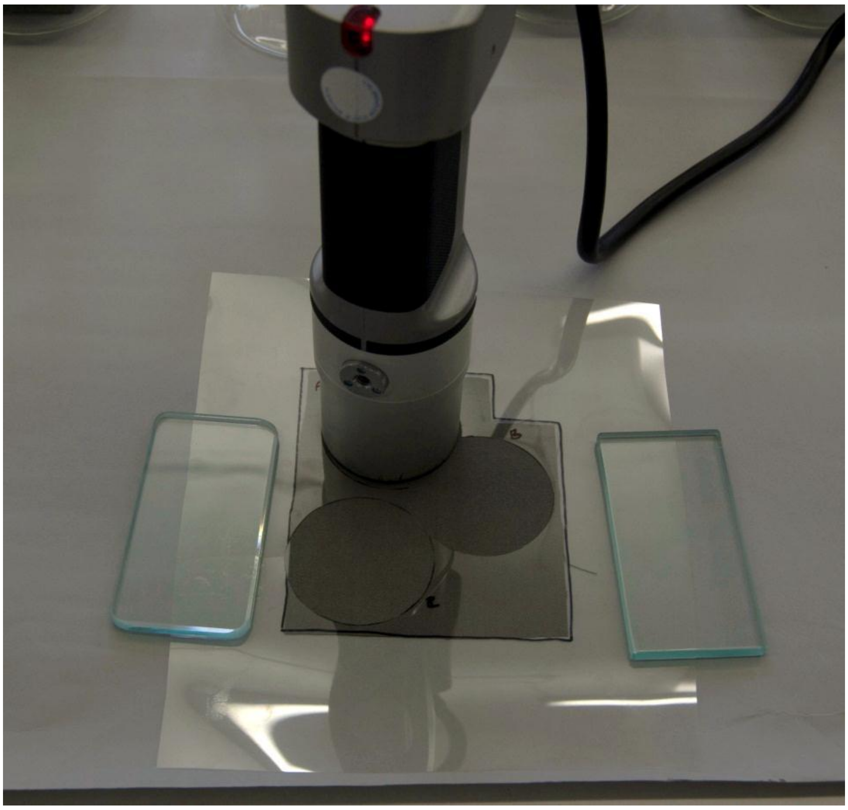


Soiled fabrics felt slightly grubby to the touch in comparison to their unsoiled counterparts, with some minor transfer to the hand when handling during sample preparation. This soil transfer was limited, however, as the soiling seemed generally quite ingrained to the fabric and the overall coloration of the soiling uniform. This ingrained quality to the soiling was considered very appropriate for the tests at hand, which aimed to show how well D5 could overcome the forces which adhere soils to substrates.

Two samples measuring approximately 10mm x 12mm were cut from the soiled cotton and two samples of the same size were cut from the soiled wool.

Color value readings of all samples were taken before treatement with a Konica-Minolta® C-Series Chromameter. As the identical locations would need to be tested after treatment, a template made from Melinex® was used to ensure constistency in testing locations. A notch was also cut from the sample before handling to indicate which side had been tested before treatment.

FIGURE 2.8: USING A MELINEX(R) TEMPLATE TO POSITION THE CHROMAMETER PROBE OVER THE NOTCHED SAMPLE



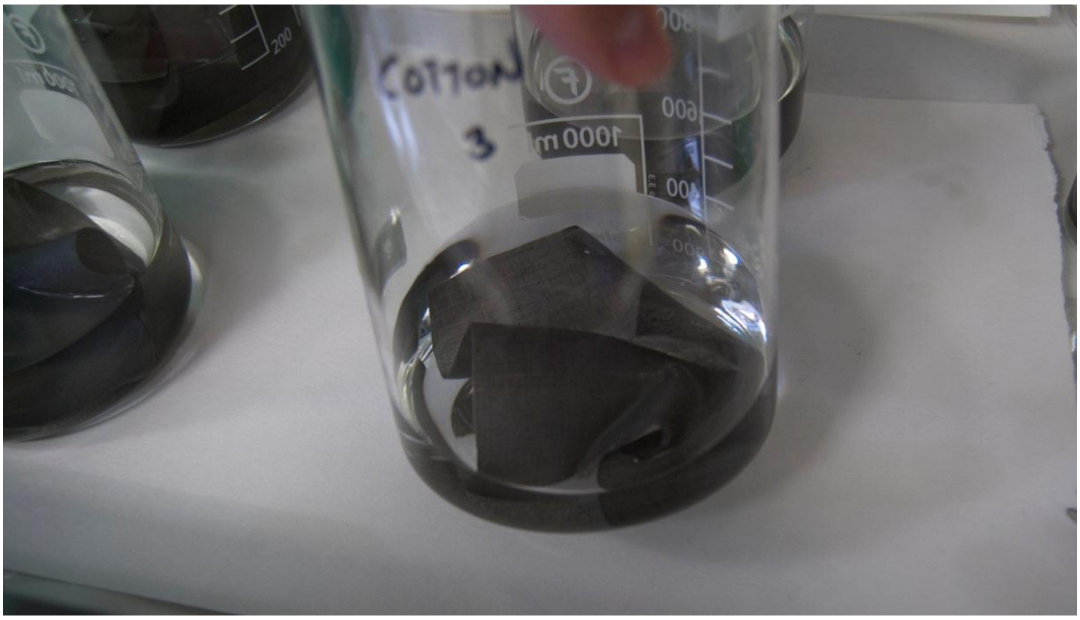
The Konica Minolta chromameter C-series is fitted with a probe which outputs light from a pulsed xenon arc lamp onto the surface of the sample being tested.. The probe measures incident and reflected light in a double-beam feedback system with silicon photocells which match the CIELAB Standard Observer Response.[54] CIELAB is discussed fully in Section 3.1.2.

2.2.1.2. IMMERSION TREATMENT

The treatment carried out for soil removal testing followed a similar procedure to the treatment performed for textile substrate testing. In this trial, cotton and wool were treated at the same time. Samples were placed in 1L glass laboratory beakers and covered with 250 mL undiluted D5. They were allowed to soak for an hour, with gentle agitation every twenty minutes.

During treatment, no dark-colored soiling was observed to come into solution with D5; the solvent remained clear throughout the immersion process, as shown in Figure 2.2.4 Agitation was increased with no change in effect. Samples were left to dry overnight.

FIGURE 2.9: IMMERSION TREATMENT OF SOILED COTTON SAMPLE



2.2.1.3. ANALYSIS

Once the samples were dry, it was surprising to see that they had significantly lightened in color, despite no visual evidence of soil release during treatment. Chromameter readings were taken, using the same testing locations and procedure as before treatment.

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[54] 'Chromameter CR-300/CR-310/CR-321/CR331/CR-331C Instruction Manual', Minolta Co., Ltd, http://www.konicaminolta.com.cn/instruments/download/manual/pdf/CR-300.pdf (accessed 25 July 2012).

It was quite clear, considering the color change, that D5 treatment had removed some type of soiling from the samples. It was thought that this unknown substance was not detected during treatment because it was thoroughly miscible with D5 and also colorless. Even though it was without color, it was able to darken the samples, as an oil might do.

Considering the nonpolar structure of the D5 molecule, it was thought that whatever was removed in the treatment was also nonpolar. It was hypothesized that it might be the olive oil; it would likely have a nonpolar composition as well. It was decided to test this hypothesis with an additional round of soil removal tests, augmented by FTIR analysis. FTIR was elected to assess whether a difference in the molecular character of the sample's surface could be detected before and after treatment: if D5 was indeed removing the olive oil, then perhaps it was possible to observe a change in the spectra which would reflect this. Further interpretation might be made through comparison with reference spectra for olive oil.

2.2.2. TREATMENT TWO

2.2.2.1. SAMPLE SELECTION AND PREPARATION

As in the initial soil removal testing treatment, two samples each of cotton and wool were taken from the pre-soiled test fabrics. Before the second immersion treatment, inquiry was made as to the standardized soiling process.

Information gathered from Materials Research Products LTD revealed that the test fabrics used in this study are treated to soiling in an immersion process which theoretically renders both sides of the fabric soiled in equal concentration.[55] Yet there was speculation that due to settling one side might end up more soiled than the other. In an attempt to determine whether this was the case, chromameter readings of the presoiled fabric were taken before sampling to see if color variation might exist between sides which could indicate variation in soiling concentration. Readings were taken from ten random locations on each either side of the cotton and wool pre-soiled fabrics, concentrated in the area where the samples would be cut. These readings revealed some color variation between the two sides with one side consistently darker than the other on both fabrics. The side that was folded *inward* in the vacuum-packed shipping envelope was the darker of the two for both fabrics.

Just as in the first round of soil removal tests, two samples each were cut from the pre-soiled cotton and wool. Samples were cut in the same shape and size as first rounds, however this time they were taken so that the darker soiled side of each bolt would be the "front" side, which was then distinguishable by the position of the notch on the top right corner. Though the samples in the first

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[55] Personal email communication with John Byrne, Materials Research Products LTD representative, 30 May, 2012.

round of testing had a distinguishable “front”, the notch had been created after cutting the samples out and handling them, and it was not possible to know which side had actually been used.

2.2.2.2. IMMERSION TREATMENT

Treatment was performed on the four samples in the same manner as the first round of soil removal testing: the samples were placed in glass 1 L beakers, covered in 250 mL D5, immersed for an hour, with gentle agitation every twenty minutes. After an hour, the samples were removed and allowed to dry. During treatment, again no visible soiling removal was observed.

2.2.2.3. ANALYSIS

As in the first round of tests, samples had significantly lightened with treatment. Chromameter readings were taken as before . In this round of tests, ATR-FTIR spectroscopy was performed. The method used was identical to that described in Stage 1 of the experimentation.

3. CHAPTER THREE: RESULTS AND DISCUSSION

This chapter presents the results from both stages of the experimental phase of the research: **Stage 1: Textile Substrate Testing**, and **Stage 2: Soil Removal Testing**. It begins with a summary of the two stages, listing the both the independent variables controlled in the experimental procedure and the dependent variables measured in the subsequent analysis. The analytical results are then considered one by one, including a summary of each test, a summary of the sample populations used to generate the data, presentation of the data, and discussion of the results.

When appropriate, the raw data has been compiled into mean values to create a statistical picture of the results, tables have been used to synopsize collected data, and graphic representation has been used to explore relationships between the dependent and independent variables. Exploring these relationships will allow inferences to be made into cause and effect resulting from treatment of textiles with D5 in order to address the overall aim of the experimental research.

3.1. SUMMARY OF TESTING STAGES

3.1.1. TEXTILE SUBSTRATE TESTING

In **Stage 1: Textile Substrate Testing**, the research objective was to show how D5 treatment would affect a woven textile. The procedure for treatment was an immersion bath with gentle agitation. The samples which populated the experiment included identically-sized pieces cut from pre-scoured cotton lawn and pre-scoured wool delaine fabrics. **The independent variables to which these samples were subjected included:**

- **Treatment with D5** or control.
- **Artificial ageing** (of both treated and untreated samples) or control.
- For Stage 1: Tensile Strength Testing only: **Weave orientation** as determined by sampling location.

Dependent variables are the presumed responses to the independent variables selected for the sample populations. They are what subsequent analysis aims to measure. All dependent variables tested in Stage 1 are summarized in Table 3.1.1 along with the corresponding testing method.

TABLE 3.1: LIST OF DEPENDENT VARIABLES MEASURED IN TEXTILE SUBSTRATE TESTING STAGE AND CORRESPONDING METHODS OF ANALYSIS

Dependent Variable	Method of Analysis
Molecular composition of sample surface	ATR-FTIR generated spectra
Tensile strength	Tensionometer measurements
Tensile elongation	Tensionometer measurements
Elemental composition of sample surface	SEM – elemental analysis
Appearance of sample surface at high magnification and resolution	SEM, Secondary Electron Imaging (SEI)

3.1.2. SOIL REMOVAL TESTING

In **Stage 2: Soil Removal Testing**, the research objective was to ascertain the effectiveness of treatment with D5 at removing soiling from textiles. The samples populating this experiment consisted of identically sized pieces cut from standardized pre-soiled wool and cotton fabrics. The soiling was comprised of a combination of soot and olive oil.

The sole independent variable in this stage was Treatment or Non-treatment with D5. The dependent variables are listed in

TABLE 3.2: LIST OF DEPENDENT VARIABLES MEASURED IN STAGE 2: SOIL REMOVAL TESING AND CORRESPONDING METHODS OF ANALYSIS

Dependent variable	Method of Analysis
CIELAB color space values	Chromameter readings
Molecular composition of sample surface	ATR-FTIR generated spectra

3.2. RESULTS OF TESTING STAGES

3.2.1. TEXTILE SUBSTRATE TESTING

The independent variables possible for each sample in Stage 1 included immersion treatment with D5 and/or artificial ageing by exposure to heat and high RH. Each sample from the overall population falls into one of four independent variable groups. For the purposes of presentation and comparison of the results, these groups have been designated the following:

- **Group 1**: No immersion treatment with D5/No artificial ageing
- **Group 2**: Immersion treatment with D5/No artificial ageing
- **Group 3**: No immersion treatment with D5/Artificial ageing
- **Group 4**: Immersion treatement with D5/Artificial ageing

3.2.2. TEXTILE SUBSTRATE TESTING: ATR-FTIR SPECTROSCOPY

3.2.2.1. SUMMARY

ATR-FTIR spectroscopy was performed to assess differences in the molecular composition of the samples in the different variable groups. This method of analysis generates data in the form of a graphic spectrum. An IR spectrum provides a record of the vibrations of the molecular bonds in the sample material produced when exposed to infrared radiation.

Both quantitative and quantitative analysis can be used to interpret spectra generated by ATR-FTIR spectroscopy. Qualitative analysis provided the most straightforward and suitable technique for the purposes of this experiment.

Qualitative spectral analysis involves comparing spectra gathered from trials against known references. In the case of this experiment, the spectra generated from the samples in independent variable Group 1 (Untreated/Unaged cotton lawn and wool delaine), would serve as the reference. These samples comprised the control group during the treatment and ageing phase of the experiment.

Comparison of the variable and control samples would be used point to relationships of cause and effect. For example: would a D5-treated sample of wool or cotton display different spectra than an untreated sample? If so, then treatment with D5 could be presumed to have affected the molecular character of the fiber surface, either by leaving a deposit on the textile surface, or by altering the chemical structure of the fibers themselves. If no change in spectra could be distinguished, then it would indicate that either D5 had not affected the substrate, or had done so in such a small quantity that it would be undetectable by FTIR analysis.

It was thought further that artificial ageing of the material would, through exposure to the catalyzing effect of high heat and high RH, begin to approximate conditions which might provoke changes in the material’s composition. The ageing treatment was admittedly brief in its application, however it might give some small indication of how treated samples would be affected by treatment with D5 over time.

The two sample specimens in each independent variable group were analyzed in three randomly chosen locations on the surface of each sample. For each testing location, the Spectrum One software scanned the area and plotted values of %Transmittance against Wavenumber (cm-1).

3.2.2.2. SAMPLE POPULATION

Table 3.2.1 summarizes the sample population for the ATR-FTIR testing. A total of 16 sample specimens were scanned in three locations on each sample, making a total forty-eight total spectra, 6 from each variable group for both cotton and wool.

TABLE 3.3. SAMPLE POPULATION FOR ATR-FTIR TESTING

Material	**Variable Group**	**Independent Variable: D5 Immersion Treatment**	Independent Variable: Ageing	No. of Sample Specimens in Variable Group	No. of FTIR testing locations per specimen	Total No. of FTIR spectra generated per variable group
Cotton	1	Untreated	Unaged	2	3	6
Cotton	2	Treated	Unaged	2	3	6
Cotton	3	Untreated	Aged	2	3	6
Cotton	4	Treated	Aged	2	3	6
Wool	1	Untreated	Unaged	2	3	6
Wool	2	Treated	Unaged	2	3	6
Wool	3	Untreated	Aged	2	3	6
Wool	4	Treated	Aged	2	3	6
			TOTAL SAMPLE SPECIMENS	16	TOTAL SPECTRA	48

3.2.2.3. DATA

The raw data from each of the 48 spectra was then exported to Microsoft Excel® in an ASCII file. Once in Excel®, the raw data was plotted, using a scatter graph.

To get an overall comparison of the variable groups, an average spectrum compiled from the six readings in each group, by taking the mean of the six % Transmittance values recorded for each wavenumber data point. In Figures 3.2.1 and 3.2.2, the mean graphs for cotton and wool are presented, with the spectra from all variable groups overlaid.

FIGURE 3.1: COMPARISON OF AVERAGE SPECTRA FOR ALL VARIABLE GROUPS: COTTON

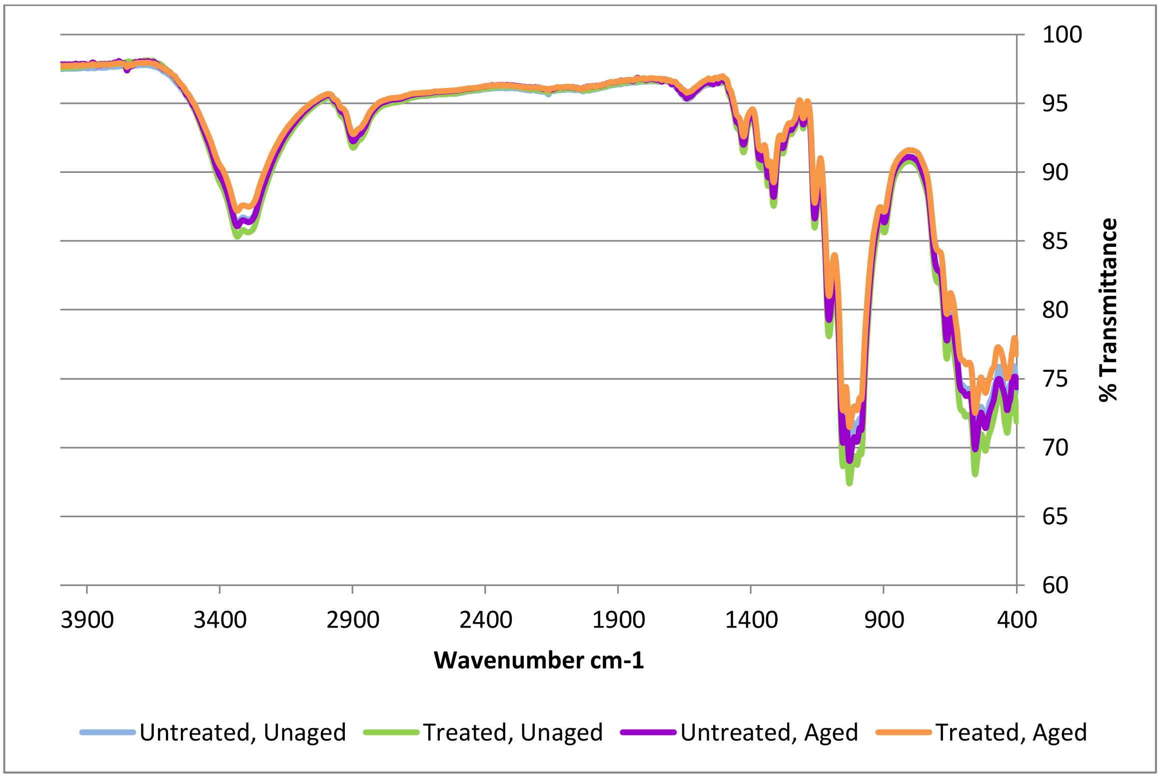
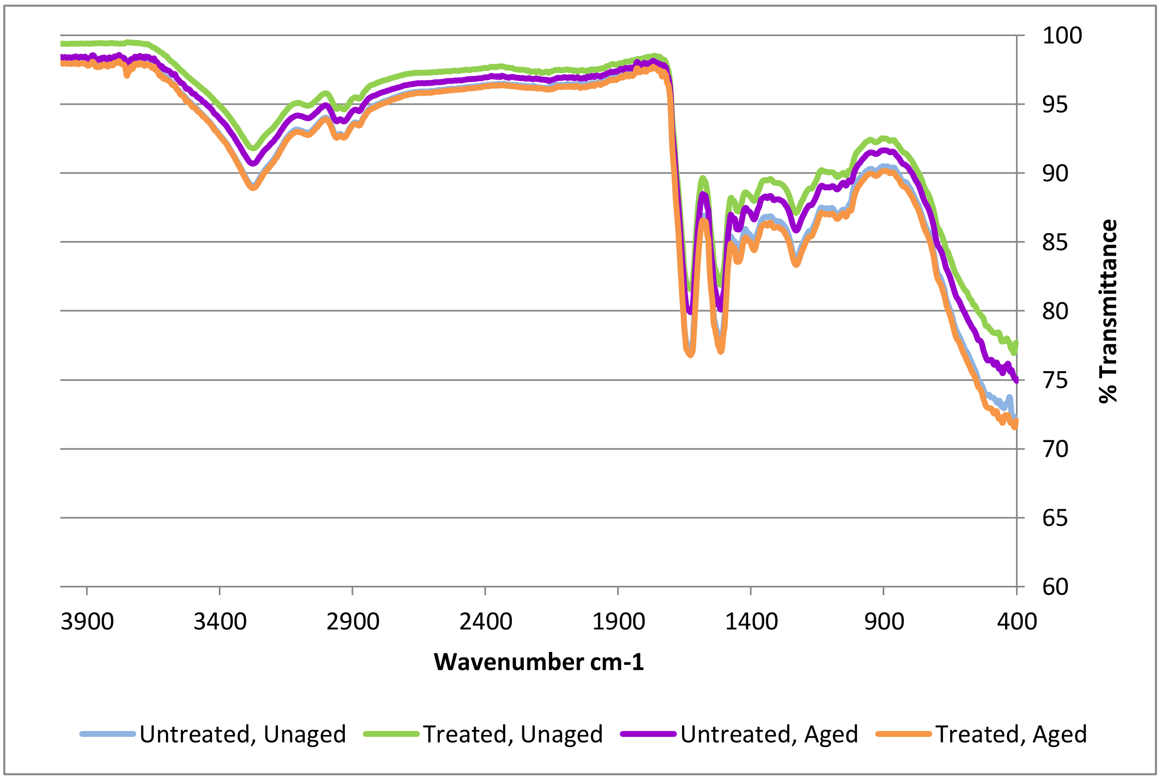


FIGURE 3.2: COMPARISON OF AVERAGE SPECTRA FOR ALL VARAIBLE GROUPS: WOOL



3.2.2.4. EVALUATION

When analyzing IR spectra qualitatively, three attributes of the spectral bands which make up the graphic image are examined: band frequency, band shape, and band intensity.[56] Band frequency relates to the exact position of the spectral peaks along the x-axis. Band shape refers to the shape of the slope between the various peaks, their angle and symmetry. Band intensity relates to the position of the spectral peaks relation to the y axis. For meaningful interpretation in this study, the qualitative analysis requires that comparison be made between treatment groups (Groups 2, 3, and 4) and the control (Group 1).

Discrepancies in band intensity are widespread in the individual results and in the average spectra of each sample group presented here. It is thought that these differences can be accounted for by variation in sample preparation, and reflect relatively minor differences in molecular composition between the samples.[57]

All of the spectra can be compared against reference spectra for cotton and wool from other sources. One example of comparison spectra is included in Appendix VII.

Considerable overlap in the spectra shown in the two figures indicates a general consistency of results, but also makes interpretation of minute differences more difficult. Because band intensity carries less interpretive weight when examining IR spectra qualitatively, it is possible to offset the spectra without compromising the interpretation.

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[56] M.R. Derrick, et al., *Infrared Spectroscopy in Conservation Science*, (Los Angeles, 1999), 83-84.
[57] Derrick, *Infrared Spectroscopy*, 83-84.

FIGURE 3.3: COMPARISON OF AVERAGE SPECTRA FOR ALL VARIABLE GROUPS: COTTON, OFFSET

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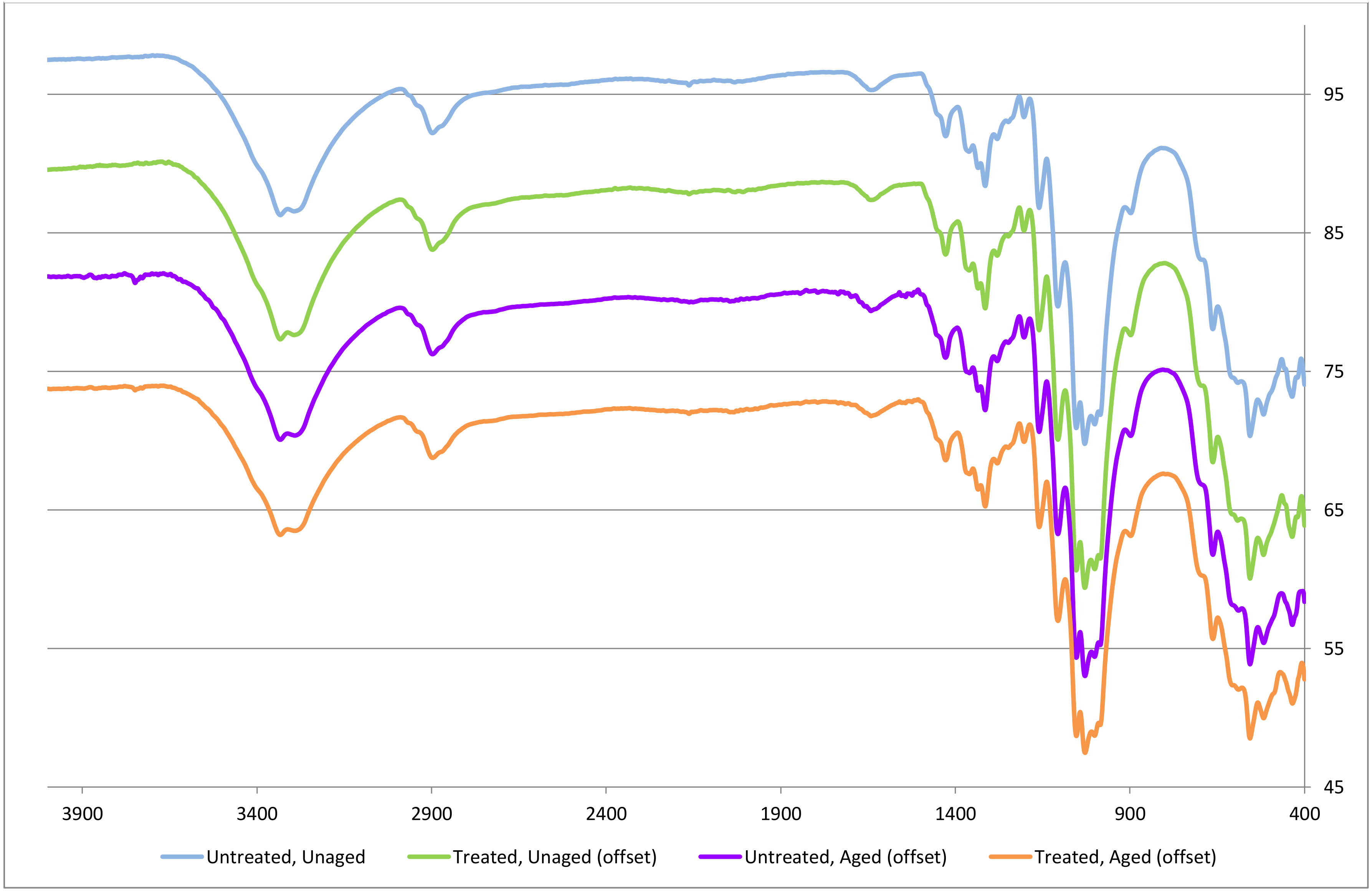

FIGURE 3.4: COMARISON OF AVERAGE SPECTRA FOR ALL VARIABLE GROUPS: WOOL, OFFSET

Even when offset, the detail of the spectral peaks appears generally consistent. The most significant comparison to make is between the treated and untreated samples within the same variable groups for ageing, comparing Group 1 against Group 2 and Group 3 against Group 4. When these are compared, the spectra across all frequencies show similarity, therefore it must be concluded that no difference in molecular composition can be detected between treated and untreated samples. If any deposition of material on the surface or change in molecular structure of the surface has occurred with treatment, it was in too small a quantity to be detected by this analysis.

Subtle disparity in the spectra does become evident when comparing the independent variable for ageing (unaged v. aged, both untreated and unaged v. aged, both treated) for both fabric types, located in the area between wavenumbers 4000 and 3500 cm-1

Changes between 4000 and 3500 cm-1 can relate to hydrogen bonding activity, but the structure underlying that activity can be difficult to assess,[58] especially without other significant changes in the spectra to cross-reference. The root cause, however, can be interpreted as conditions of heat and RH ageing.

3.2.2.5. NOTE ON ATR-FTIR RESULTS FOR WOOL

Obtaining accurate IR spectra for Group 2 (Treated, Unaged) wool samples required two rounds of testing. In the initial round of the ATR-FTIR analysis of these groups, large discrepancies in band frequency and intensity were noted between the treated and untreated samples as the tests were being administered. The untreated, unaged samples closely resembled reference spectra for wool (see Appendix VII), while the treated, unaged samples were very dissimilar. Though average spectra for cotton had not yet been compiled through calculation, the overall observation during cotton analysis was a general consistency in individual spectra between treated and untreated samples.

Considering these observations, it was thought that the differences arising between treated and untreated unaged wool samples at the time of testing were caused by one of two situations:

1) Treatment with D5 affected the molecular structure of the surface of the wool
(but not cotton) fibers
2) The solvent had not fully evaporated and was still present in the textile during testing, but would, given more time, evaporate away.

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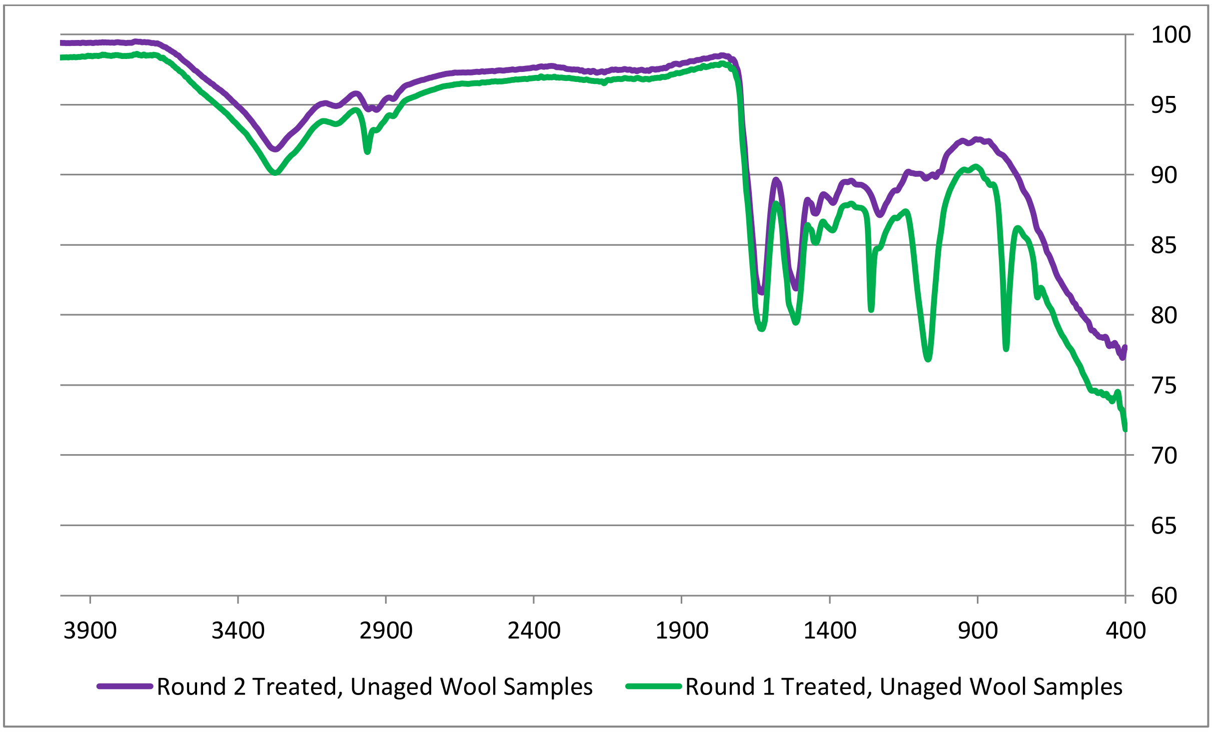
[58] J. Coates, 'Interpretation of Infrared Spectra, A Practical Approach', In *Encyclopedia of Analytical Chemistry*, R.A. Meyers, ed. 10815-37, (Chichester, 2000).

It was reasonable to suspect the latter considering the testing circumstances: to maximize time during the experimentation process, tests on wool unaged samples (Groups 1 & 2) had been performed immediately after assessing the samples as dry to the touch. Groups 3 and 4 were already in the ageing chamber and would be tested later. For the Group 2 wool samples, the time between treatment and testing amounted to a little over four hours. Due to the likelihood that the solvent had not completely evaporated in the short time frame given, it was decided to leave these samples overnight and test them again.

The next day, the testing process was repeated for the Group 2 samples. When the analysis was repeated, the readings were virtually identical to the untreated Group 1 wool samples. The second round of FTIR data was used in offset spectra for wool. It was apparent that the solvent had not yet evaporated when the first round testing took place. This provided an opportunity to compare readings from the solvent-ridden samples to reference spectra for D5.

FIGURE 3.5: COMPARISON OF AVERAGE SPECTRA OF WOOL GROUP 2 SAMPLES FROM 2 ROUNDS OF TESTING



A reference spectrum for Decamethylcyclopentasiloxane was obtained from the online spectral database maintained National Institute for Standards and Technology[59] as a JCAMP file. The JCAMP

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[59] Coblentz Society, Inc., ‘Evaluated Infrared Reference Spectra’ in *NIST Chemistry WebBook, NIST Standard Reference Database Number 69*, P.J. Linstrom, et al., eds., National Institute of Standards and Technology, Gaithersburg MD, 20899, http://webbook.nist.gov, (accessed 06 May 2012).

file was then exported into Microsoft Excel® and plotted to allow for direct comparison with the average spectrum compiled from the solvent-impregnated samples.

FIGURE 3.6: REFERENCE SPECTRA: D5

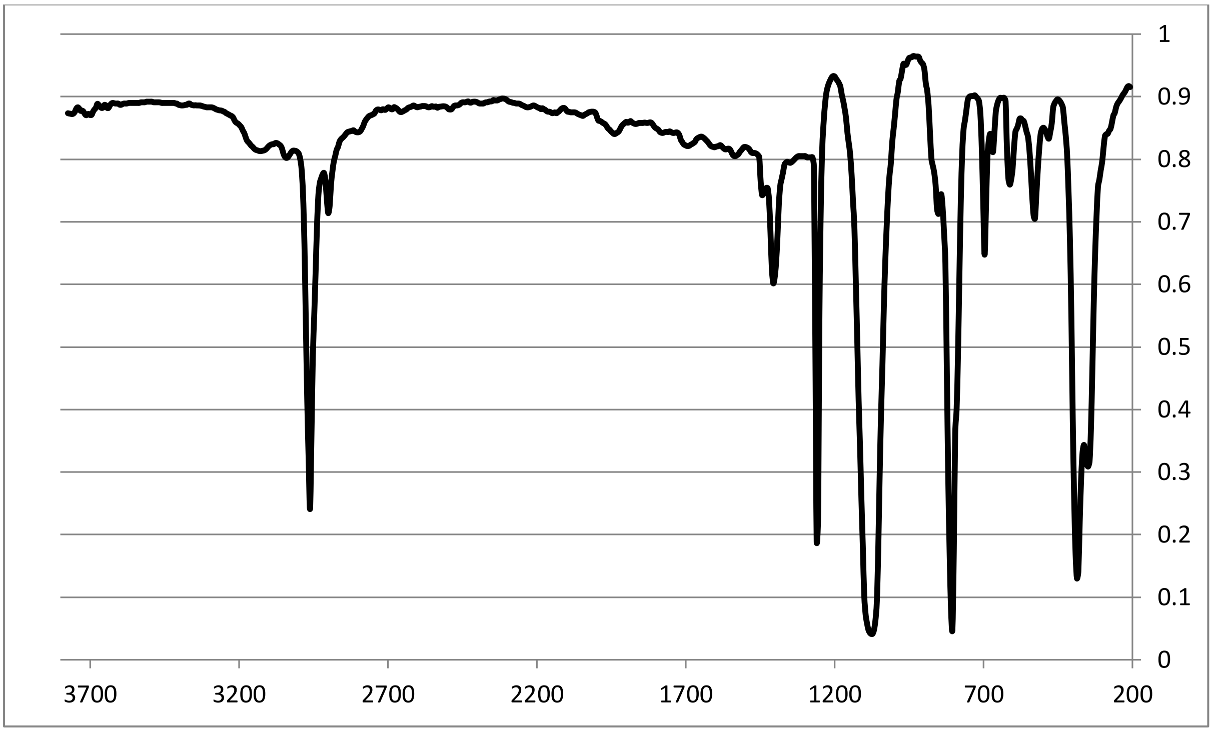

FIGURE 3.7: AVERAGE SPECTRA OF SOLVENT-RIDDEN SAMPLES

There are directly related peaks of similar shape in several areas, especially around 1250 cm-1, between 1100 and 100 cm-1, and at 800 cm-1, the fingerprint region of the spectra. The fingerprint region generally more useful for confirmation and spectral comparison than direct identification, [60] and in this instance it does relate the sample to the reference.

It is reasonable to conclude that the ATR- FTIR in the initial readings detected D5 on the sample surface. These results are notable for several reasons. First, that D5 on textile samples is indeed detectable with ATR-FTIR spectroscopy. This highlights the fact that no trace was found in the treated samples where the solvent had evaporated completely. The fact is also underscored that D5 may not be fully evaporated from a textile even if it feels dry to the touch, which can inform how it should be handled if used in practice.

3.2.3. TEXTILE SUBSTRATE TESTING: TENSILE STRENGTH

3.2.3.1. SUMMARY

FTIR analysis provided qualitative information regarding the effects of D5 on the molecular composition of the textile surface. Further examination was indicated in order to determine changes in the physical properties in the substrate. Tensile testing would also provide a quantifiable approach to testing, as the data gathered could be interpreted through numerical relationships rather than subjective visual analysis.

In the tests, samples were subjected to tensile stress as the grips of the tensile tester increased the length of the material to the point of rupture. Material under stress undergoes deformation, and in the case of tension, the deformation that occurs is termed elongation. In tensile testing the applied force is measured along with the elongation of the material in order to get an overall picture of the physical properties of a tested material.

3.2.3.2. SAMPLE POPULATION

As in the FTIR analysis, the independent variables affecting the condition of samples at the time of instrumental analysis included treatment or non-treatment with D5, artificial ageing or no artificial ageing. However, in this phase of testing, each sample’s orientation created an additional independent variable which would affect the results. The location of the sample’s origin, whether oriented in the warp or weft direction when tested, would have an impact on the tensile strength and elongation results of the sample tested.

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[60] Derrick, *Infrared Spectroscopy*, 94.

The tensile testing required dividing of each sample into three identical strips (as described in Section 2.2.4.2, Tensile Strength). Each sample from the treatment stage yielded three specimens for tensile testing.

TABLE 3.4: SAMPLE POPULATION FOR TENSILE TESTING

Reference	**Material**	**Treatment Variable**	**Ageing Variable**	**Weave Orientation Variable**	**No. of Sample Specimens**
Cotton: Group 1 Warp	Cotton	Untreated	Unaged	Warp	3
Cotton: Group 2 Warp	Cotton	Treated	Unaged	Warp	3
Cotton: Group 3 Warp	Cotton	Untreated	Aged	Warp	3
Cotton: Group 4 Warp	Cotton	Treated	Aged	Warp	3
Cotton: Group 1 Weft	Cotton	Untreated	Unaged	Weft	3
Cotton: Group 2 Weft	Cotton	Treated	Unaged	Weft	3
Cotton: Group 3 Weft	Cotton	Untreated	Aged	Weft	3
Cotton: Group 4 Weft	Cotton	Treated	Aged	Weft	3
Wool: Group 1 Warp	Wool	Untreated	Unaged	Warp	3
Wool: Group 2 Warp	Wool	Treated	Unaged	Warp	3
Wool: Group 3 Warp	Wool	Untreated	Aged	Warp	3
Wool: Group 4 Warp	Wool	Treated	Aged	Warp	3
Wool: Group 1 Weft	Wool	Untreated	Unaged	Weft	3
Wool: Group 2 Weft	Wool	Treated	Unaged	Weft	3
Wool: Group 3 Weft	Wool	Untreated	Aged	Weft	3
Wool: Group 4 Weft	Wool	Treated	Aged	Weft	3

3.2.3.3. DATA

Plotting the maximum load along an x-axis and elongation along the y-axis results in a will result in a modulus, or curve, characteristic to each material. Typically, cotton will bear a higher load than wool, but wool will elongate considerably before it breaks.

The purpose of the analysis was to determine if any changes in tensile properties could be observed between treated and untreated variable groups. The two numeric values which would inform this were maximum load at rupture (measured in N) and elongation at maximum load (measured in mm).

In order to synthesize this information in a way that can be interpreted, the average from the three replications each value for have been presented in a column graph, providing side-by-side comparison of strength and fiber elongation properties of the samples from each variable group.

Averages were compiled from the raw data exported recorded by the Bluehill® software tests and exported into Excel®.

FIGURE 3.9: AVERAGE MAXIMUM LOAD TAKEN FROM THREE COTTON SPECIMENS: NOTING STANDARD DEVIATION

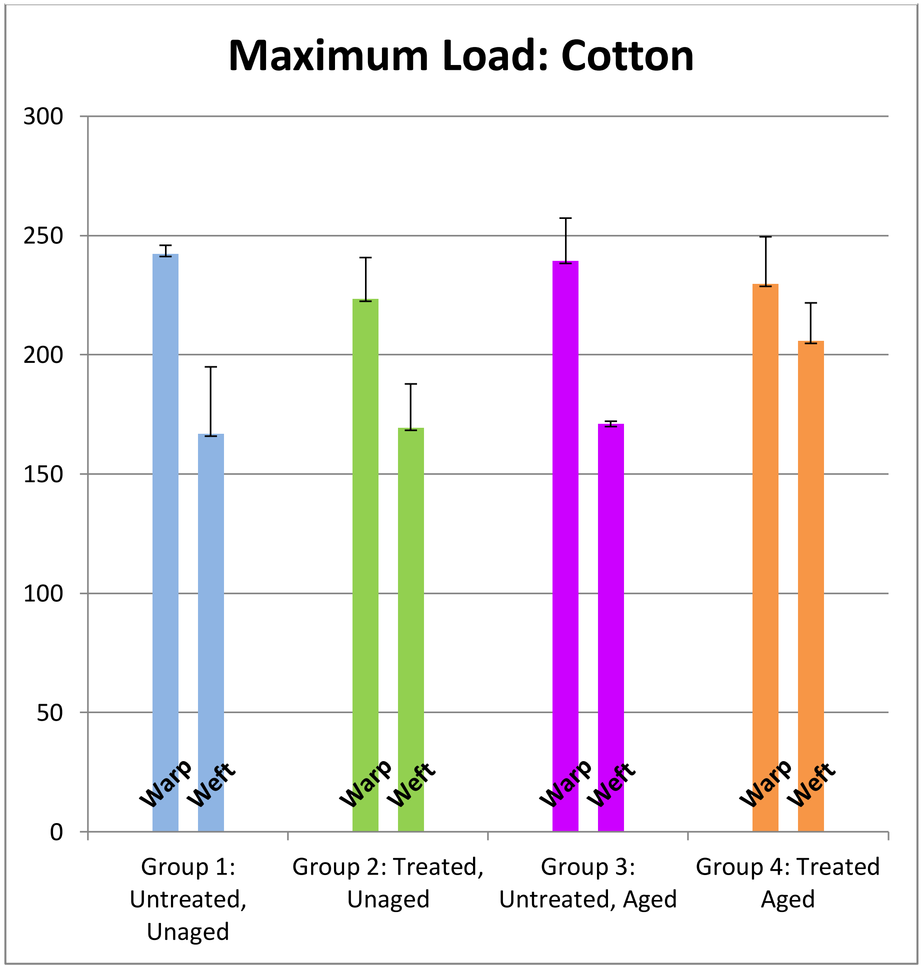


FIGURE 3.8: AVERAGE EXTENSION AT MAXIMUM LOAD TAKEN FROM THREE COTTON SPECIMENS: NOTING STANDARD DEVIATION

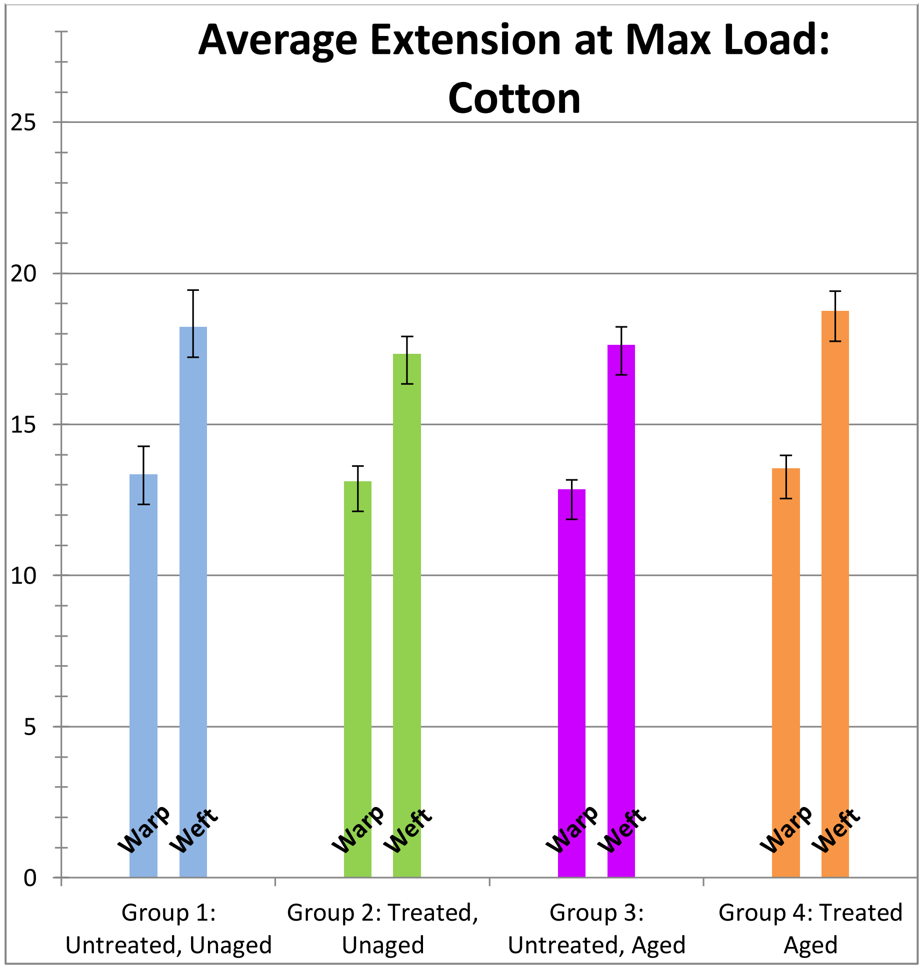


FIGURE 3.11: AVERAGE MAXIMUM LOAD TAKEN FROM THREE WOOL SPECIMENS: NOTING STANDARD DEVIATION

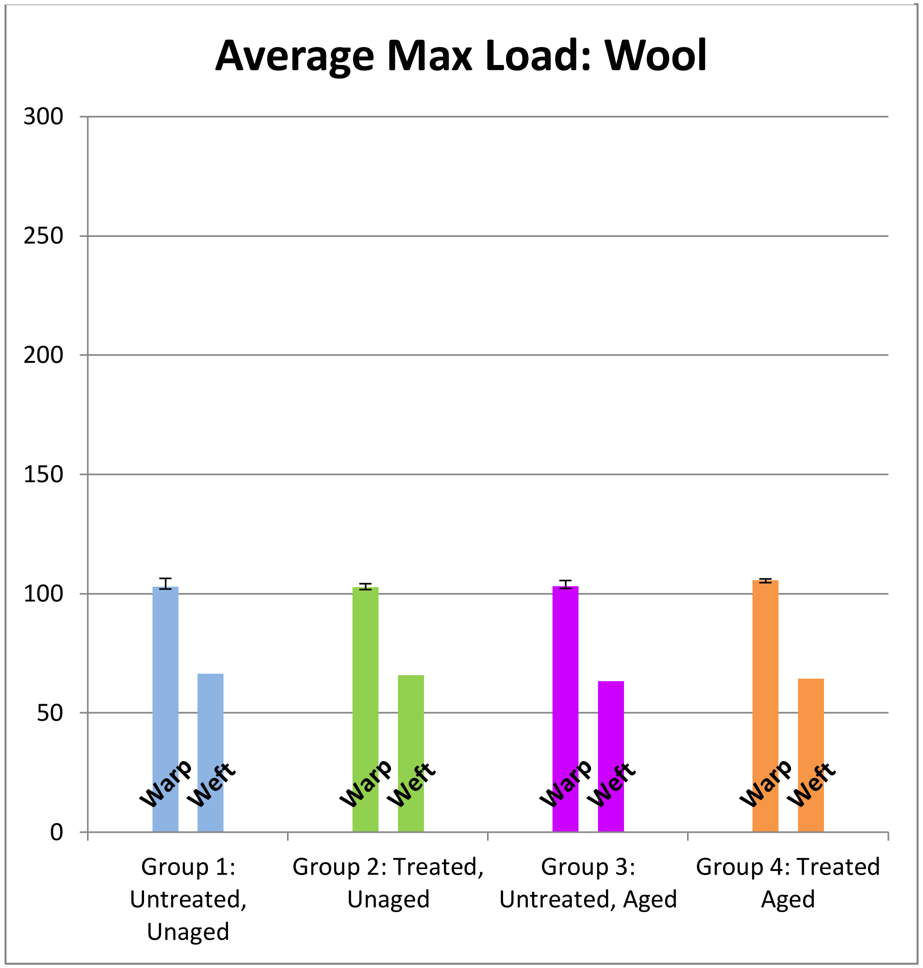
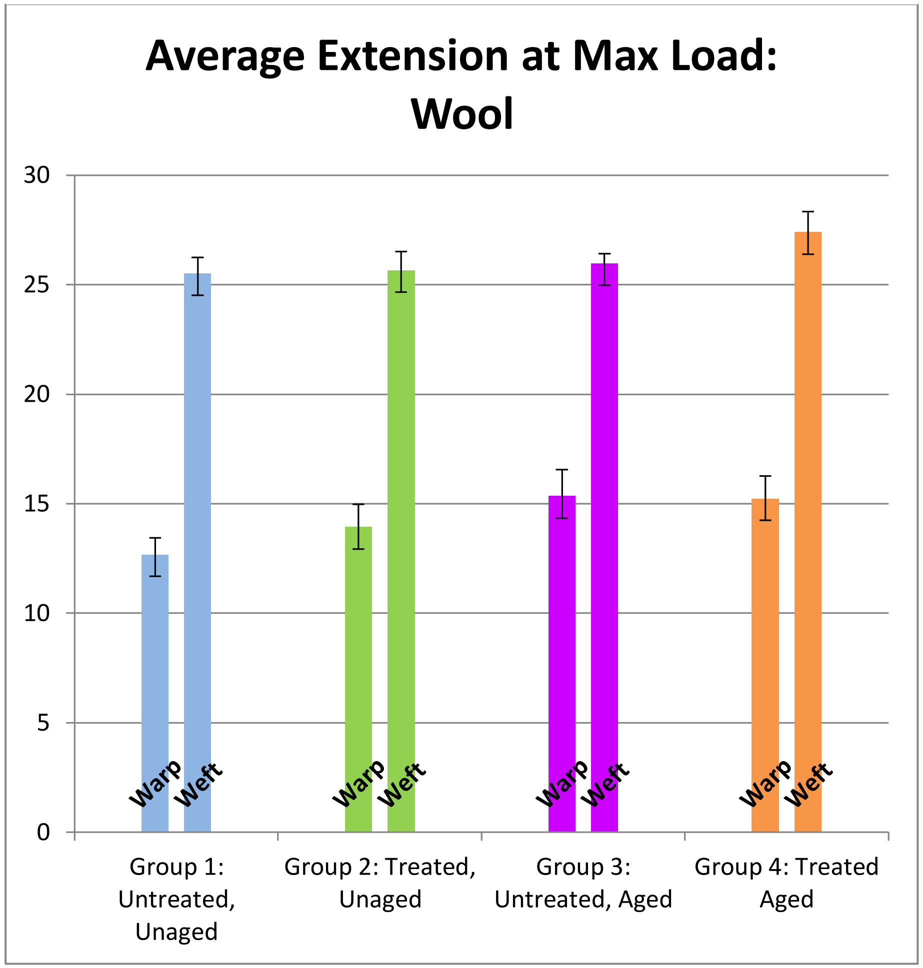


FIGURE 3.10: AVERAGE EXTENSION AT MAXIMUM LOAD TAKEN FROM THREE COTTON SPECIMENS: NOTING STANDARD DEVIATION



3.2.3.4. EVALUATION

Major differences in the column height occur across materials, with cotton showing greater strength, and wool showing greater elongation. This is characteristic of the two materials. Warp and weft also differ significantly within variable groups, indicating that, as expected, the weave orientation from which each sample originated is a major distinguishing factor in tensile strength and elongation for the samples.

Comparing warp to warp and weft to weft is necessary to make comparison of the treatment and ageing variables. If great differences were to occur between column heights, then would suggest that treatment with D5 has an effect on the strength or elasticity of fibers. Conversely, overall similarity of the height of the columns would indicate that treatment with D5 had no measureable effect on the tensile properties of the cotton lawn and wool delaine tested.

The graphs present generally consistent results between treated and untreated samples, with some exceptions. The samples for cotton in Group 4 (Treated, Aged)/Weft show a much higher average value for maximum load than the other three variable groups. These results would suggest that somehow treatment with D5 had made the fibers able to carry a greater load before rupture. According to the accompanying graph for extension at maximum load for the same variable, the proposed improved strength does not appear to come at the expense of elongation: this value is comparable to the rest of the field. It is unlikely, but possible, that these results are derived from the properties of the sample.

Taking into account the standard deviation across the trials for cotton shows that there is great disparity in the range of values for both maximum load and extension at maximum load, resulting in a high standard deviation. The results for wool show a much more consistent set of values for the samples. Based on this information, it would suggest that there is more margin of error in the cotton tensile tests, leading to the greater average value in column height for this Group. Additionally, the cotton Group 4/Warp column is consistent with the other average values. If the Group 4/Warp samples showed the same disparity then it would be more likely a matter of inherent properties.

The tests for wool show greater consistency in the results, however there is a slight increase again in the height of the Group 4/Weft samples, but this increase is very minimal.

For all of the tensile tests, the limited number of trials results in statistically weaker results than if there had been a greater number of samples tested. It may be valuable to revisit tensile testing in future studies.

3.2.4. TEXTILE SUBSTRATE TESTING: SCANNING ELECTRON MICROSCOPY

SUMMARY

SEM analysis uses focused electron beams to scan samples and gather information about elemental composition. It is particularly useful for analysis of metallic elements. If D5 had deposited a residue on the samples, then the EDS capability of the SEM might be able to detect the residual silicon atoms present on the material. Silicon is a semi-metallic atom which is not present in cotton and wool fibers, so if it were detected in treated samples then there would be a good indication for concern about contamination.

High resolution images taken in SEI mode would provide a record of the appearance of the sample surface at high magnification. Viewing these images would enable detection of any damage to the fibers on a microscopic level.

3.2.4.1. SAMPLE POPULATION

The population of this analysis was very limited due to restrictions of time and resources: only unaged samples were able to be analyzed in the time allotted. The total sample population was comprised of one treated and one untreated sample for both cotton and wool. For each of the four samples, a single area was viewed in SEI (Secondary Electron Imaging) mode. This area was also scanned using EDS (Energy Dispersive X-ray Spectrometry).

3.2.4.2. DATA

FIGURE 3.12: SEI IMAGE OF COTTON FIBERS, UNTREATED

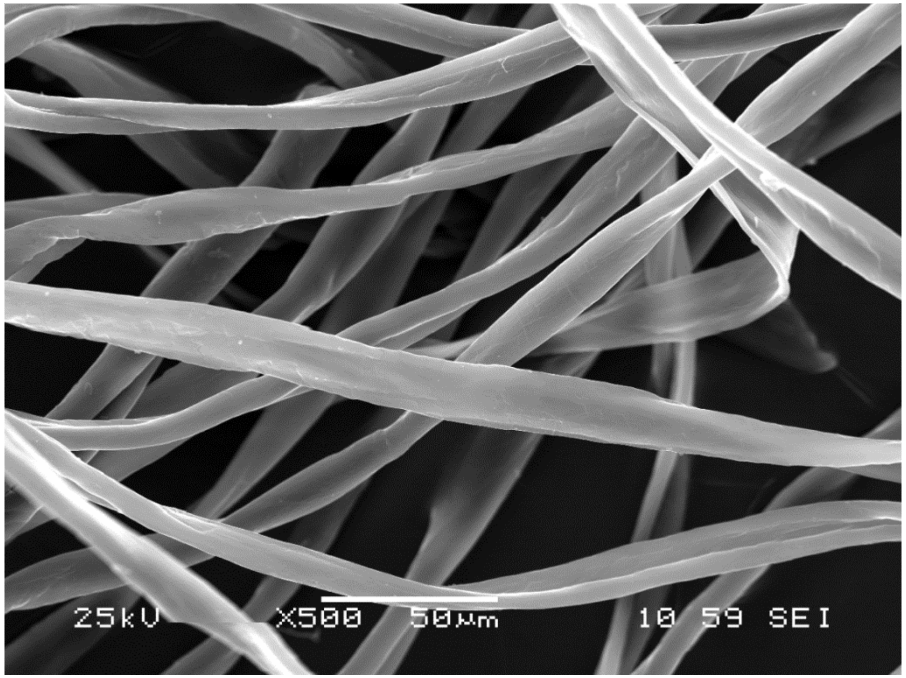


FIGURE 3.13: SEI IMAGE OF COTTON FIBERS, TREATED WITH D5

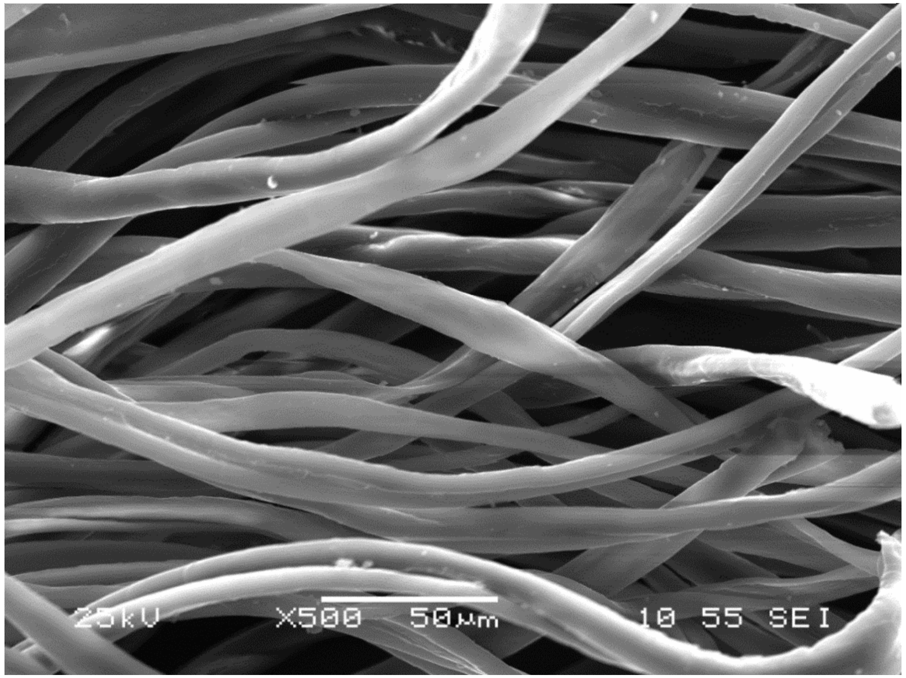


FIGURE 3.15: SEI IMAGE OF WOOL FIBERS, UNTREATED

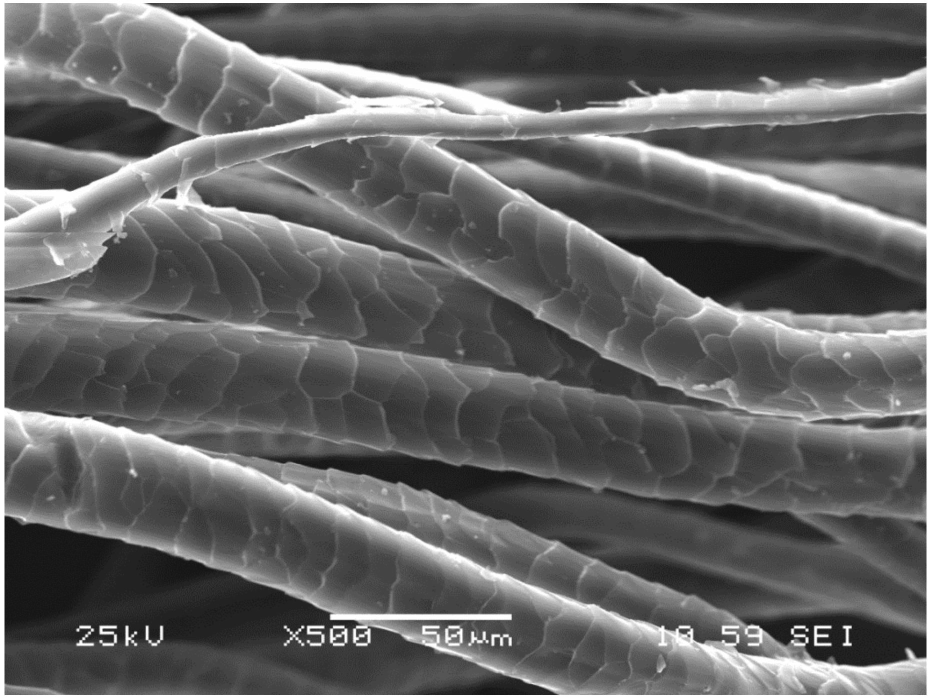
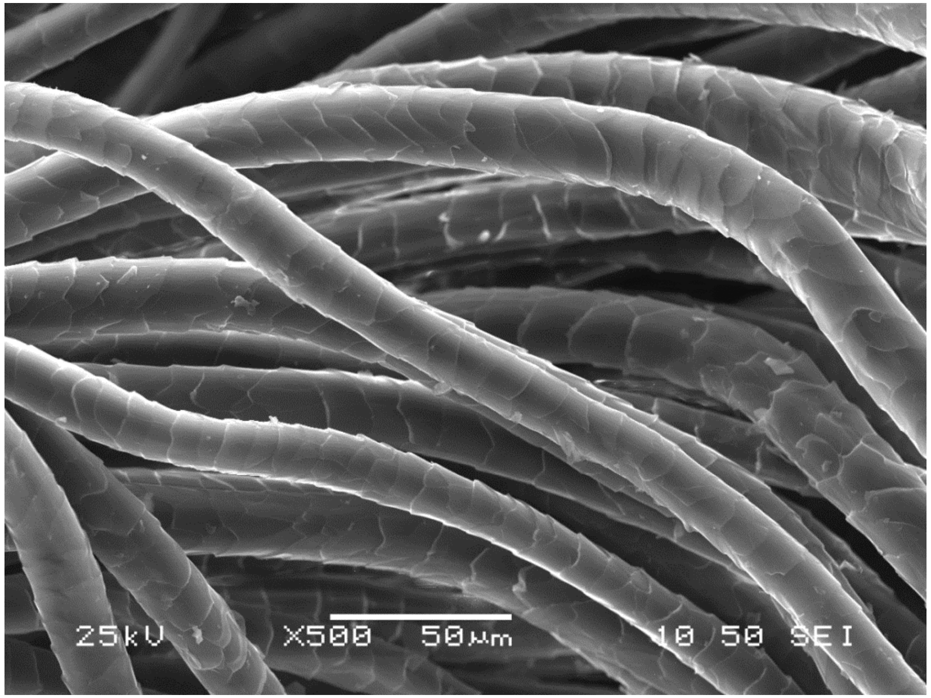


FIGURE 3.14: SEI IMAGE OF WOOL FIBERS, TREATED WITH D5

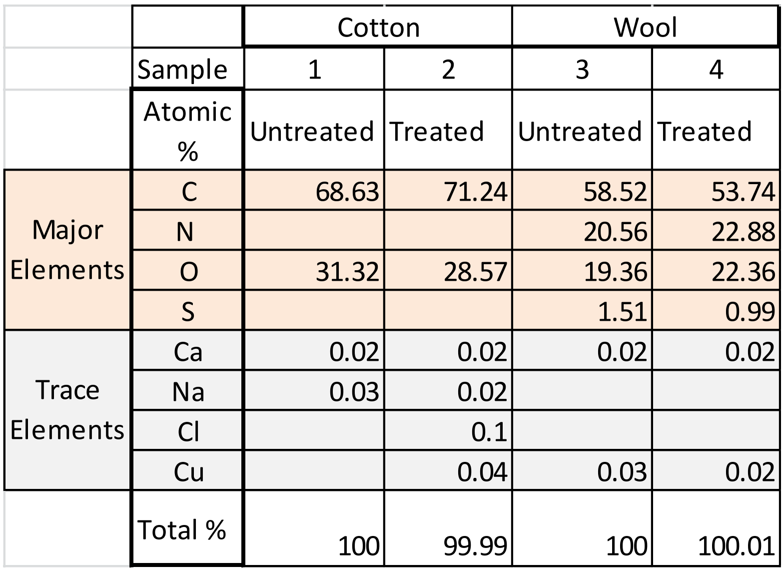


3.2.4.3. EVALUATION

There is no appreciable difference in the appearance of fibers at 500x magnification. The appearance of white flecks on the samples has been interpreted to be dust that was picked up during handling. The sample population at this stage of the analysis had already been through FITR analysis and been retained from the remnants of the samples used for tensile testing. There is a good chance that some dust was picked during the successive rounds of testing.

Data for the elemental analysis is shown according to % elemental composition in Table 3.2.3. See Appendix XIII for the individual spectra and other elemental measurement. Trace mineral content was detected in all of the samples, also thought to be a result of dust particles.

TABLE 3.5: RESULTS OF ELEMENTAL ANALYSIS BY ATOMIC %



		Cotton		Wool	
	Sample	1	2	3	4
	Atomic %	Untreated	Treated	Untreated	Treated
Major Elements	C	68.63	71.24	58.52	53.74
	N			20.56	22.88
	O	31.32	28.57	19.36	22.36
	S			1.51	0.99
Trace Elements	Ca	0.02	0.02	0.02	0.02
	Na	0.03	0.02		
	Cl		0.1		
	Cu		0.04	0.03	0.02
	Total %	100	99.99	100	100.01

No evidence of silicon was found in the samples tested. This does seem to suggest that there is no D5 residue left behind, however it does not rule out any traces of D5which might be too minute to be detected by the EDS used in this trial.

3.3. SOIL REMOVAL TESTING

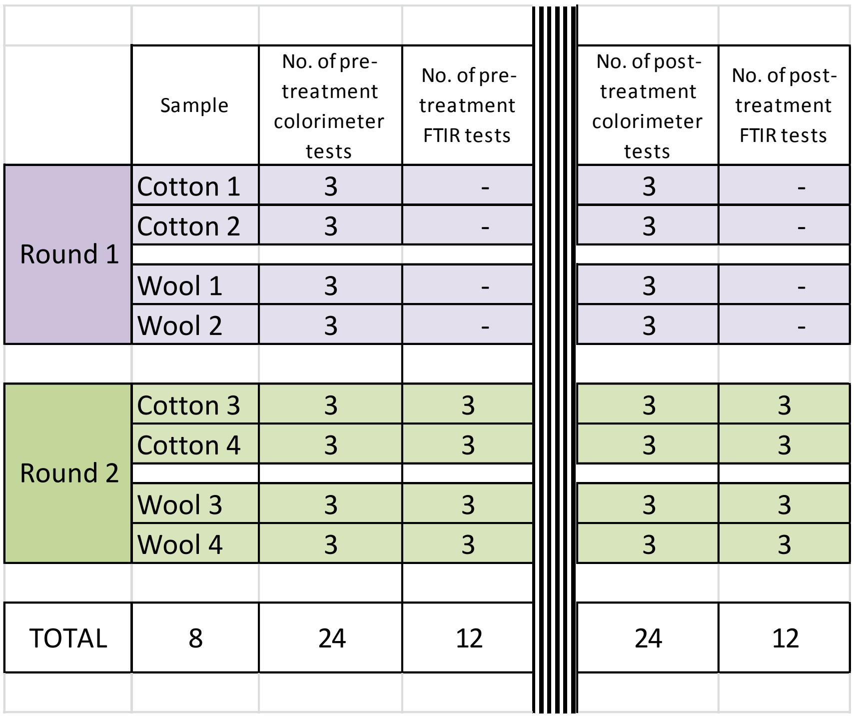
D5 was used to treat samples of new cotton and wool fabric which had been acquired pre-soiled with standardized applications of olive oil and carbon black. A combination of colorimetry and ATR-FTIR analysis was performed before and after treatment to assess the degree of soil removal achieved by the treatment.

3.3.1. SOIL REMOVAL TESTING: COLORIMETRY

3.3.1.1. SAMPLE POPULATION

Table 3.2.4 summarizes the sample population for the colorimetry and ATR-FTIR tests. Two separate trials were performed in this stage, with the first trial consisting of Samples 1 and 2 of cotton and Samples 1 and 2 of wool, and the second trial for Samples 3 and 4.

TABLE 3.6: SAMPLE POPULATION FOR SOIL REMOVAL TESTING



	Sample	No. of pre-treatment colorimeter tests	No. of pre-treatment FTIR tests	No. of post-treatment colorimeter tests	No. of post-treatment FTIR tests
Round 1	Cotton 1	3	-	3	-
	Cotton 2	3	-	3	-
	Wool 1	3	-	3	-
	Wool 2	3	-	3	-
Round 2	Cotton 3	3	3	3	3
	Cotton 4	3	3	3	3
	Wool 3	3	3	3	3
	Wool 4	3	3	3	3
TOTAL	8	24	12	24	12

The overall procedure of the two treatments was identical. It can be recalled, however that chromameter readings were able to detect that one side of the fabrics from which the samples were taken was consistently darker than the other. This was not controlled for in treatment of Samples 1 and 2.

Visual assessment established that treatment had had a significant lightening effect on the samples. Color assessment in this stage of testing was not confined to visual assessment alone, however. A Konica Minolta C-series chromameter was used to quantify color change measured before and after the immersion treatment. It was also used to measure the color difference in the soiled fabric and its unsoiled counterpart.

The chromameter is programmed to analyze color according the CIELAB Standard Observer Response. In CIELAB, the measurements L*, a*, b* correspond to different color qualities. L* is the measurement for luminosity, quantifying the brightness or darkness of the color. The measurements a* and b* are indicative of hue: a indicates location on a red-green continuum, with negative values closer to green and positive values closer to red; b* locates the value on the blue-yellow continuum, with positive numbers indicating more “yellowness”, and negative numbers indicating more “blueness”.

To quantify the color difference between the soiled and unsoiled fabrics, readings of the unsoiled fabrics were compared with pre-treatment readings of the experimental samples. The table below compares the average before treatment readings of the four cotton and four wool samples to an average of readings taken from a sample of the unsoiled corresponding cotton and wool fabrics.

The greatest difference between the soiled and unsoiled fabrics is shown in their L values, the measurement of luminosity (darkness/lightness).

TABLE 3.7: COMPARISON OF SOILED AND UNSOILED TEST FABRICS

	L	a	b			L	a	b
Pretreatment Soiled Cotton	48.69	0.88	2.35		Pretreatment Soiled wool	41.83	0.73	2.79
Unsoiled Cotton	78.85	-1.28	12.91		Unsoiled wool	83.41	0.60	-0.06

3.3.1.2. DATA

A total of nine readings of each color value L*, a*, b* were taken for each sample (3 testing locations, 3 readings per location). The nine readings for each value were averaged, and these mean values were compared.

TABLE 3.8: AVERAGE CIELAB COLOR VALUES OF SAMPLES IN SOIL REMOVAL TESTING

	Before Treatment			After Treatment						Overall Color Change
	L	a	b	L	a	b	ΔL	Δa	Δb	
Cotton 1	48.98	1.02	2.27	55.13	0.66	1.95	6.16	-0.35	-0.33	6.18
Cotton 2	48.43	1.03	2.32	55.13	0.68	1.94	6.70	-0.36	-0.37	6.72
Cotton 3	48.87	0.75	2.45	54.82	0.77	2.00	5.95	0.02	-0.45	5.96
Cotton 4	48.48	0.71	2.37	54.77	0.74	1.88	6.29	0.03	-0.49	6.31
AVERAGE	48.69	0.88	2.35	54.96	0.71	1.94	6.27	-0.17	-0.41	6.29
Standard Dev.	0.24	0.15	0.07	0.17	0.04	0.04	0.27	0.19	0.06	0.27
Wool 1	42.38	0.81	2.72	49.33	0.41	2.99	6.95	-0.40	0.27	6.97
Wool 2	41.92	1.01	2.60	49.04	0.42	2.96	7.12	-0.59	0.35	7.15
Wool 3	41.53	0.45	2.92	48.46	0.68	3.00	6.94	0.23	0.07	6.94
Wool 4	41.49	0.66	2.91	48.37	0.70	2.95	6.88	0.03	0.04	6.88
AVERAGE	41.83	0.73	2.79	48.80	0.55	2.97	6.97	-0.18	0.18	6.99
Standard Dev.	0.36	0.21	0.13	0.40	0.14	0.02	0.09	0.33	0.13	0.10

3.3.1.3. EVALUATION

When comparing color values to each other, it is possible to compare the individual color properties L*, a*, b* or to look at an overall color change value compiled from a calculation of the three values.

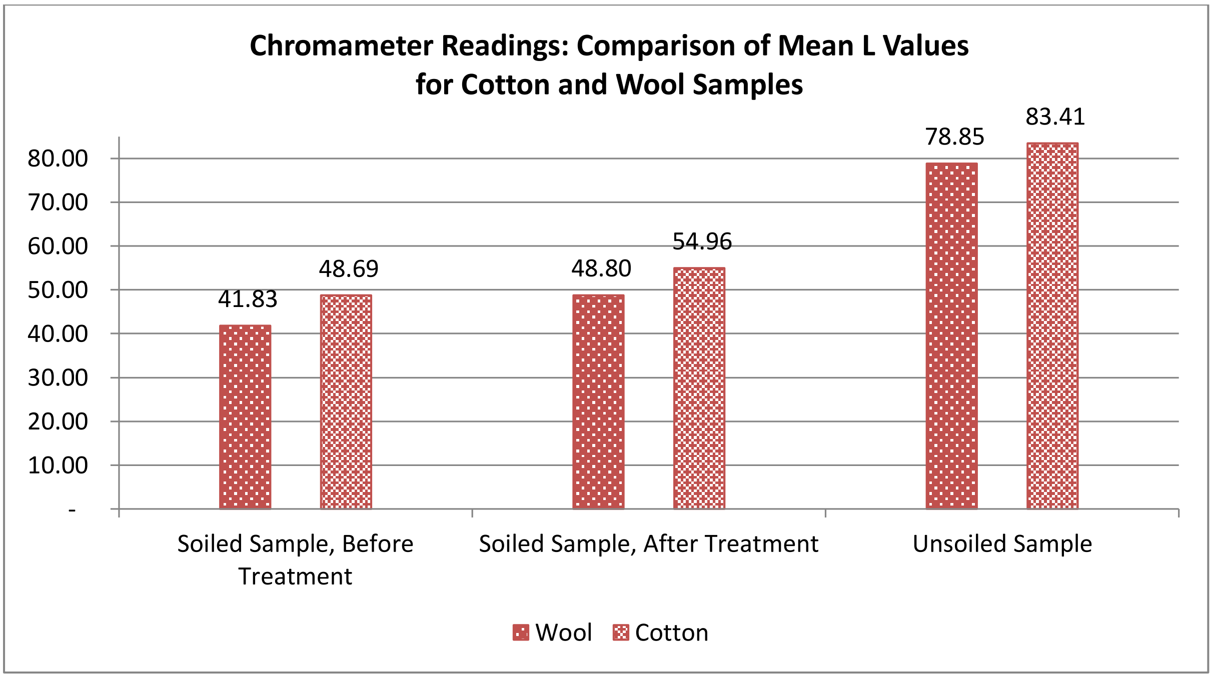
Overall color change, ΔE, is obtained combining the values for of L* (luminosity), a* (hue: re-green) and b* (hue: blue-yellow) into a single value using the following calculation:

$$\Delta E = \sqrt{L^{*2} + a^{*2} + b^{*2}}$$

Looking at Table 3.2.6 it becomes apparent that across the samples, absolute values for ΔL* are significantly higher than absolute values for Δ a* or Δ b*. This makes sense when considering that soot and olive oil soiled samples are a dark gray, inherently lacking in strong hue. Reflected numerically, the values show how the dark gray soil lacks a very strong tendency toward hue in either direction on the continuum.

It is evident that the factor ΔL* has the most impact on the color change for both cotton and wool. Changes in hue, values carry considerably less weight in the resulting calculation.

FIGURE 3.16: MEAN L* VALUES FOR COTTON AND WOOL BEFORE AND AFTER TREATMENT, COMPARED TO UNSOILED L* VALUES



The ΔL values depict a shift toward higher numeric values, which is equivalent to a shift toward lighter color value. Using values for the unsoiled samples of cotton and wool as the denominator in percent calculation, before and after treatment values can be compared in terms of percentage change.

TABLE 3.9: PERCENTAGE CHANGE OF SOILED SAMPLES BEFORE AND AFTER TREATMENT

	Unsoiled Sample	Pre-Treatment Soiled Sample	Post-Treatment Soiled Sample	Change in L value
Cotton	83.41	48.69	54.96	6.27
Percent	100%	58%	66%	8%
Wool	78.85	41.83	48.8	6.97
Percent	100%	53%	62%	9%

After treatment, cotton pre-soiled samples were 8% closer to the shade of unsoiled samples. Wool samples after treatment were 9% closer.

It is evident that the treatment has reduced soiling in the samples, even though no dark material was able to be observed lifting off into solution during the sample treatment. In order to understand what could be causing this, ATR-FTIR was employed in the second round of testing to analyze the molecular composition of the samples.

3.3.2. SOIL REMOVAL TESTING: ATR-FTIR

3.3.2.1. SUMMARY

The first soil removal test revealed significant color change after immersion treatment with D5. The composition of the soiling which was carried away by D5 was thought to be olive oil, or a colorless compound in olive oil which was highly nonpolar.

A second soil removal test was performed, and analysis with the addition of ATR-FTIR analysis before and after treatment.

3.3.2.2. SAMPLE POPULATION

The sample population for ATR-FTIR spectroscopy was comprised of two samples for cotton and two samples for wool. As in the FTIR analysis in the Stage 1, the resulting spectra from the three readings for each sample were saved as an ASCII file and exported into Excel®. An average spectra was compiled from the three readings taken from each sample.

3.3.2.3. DATA

FIGURE 3.17: COMPARISON OF AVERAGE SPECTRA FOR SOILED COTTON SAMPLES BEFORE AND AFTER TREATMENT WITH D5

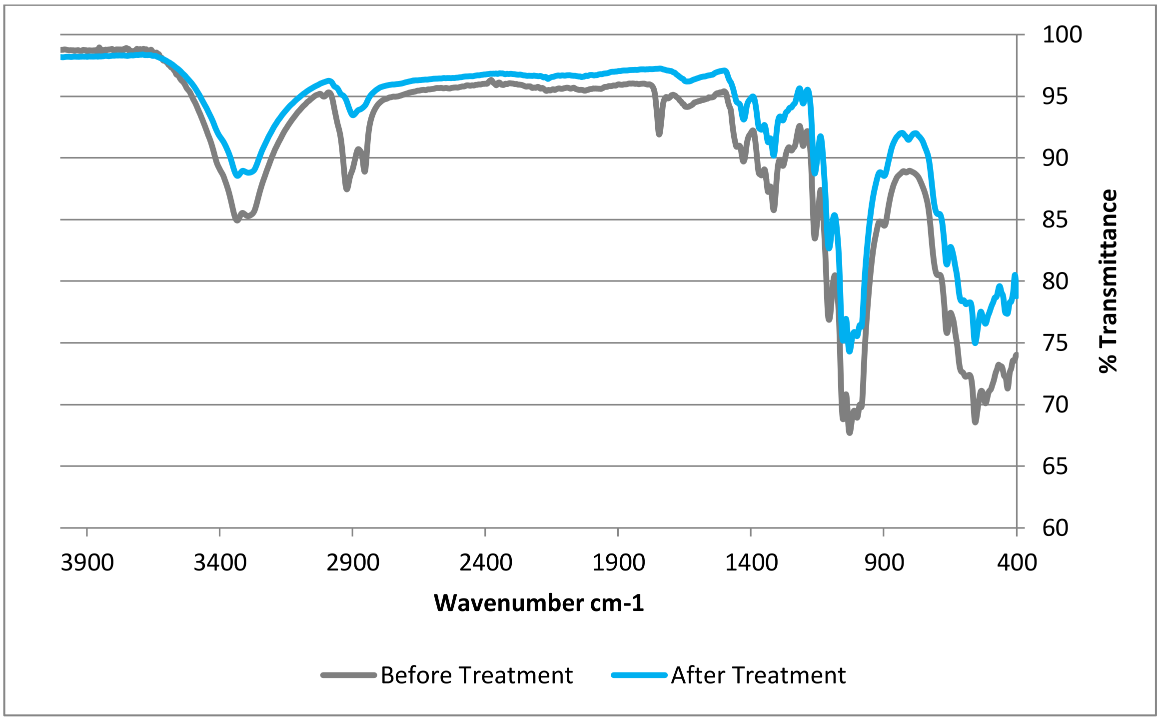
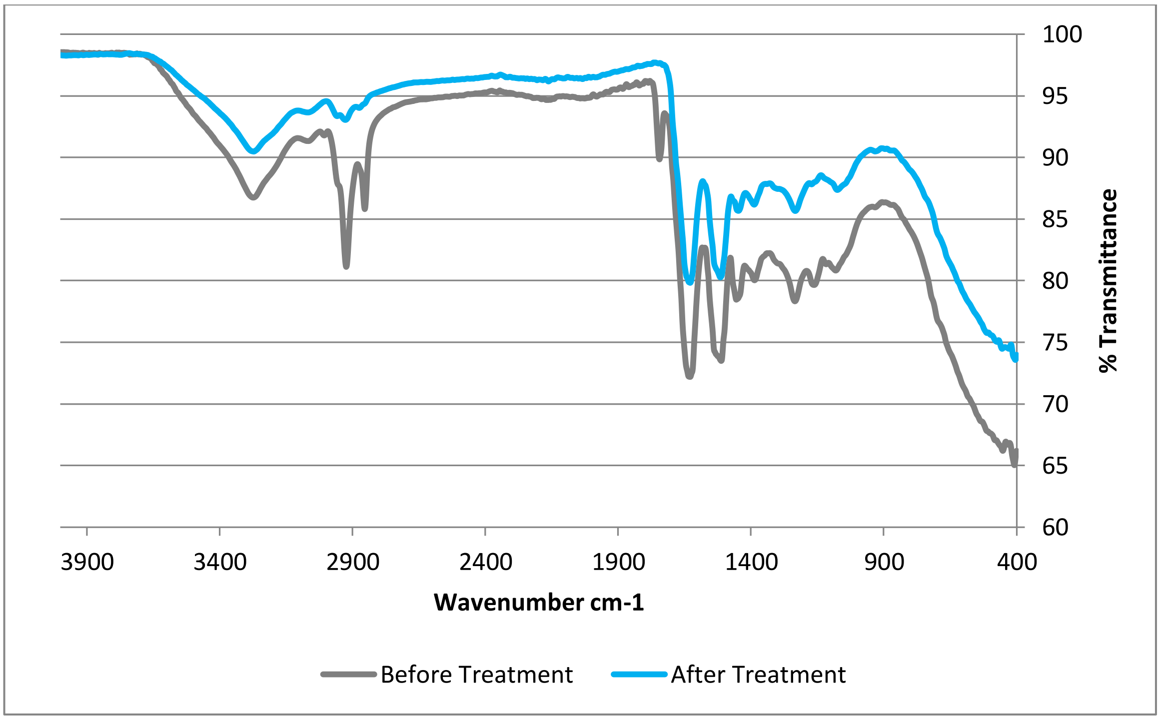


FIGURE 3.18: COMPARISON OF AVERAGE SPECTRA FOR SOILED WOOL SAMPLES BEFORE AND AFTER TREATMENT WITH D5

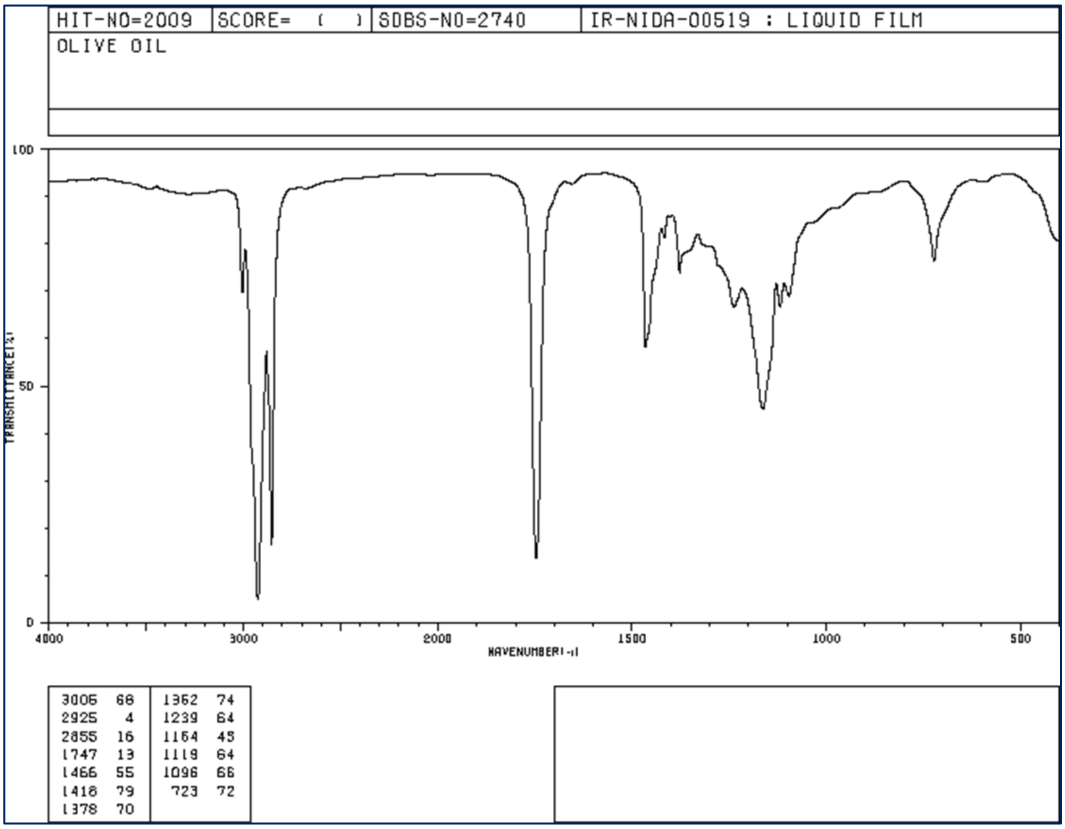


3.3.2.4. EVALUATION

There are pronounced spectral differences between the readings taken before and after soil removal, which echoes the colorimetry results: D5 solubilized some material present on the presoiled samples and carried it away into solution, however the identity of the material was not known.

Comparison made with reference spectra for olive oil indicates some distinct similarities between the reference and peaks present in the samples before soil removal.

FIGURE 3.19: REFERENCE SPECTRA FOR OLIVE OIL FROM SPECTRAL DATABASE FOR ORGANIC COMPOUNDS, *NO.:2740, COMPOUND NAME: OLIVE OIL,IR : LIQUID FILM*, HTTP://RIODB01.IBASE.AIST.GO.JP/SBDS/ (NATIONAL INSTITUTE OF ADVANCED INDUSTRIAL SCIENCE AND TECHNOLOGY) (ACCESSED 1 AUGUST 2012)



HIT-NO=2009 | SCORE= ( ) | SDBS-NO=2740 | IR-NIDA-00519 : LIQUID FILM

OLIVE OIL

3006	68	1362	74
2925	4	1239	64
2855	16	1164	45
1747	13	1119	64
1466	55	1096	66
1418	79	723	72
1378	70		

Peaks of particular intensity in the reference sample, around 2900 cm-1 and between 1800-1700 cm-1, are also seen in the before treatment spectra, though at significantly less intensity. Their presence, however, does indicate that the compounds removed by D5 may indeed be from the olive oil component of the soiling.

In Chapter 1,, the Hildebrand solubility parameter for D5 was cited from the *Journal of Cosmetic Science* as 5.77. This publication also includesa Hildebrand value of 7.87 for olive oil.[61] Considering

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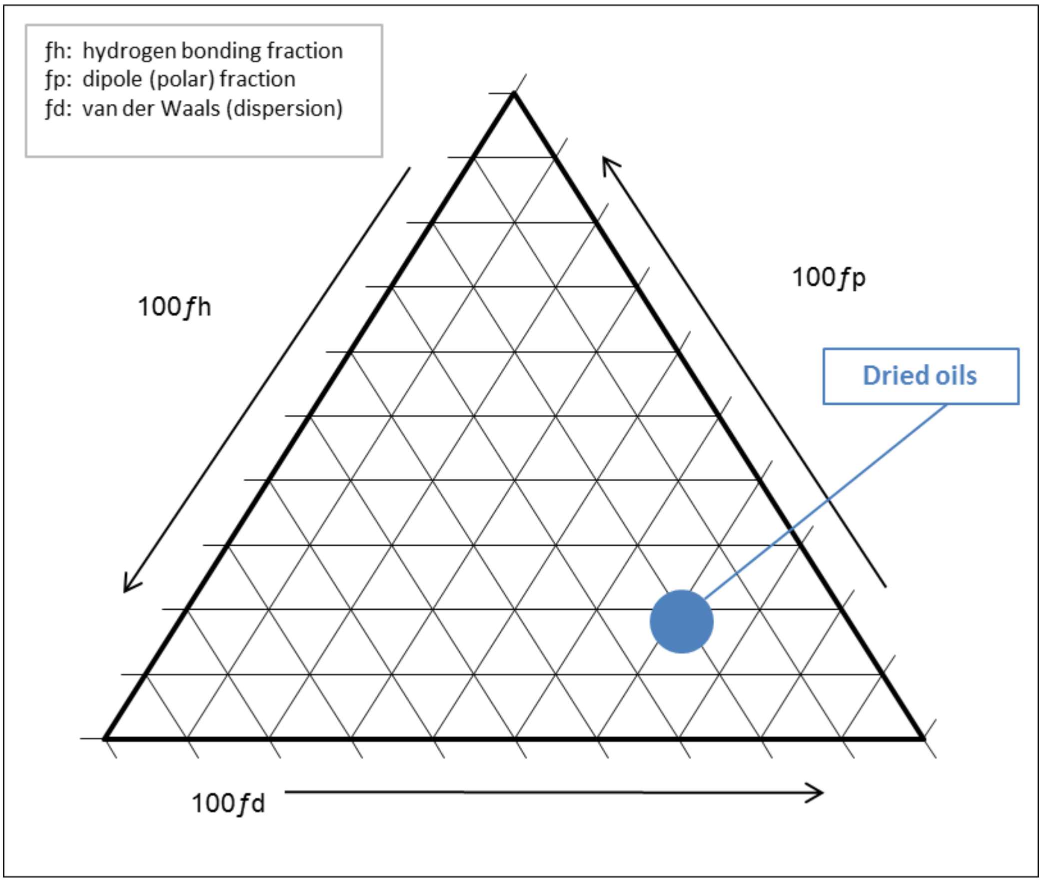
[61] C.D. Vaughn, 'Using Solubility Parameters in Cosmetics Formulation', *Journal of Cosmetic Science* 36 (September/October 1985): 329.

that values for other materials range from 2.59 for “Propellant 13” to 23.40 for water[62], the margin of difference between the two materials is relatively small in terms of the overall potential for disparity.

A Tea’s Graph may also be of use when considering the ability of D5 to solubilize olive oil according to Hansen solubility parameters.

FIGURE 3.20: TEA'S GRAPH SHOWING THE SOLUBILITY OF DRIED OILS, ADAPTED FROM TÍMÁR-BALÁSZY AND EASTOP, *CHEMICAL PRINCIPLES OF TEXTILE CONSERVATION*, 168.



As shown in Figure 3.20: Tea's graph showing the solubility of dried oils, adapted from TímÁr-balÁszy and eastop, *chemical Principles of Textile conservation*, 168. the solubility of dried oils is located in the bottom right hand corner of the Tea’s Graph, which is the same location that was predicted for D5 in Chapter 1.. As nonpolar compounds, oils have greater ability to form intermolecular bonds via dispersion forces and hydrogen bonding than through dipole-dipole interactions. The same is thought to be true of D5.

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[62] C.D. Vaughn, ‘Using Solubility Parameters in Cosmetics Formulation’, *Journal of Cosmetic Science* 36 (September/October 1985): 329-331

4. CHAPTER FOUR: CONCLUSIONS

To conclude this study on D5's potential in textile conservation cleaning applications, the research questions which guided the research are addressed:

4.1.1. D5 AS A GREEN SOLVENT

The use of D5 may present some immediate health advantages over organic solvents which are known to have more toxic effects, Long term effects of exposure on humans is not known, but in some assessments, evidence shows D5 may have the potential to cause serious harm with overexposure. Some major environmental agencies have not yet performed formal assessments of D5, classing it as a low priority, since in its major applications users are exposed to such limited quantities. .

Current and future health assessments made regarding D5 may be based on its large-scale uses and will not necessarily reflect the type of applications conservators employ. In the dry cleaning trade, direct contact with D5 is minimized by use of a closed system and solvent recapturing; and in cosmetics, concentrations used are very low. More information is needed to determine safe thresholds of use for those who come into direct contact with D5, In the meantime, it is recommended to follow the manufacturer's safety recommendations, by wearing gloves when handling and employing fume extraction, or at the very least, ventilation, as indicated by the MSDS for the product being used. Pending further study, D5 appears to present some environmental benefits over traditional organic solvents, however it is not without impact.

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4.1.2. EFFECT ON TEXTILES

As a nonreactive, volatile compound, it was expected that D5 would evaporate away completely from the surface of textiles. With minimal exception, the experimental data from the textile substrate stage of testing presents consistent evidence that little to no difference in the physical and chemical condition of the samples can be detected before and after treatment with D5. This is true of both aged and unaged samples.

The results of this study are limited by several factors, including the methods of artificial ageing and the small number of trials for some experiments, especially for tensile strength and SEM analysis. Additionally, only one procedure for application of D5 to textiles was examined as an independent variable. Immersion treatment for longer than one hour was not examined, nor was the use of spot cleaning. The samples which were tested were limited to new, natural fibers. There is a clear need for further study of the effects of D5 on textile substrates which can take into account the complexities of treatment of historic textiles.

4.1.3. EFFECT ON SOILING

A look at the properties of D5 and the theory underpinning its structure has suggested how it might behave in the presence of certain soiling: a hypothesis was formed that D5 would be effective on nonpolar soiling through interactions of the van der Waals and hydrogen bonds between molecules. Results from the soil removal stage of testing seem to agree with this hypothesis, as the results have been interpreted as showing the removal of dried olive oil from the surface of the sample. Dried oils have a theoretical solubility profile similar to that of D5.

The evidence shows that D5 can be effective at removing nonpolar componds from new fabrics with recently acquired soiling. The study does not give any information about how effective D5 might be in removing the aged soiling which affects historic textiles.. More study is required in order to assess how D5 will affect a range of substrates and soils, aged and unaged.

Though D5 did have a significant effect in the tests, a good part soiling was not removed from the samples. This underscores the fact that if D5 were to be adopted for textile conservation, it would serve to become but one of many tools in the conservator's kit, and not a cure-all for every type of soil or stain.

4.1.3.1. HANDLING AND PRACTICALITY

D5 is sourced from a number of different manufacturers supplying both the cosmetics and dry cleaning industries, and is widely distributed. The practical use of D5 in the lab was similar in setup, time, and execution as what might be expected with any commonly-used solvent. Samples wet out quickly. D5 was clear and odorless and did not cause any irritation as observed by the researcher. With a drying time on the longer end, this might complicate treatment of certain objects, including large and many-layered textiles.

4.1.4. THE POTENTIAL OF D5 IN TEXTILE CONSERVATION

D5 shows promise for use in textile conservation. Time will tell how green D5 proves to be, but as conservators endeavor to "go green" in their practice, they may discover that there are no black and white solutions. As Brophy and Wylie put it, "shades of green" exist when it comes to best practices.[63] The conservator cannot be expected to work without having any impact on the environment and must weigh the importance of maintaining cultural heritage materials against the risks indicated by their methods.

Further research will be necessary to determine how treatment with D5 may affect aged and fragile soiled textiles. More research into its solubility performance is also required, including its

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[63] Brophy and Wylie, *The Green Museum,* 4.

performance in spot cleaning. It may also be useful to examine ‘charged’ detergent solutions incorporating D5 to see how polar soiling might be addressed.

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APPENDIX I: LIST OF SOLVENTS IN TEXTILE CONSERVATION

		Fractional Solubility Parameters		
Solvent Group	Name	ƒd	ƒp	ƒh
Aliphatic Hydrocarbons	White spirit	90	4	6
	n-heptane	100	0	0
Aromatic Hydrocarbons	Benzene	78	8	14
	Toluene	80	7	13
	Xylene	83	5	12
Cyclic Hydrocarbons	Turpentine	77	18	5
	Cyclohexane	94	2	4
Alcohols	Methanol	30	22	48
	Ethanol	36	18	46
	Propanol	40	16	44
	Isopropanol	41	18	41
	Buthanol	43	15	42
	n-pentanol (amyl alcohol)	46	13	41
	Cyclohexanol	25	23	52
	Glycerine	30	18	52
	Ethlyene Glycol	45	24	31
	Diacetone alcohol	18	28	54
Ketones	Acetone	47	32	21
	Methyl ethyl ketone	53	26	21
	Methyl isobutyl ketone	58	22	20
	Methyl isoamyl ketone	62	20	18
	Cyclohexanone	55	28	17
Esters	Ethyl acetate	51	18	31
	Propyl acetate	57	15	28
	Butyl acetate	60	13	27
	Amyl acetate	60	12	28
Ethers	Methyl cellosolve	39	22	39
	Ethyl cellosolve	42	20	38
	Butyl cellosolve	46	18	36
	Diethylene glycol ethyl ether (Carbitol)	48	23	29
	Tetrahydrofuran	55	19	26
	Dioxane	67	7	26
Chlorinated Solvents	Methylene chloride (dichloromethane	62	26	12
	Ethylene chloride 1,2-dichloroethane	67	19	14
	Trichloroethylene (Trielen)	70	19	11
	Trichloromethane (chloroform)	67	12	21
	Carbon tetrachloride	85	2	13
Nitrogen Compounds	Dimethyl formamide	41	32	27
Organic Bases	Ethanol amine	32	29	40
	Pyridine	56	26	18
	Morpholine	53	21	26
Sulfur Compounds	Dimethyl sulphoxide	41	36	23
	Carbon disulphide	88	8	4

Adapted from *Chemical Principles of Textile Conservation*, Tímár-Balázsy and Eastop, 1998

APPENDIX II: ETHICAL GUIDELINES

AIC CODE OF ETHICS

PREAMBLE

The primary goal of conservation professionals, individuals with extensive training and special expertise, is the preservation of cultural property. Cultural property consists of individual objects, structures, or aggregate collections. It is material which has significance that may be artistic, historical, scientific, religious, or social, and it is an invaluable and irreplaceable legacy that must be preserved for future generations.

In striving to achieve this goal, conservation professionals assume certain obligations to the cultural property, to its owners and custodians, to the conservation profession, and to society as a whole. This document, the Code of Ethics and Guidelines for Practice of the American Institute for Conservation of Historic & Artistic Works (AIC), sets forth the principles that guide conservation professionals and others who are involved in the care of cultural property.

CODE OF ETHICS

I. The conservation professional shall strive to attain the highest possible standards in all aspects of conservation, including, but not limited to, preventive conservation, examination, documentation, treatment, research, and education.

II. All actions of the conservation professional must be governed by an informed respect for the cultural property, its unique character and significance, and the people or person who created it.

III. While recognizing the right of society to make appropriate and respectful use of cultural property, the conservation professional shall serve as an advocate for the preservation of cultural property.

IV. The conservation professional shall practice within the limits of personal competence and education as well as within the limits of the available facilities.

V. While circumstances may limit the resources allocated to a particular situation, the quality of work that the conservation professional performs shall not be compromised.

VI. The conservation professional must strive to select methods and materials that, to the best of current knowledge, do not adversely affect cultural property or its future examination, scientific investigation, treatment, or function.

VII.The conservation professional shall document examination, scientific investigation, and treatment by creating permanent records and reports.

VIII.The conservation professional shall recognize a responsibility for preventive conservation by endeavoring to limit damage or deterioration to cultural property, providing guidelines for continuing use and care, recommending appropriate environmental conditions for storage and exhibition, and encouraging proper procedures for handling, packing, and transport.

IX.The conservation professional shall act with honesty and respect in all professional relationships, seek to ensure the rights and opportunities of all individuals in the profession, and recognize the specialized knowledge of others.

X. The conservation professional shall contribute to the evolution and growth of the profession, a field of study that encompasses the liberal arts and the natural sciences. This contribution may be made by such means as continuing development of personal skills and knowledge, sharing of information and experience with colleagues, adding to the profession's written body of knowledge, and providing and promoting educational opportunities in the field.

XI. The conservation professional shall promote an awareness and understanding of conservation through open communication with allied professionals and the public.

XII. The conservation professional shall practice in a manner that minimizes personal risks and hazards to co-workers, the public, and the environment.

XIII. Each conservation professional has an obligation to promote understanding of and adherence to this Code of Ethics.

The conservation professional should use the following guidelines and supplemental commentaries together with the AIC Code of Ethics in the pursuit of ethical practice. The commentaries are separate documents, created by the AIC membership, that are intended to amplify this document and to accommodate growth and change in the field.

ICON PROFESSIONAL GUIDELINES

PROFESSIONAL GUIDELINES

Promoted by the European Confederation of Conservator-Restorers' Organisations and adopted by its General Assembly , Brussels 1 March 2002
Please note that since Icon ceased to be a member of ECCO in September 2007, Icon and its members no longer conform to the part of Section III(ii) indicated by asterisks.

Preamble

The objects, buildings and environments to which society attributes particular aesthetic, artistic, documentary, environmental, historic, scientific, social, or spiritual values are commonly designated "Cultural Heritage" and constitute a material and cultural patrimony to be passed on to coming generations.

Since it is entrusted to the care of the Conservator-Restorer by society, s/he has a responsibility not only to the cultural heritage itself, but also to the owner or legal guardian, the originator or creator, the public, and to posterity. The following conditions serve to safeguard all cultural heritage regardless of its owner, age, state of completeness or value.

I. Definition of the Conservator-Restorer

The Conservator-Restorer is a professional who has the training, knowledge, skills, experience and understanding to act with the aim of preserving cultural heritage for the future, and according to the considerations outlined below.

The fundamental role of the Conservator-Restorer is the preservation of cultural heritage for the benefit of present and future generations. The Conservator-Restorer contributes to the perception, appreciation and understanding of cultural heritage in respect of its environmental context and its significance and physical properties.

The Conservator-Restorer undertakes responsibility for, and carries out strategic planning; diagnostic examination; the drawing up of conservation plans and treatment proposals; preventive conservation; conservation-restoration treatments and documentation of observations and any interventions.

Diagnostic examination consists of the identification, the determination of the composition and the assessment of the condition of cultural heritage; the identification, nature and extent of alterations; the evaluation of the causes of deterioration and the determination of the type and extent of treatment needed. It includes the study of relevant existing information.

Preventive Conservation consists of indirect action to retard deterioration and prevent damage by creating conditions optimal for the preservation of cultural heritage as far as is compatible with its social use. Preventive conservation also encompasses correct handling, transport, use, storage and display. It may also involve issues of the production of facsimiles for the purpose of preserving the

original.
Conservation consists mainly of direct action carried out on cultural heritage with the aim of stabilising condition and retarding further deterioration.
Restoration consists of direct action carried out on damaged or deteriorated cultural heritage with the aim of facilitating its perception, appreciation and understanding, while respecting as far as possible its aesthetic, historic and physical properties.
Documentation consists of the accurate pictorial and written record of all procedures carried out, and the rationale behind them. A copy of the report must be submitted to the owner or custodian of the cultural heritage and must remain accessible. Any further requirements for the storage, maintenance, display or access to the cultural property should be specified in this document.
The record remains the intellectual property of the Conservator-Restorer and shall be retained for future reference.

Furthermore, it is within the Conservator-Restorer’s competence to:

- develop programmes, projects and surveys in the field of conservation-restoration
- provide advice and technical assistance for the preservation of cultural heritage
- prepare technical reports on cultural heritage (excluding any judgement of its market value)
- conduct research
- develop educational programmes and teach
- disseminate information gained from examination, treatment or research
- promote a deeper understanding of the field of conservation-restoration

II. Education and Training

To maintain the standards of the profession, the Conservator-Restorer’s professional education and training shall be at the level of a university Master’s degree (or recognised equivalent) in conservation-restoration. The training is further detailed in “E.C.C.O. Professional Guidelines III”.
Conservation-Restoration is a complex and rapidly developing field. Therefore, the qualified Conservator-Restorer has a professional responsibility to keep up to date with new findings, and ensure that s/he practices her/his profession in line with current ethical thought. Continuing Professional Development is further detailed in “E.C.C.O. Professional Guidelines II”.

III. Distinction from other Related Fields

Conservation-Restoration is distinct from related fields (eg art and crafts) in that its primary aim is the preservation of cultural heritage, as opposed to the creation of new objects or maintaining or repairing objects in a functional sense.
The Conservator-Restorer is distinguished from other professionals by her/his specific education in conservation-restoration.

E.C.C.O. PROFESSIONAL GUIDELINES (II): CODE OF ETHICS

Promoted by the European Confederation of Conservator-Restorers' Organisations and adopted by its General Assembly, Brussels 7 March 2003

I General Principles for the Application of the Code
Article 1: The Code of Ethics embodies the principles, obligations and behaviour which every Conservator-Restorer belonging to a member organisation of E.C.C.O. should strive for in the practice of the profession.
Article 2: The profession of Conservator-Restorer constitutes an activity of public interest and must be practised in observance of all pertinent national and European laws and agreements, particularly those concerning stolen property.
Article 3: The Conservator-Restorer works directly on cultural heritage and is personally responsible to the owner, to the heritage and to society. The Conservator-Restorer is entitled to practise without hindrance to her/his liberty and independence.
The Conservator-Restorer has the right in all circumstances to refuse any request which s/he believes is contrary to the terms or spirit of this Code.
The Conservator-Restorer has a right to expect that all relevant information regarding a conservation-restoration project (of any size) is given to her/him by the owner or custodian.

Article 4: Failure to observe the principles, obligations and prohibitions of the Code constitutes unprofessional practice and will bring the profession into disrepute. It is the responsibility of each national professional body to ensure that its members comply with the spirit and letter of the Code, and to take action in the case of proven non-compliance.

II Obligations towards Cultural Heritage

Article 5: The Conservator-Restorer shall respect the aesthetic, historic and spiritual significance and the physical integrity of the cultural heritage entrusted to her/his care.

Article 6: The Conservator-Restorer, in collaboration with other professional colleagues involved with cultural heritage, shall take into account the requirements of its social use while preserving the cultural heritage.

Article 7: The Conservator-Restorer must work to the highest standards regardless of any opinion of the market value of the cultural heritage. Although circumstances may limit the extent of a Conservator-Restorer's action, respect for the Code should not be compromised.

Article 8: The Conservator-Restorer should take into account all aspects of preventive conservation before carrying out physical work on the cultural heritage and should limit the treatment to only that which is necessary.

Article 9: The Conservator-Restorer shall strive to use only products, materials and procedures which, according to the current level of knowledge, will not harm the cultural heritage, the environment or people. The action itself and the materials used should not interfere, if at all possible, with any future examination, treatment or analysis. They should also be compatible with the materials of the cultural heritage and be as easily and completely reversible as possible.

Article 10: The conservation-restoration treatment of cultural heritage should be documented in written and pictorial records of the diagnostic examination, any conservation / restoration intervention and other relevant information. The report should also include the names of all those who have carried out the work. A copy of the report must be submitted to the owner or custodian of the cultural heritage and must remain accessible. The record remains the intellectual property of the Conservator-Restorer and shall be retained for future reference.

Article 11: The Conservator-Restorer must undertake only such work as s/he is competent to carry out. The Conservator-Restorer must neither begin nor continue a treatment which is not in the best interest of the cultural heritage.

Article 12: The Conservator-Restorer must strive to enrich her/his knowledge and skills with the constant aim of improving the quality of her/his professional work.

Article 13: Where necessary or appropriate, the Conservator-Restorer shall collaborate with other professionals and shall participate with them in a full exchange of information.

Article 14: In any emergency where cultural heritage is in immediate danger, the Conservator-Restorer - regardless of her/his field of specialisation - shall render all assistance possible.

Article 15: The Conservator-Restorer shall not remove material from cultural heritage unless this is indispensable for its preservation or it substantially interferes with the historic and aesthetic value of the cultural heritage. Materials which are removed should be conserved, if possible, and the procedure fully documented.

Article 16: When the social use of cultural heritage is incompatible with its preservation, the Conservator-Restorer shall discuss with the owner or legal custodian, whether making a reproduction of the object would be an appropriate intermediate solution. The Conservator-Restorer shall recommend proper reproduction procedures in order not to damage the original.

III Obligations to the Owner or Legal Custodian

Article 17: The Conservator-Restorer should inform the owner fully of any action required and specify the most appropriate means of continued care.

Article 18: The Conservator-Restorer is bound by professional confidentiality. In order to make a reference to an identifiable part of the cultural heritage s/he should obtain the consent of its owner or legal custodian.

Article 19: The Conservator-Restorer should never support the illicit trade in cultural heritage, and must work actively to oppose it. Where legal ownership is in doubt, the Conservator-Restorer must check all the available sources of information before any work is undertaken.

IV Obligations to Colleagues and the Profession
Article 20: The Conservator-Restorer must maintain a spirit of respect for the integrity and dignity of colleagues, the Conservation-Restoration profession, and related professions and professionals
Article 21: The Conservator-Restorer should, within the limits of her/his knowledge, competence, time and technical means, participate in the training of interns and assistants.
The Conservator-Restorer is responsible for supervising the work entrusted to her/his assistants and interns and has ultimate responsibility for the work undertaken under her/his supervision S/he must maintain a spirit of respect and integrity towards such colleagues.
Article 22: Where work is (in whole or in part) sub-contracted to another Conservator-Restorer, for whatever reason, the owner or custodian must be kept informed. The original Conservator-Restorer is ultimately responsible for the work, unless prior arrangements are made to the contrary.
Article 23: The Conservator-Restorer must contribute to the development of the profession by sharing experience and information.
Article 24 : The Conservator-Restorer shall strive to promote a deeper understanding of the profession and a greater awareness of conservation-restoration among other professions and the public.
Article 25: Records concerning conservation-restoration for which the Conservator-Restorer is responsible are her/his intellectual property (subject to the terms of her/his contract of employment). S/he has the right to be acknowledged as the author of the work.
Article 26: Involvement in the commerce of cultural property is not compatible with the activities of the Conservator-Restorer.
Article 27: When a professional Conservator-Restorer undertakes work that is outside the scope of Conservation-Restoration, s/he must ensure that it does not conflict with this Code.
Article 28: To maintain the dignity and credibility of the profession, the Conservator-Restorer should employ only appropriate and informative forms of publicity in relation to her/his work. Particular care should be exercised in relation to Information Technology (IT) in order to avoid the dissemination of inappropriate, misleading, illegal or unauthorised information.

Acknowledgements
The European Confederation of Conservator-Restorers' Organisations (E.C.C.O.) prepared the E.C.C.O. Professional Guidelines based on the study of documents of national and international organisations for conservation-restoration and heritage. The "Conservator-Restorer: a definition of the profession" (ICOM-CC, Copenhagen 1984) was the first document adopted by E.C.C.O.

http://www.icon.org.uk/index.php?option=com_content&task=view&id=121

APPENDIX III: LIST OF TRADE SYNONYMS FOR D5

LIST OF TRADE SYNONYMS FOR DECAMETHYLCYCLOPENTASILOXANE

http://pubchem.ncbi.nlm.nih.gov/summary/summary.cgi?q=nama&cid=10913

Synonyms: (CID 10913) Total:
41 Display: Filtered, sorted by weight ▾

DECAMETHYLCYCLOPENTASILOXANE
Cyclopentasiloxane, decamethyl-
Dow corning 345
541-02-6
Silicon SF 1202
Dimethylsiloxane pentamer
Dow corning 345 fluid
NUC silicone VS 7158
CCRIS 1328
Cyclic dimethylsiloxane pentamer
HSDB 5683
Dekamethylcyklopentasiloxan [Czech]
Union carbide 7158 silicone fluid
EINECS 208-764-9
KF 995
SF 1202
VS 7158
BRN 1800166
CYCLOMETHICONE
volasil(tm) 245
dimethylcyclopentasiloxane
Dekamethylcyklopentasiloxan
UNII-0THT5PCI0R
AC1L1W7T
Decamethyl-cyclopentasiloxane
444278_ALDRICH
MolPort-000-153-808
LTBB003119
AG-F-86953
NCGC00163981-01
LS-58254
TR-019129
D1890
Decamethylcyclopentasiloxane (cyclic monomer)

S05475
4-04-00-04128 (Beilstein Handbook Reference)
I14-53249
Cyclopentasiloxane, 2,2,4,4,6,6,8,8,10,10-decamethyl-
2,2,4,4,6,6,8,8,10,10-Decamethyl-1,3,5,7,9,2,4,6,8,10-pentaoxapentasilecane
2,2,4,4,6,6,8,8,10,10-decamethyl-1,3,5,7,9,2,4,6,8,10-pentoxapentasilecane
Cyclopentasiloxane,decamethyl- (6CI,8CI,9CI);Botanisil CP 33;Cyclic dimethylsiloxane pentamer;Cyclo-decamethylpentasiloxane;Cyclopentadimethylsiloxane;Cyclopentasiloxane;DC 345 Fluid;Dow Corning 345 Fluid;Dow Corning 345EU;Execol D5;KF 995;Pentacyclomethicone;SF1202;SH 245 (siloxane);Silbione 70045V5;Silbione V 5;TFS 405;TSF 465;Union Carbide 7158 Silicone Fluid;VS 7158;Volasil 245;VolatileSilicone Fluid 345;

APPENDIX IV: PROPERTIES OF D5

http://pubchem.ncbi.nlm.nih.gov/summary/summary.cgi?q=all&cid=10913#x27

Chemical and Physical Properties	
Color/Form	Oily liquid
Boiling Point	210 deg C
Melting Point	-38 deg C
Density/Specific Gravity	0.9593 at 20 deg C/4 deg C
Octanol/Water Partition Coefficient	log Kow = 5.20
Solubilities	In water, 1.7X10-2 mg/L at 25 deg C [Kochetkov A et al; Environ Toxicol Chem 20: 2184-88 (2001)] PubMed Abstract

Spectral Properties

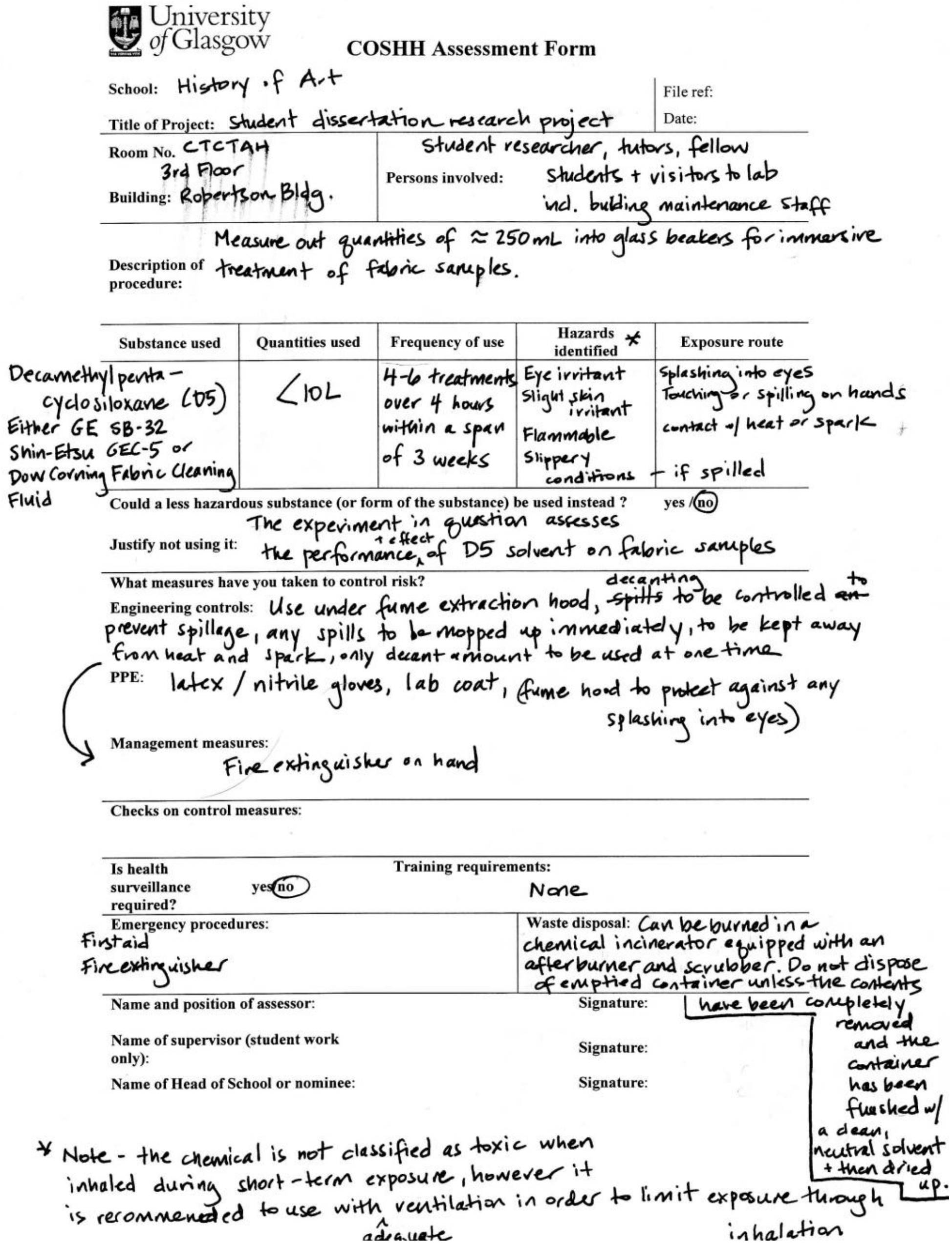
- Index of refraction = 1.3982 at 20 deg C/D
- IR: 5518 (Coblentz Society Spectral Collection)
- MASS: 52165 (NIST/EPA/MSDC Mass Spectral database, 1990 version)

VaporPressure

0.2 mm Hg at 25 deg C (est)

Computed Properties	
Molecular Weight	370.7697 [g/mol]
Molecular Formula	$C_{10}H_{30}O_5Si_5$
H-Bond Donor	0
H-Bond Acceptor	5
Rotatable Bond Count	0
Exact Mass	370.093957
MonoIsotopic Mass	370.093957
Topological Polar Surface Area	46.2
Heavy Atom Count	20
Formal Charge	0
Complexity	258
Isotope Atom Count	0
Defined Atom Stereocenter Count	0
Undefined Atom Stereocenter Count	0
Defined Bond Stereocenter Count	0
Undefined Bond Stereocenter Count	0
Covalently-Bonded Unit Count	1
Effective Rotor Count	2
Conformer Sampling RMSD	0.6
CID Conformer Count	3

APPENDIX V: COSHH FORM



University of Glasgow

COSHH Assessment Form

School: History of Art

File ref:

Title of Project: Student dissertation research project

Date:

Room No. CTCTAH 3rd Floor

Building: Robertson Bldg.

Persons involved: Student researcher, tutors, fellow students + visitors to lab incl. building maintenance staff

Description of procedure: Measure out quantities of ≈ 250mL into glass beakers for immersive treatment of fabric samples.

Substance used	Quantities used	Frequency of use	Hazards identified *	Exposure route
Decamethylpenta-cyclosiloxane (D5) Either GE SB-32 Shin-Etsu GEC-5 or Dow Corning Fabric Cleaning Fluid	< 10L	4-6 treatments over 4 hours within a span of 3 weeks	Eye irritant Slight skin irritant Flammable Slippery conditions	Splashing into eyes Touching or spilling on hands contact w/ heat or spark – if spilled

Could a less hazardous substance (or form of the substance) be used instead ? yes [✓ no]

Justify not using it: The experiment in question assesses the performance + effect of D5 solvent on fabric samples

What measures have you taken to control risk?

Engineering controls: Use under fume extraction hood, ~~spills~~ decanting to be controlled ~~an~~ to prevent spillage, any spills to be mopped up immediately, to be kept away from heat and spark, only decant amount to be used at one time

PPE: latex / nitrile gloves, lab coat, (fume hood to protect against any splashing into eyes)

Management measures: Fire extinguisher on hand

Checks on control measures:

Is health surveillance required? yes [✓ no]

Training requirements: None

Emergency procedures: First aid
Fire extinguisher

Waste disposal: Can be burned in a chemical incinerator equipped with an afterburner and scrubber. Do not dispose of emptied container unless the contents have been completely removed and the container has been flushed w/ a clean, neutral solvent + then dried up.

Name and position of assessor: **Signature:**

Name of supervisor (student work only): **Signature:**

Name of Head of School or nominee: **Signature:**

* Note - the chemical is not classified as toxic when inhaled during short-term exposure, however it is recommended to use with adequate ventilation in order to limit exposure through inhalation

APPENDIX VI: LIST OF SUPPLIERS

GreenEarth

GreenEarth® Cleaning.
51 West 135th Street
Kansas City, MO 64145-1289
(877) 926-0895
President: Tim Maxwell

Alex Reid
Alex Reid HQ
9 Ashville Way
Whetstone
Leicester
LE8 6NU

Tel: 08456 34 44 54
Fax: 0116 275 3838

Standardized Soiled Samples

Materials Research Products LTD

Cotton C-02
Wool C-02

Post & Deliveries: 4 Montpelier Street (#236), London, England SW7 1EX

Telephone: +44 (0)20-7823-4146

E-mail: info@mrpltd.com
Contact: John Byrne

Appendix VII: Reference Spectra for Wool and Cotton

REFERENCE SPECTRA: COTTON

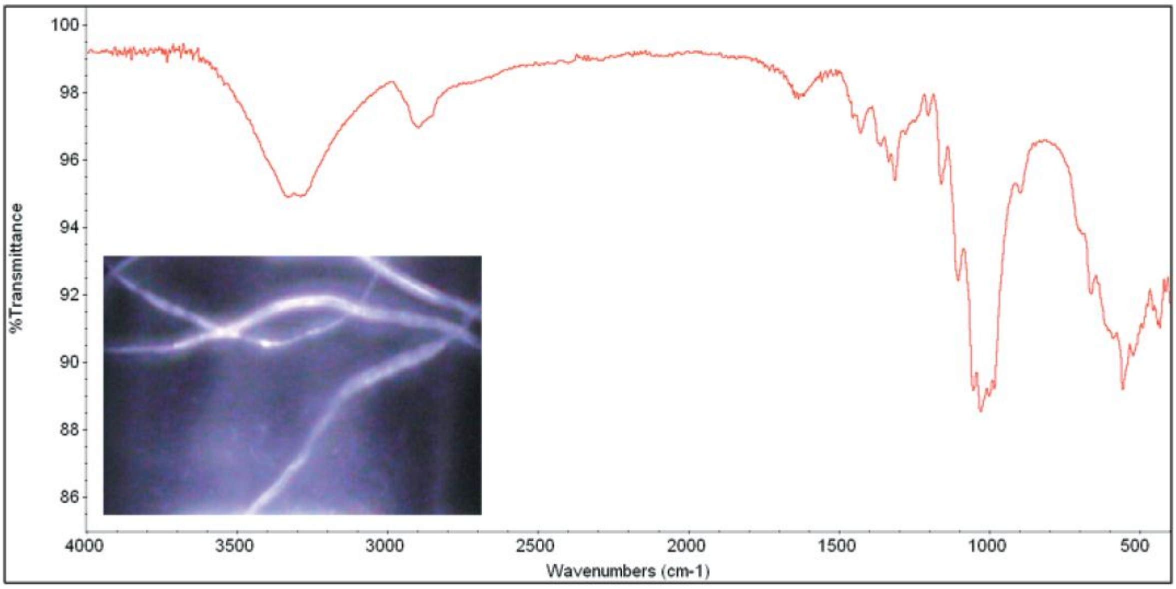


Figure 1. Cotton fibers; diameter 30 micron.

REFERENCE SPECTRA: WOOL

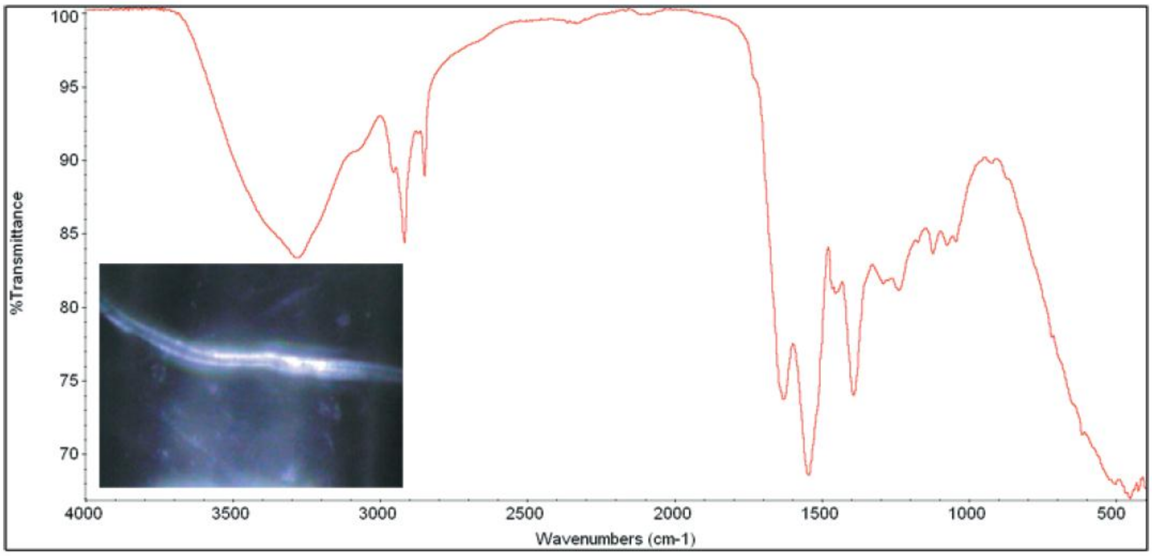
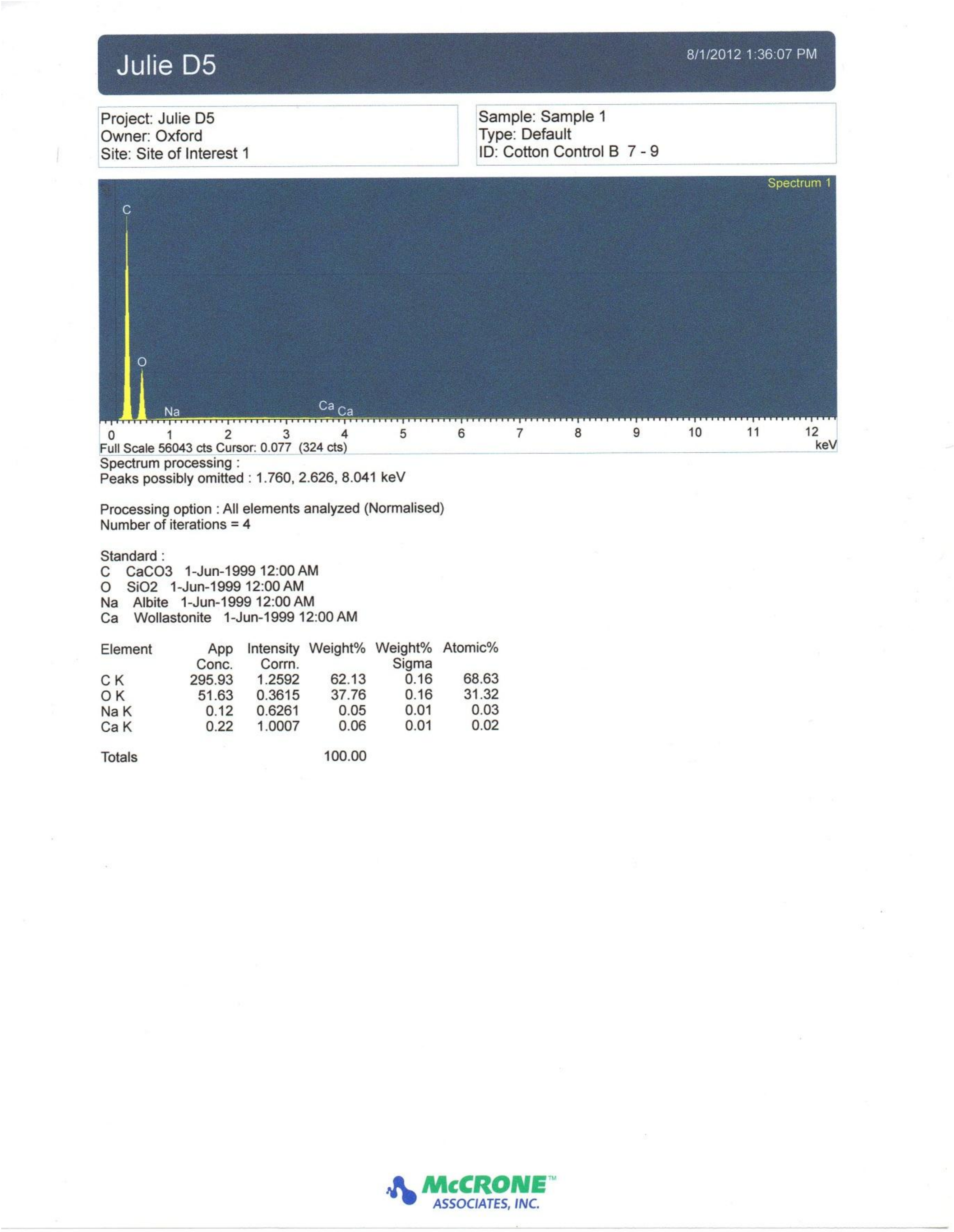


Figure 2. Wool fiber; diameter 30 micron.

Obtained from:

Pike Technologies Spectroscopic Creativity "Fiber Analysis Using FTIR-ATR with Optical Viewing"http://www.piketech.com/skin/fashion_mosaic_blue/application-pdfs/FiberAnalysisUsingFTIR-ATRwithOpticalViewing.pdf (accessed 17 August 2012)

APPENDIX VIII: ELEMENTAL ANALYSIS



Julie D5

8/1/2012 1:36:07 PM

Project: Julie D5
Owner: Oxford
Site: Site of Interest 1

Sample: Sample 1
Type: Default
ID: Cotton Control B 7 - 9

Full Scale 56043 cts Cursor: 0.077 (324 cts)

Spectrum processing :
Peaks possibly omitted : 1.760, 2.626, 8.041 keV

Processing option : All elements analyzed (Normalised)
Number of iterations = 4

Standard :

C	CaCO3	1-Jun-1999 12:00 AM
O	SiO2	1-Jun-1999 12:00 AM
Na	Albite	1-Jun-1999 12:00 AM
Ca	Wollastonite	1-Jun-1999 12:00 AM

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	295.93	1.2592	62.13	0.16	68.63
O K	51.63	0.3615	37.76	0.16	31.32
Na K	0.12	0.6261	0.05	0.01	0.03
Ca K	0.22	1.0007	0.06	0.01	0.02
Totals			100.00		

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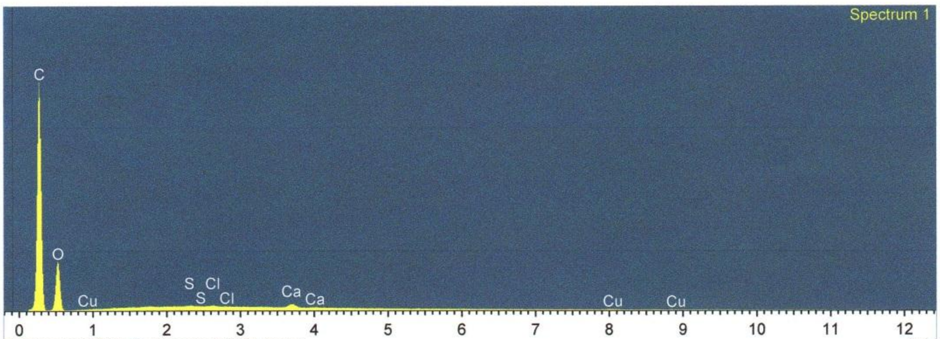
Julie D5

8/1/2012 2:00:14 PM

Project: Julie D5
Owner: Oxford
Site: Site of Interest 1

Sample: Sample 2
Type: Default
ID: Cotton Treated B 10-12



Full Scale 45427 cts Cursor: -0.087 (0 cts)

Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 4

Standard :
C CaCO3 1-Jun-1999 12:00 AM
O SiO2 1-Jun-1999 12:00 AM
S FeS2 1-Jun-1999 12:00 AM
Cl KCl 1-Jun-1999 12:00 AM
Ca Wollastonite 1-Jun-1999 12:00 AM
Cu Cu 1-Jun-1999 12:00 AM

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	220.00	1.2691	64.78	0.20	71.24
O K	31.66	0.3419	34.61	0.20	28.57
S K	0.14	0.9567	0.05	0.01	0.02
Cl K	0.15	0.8341	0.07	0.01	0.02
Ca K	0.83	1.0008	0.31	0.01	0.10
Cu K	0.38	0.7625	0.19	0.02	0.04
Totals			100.00		

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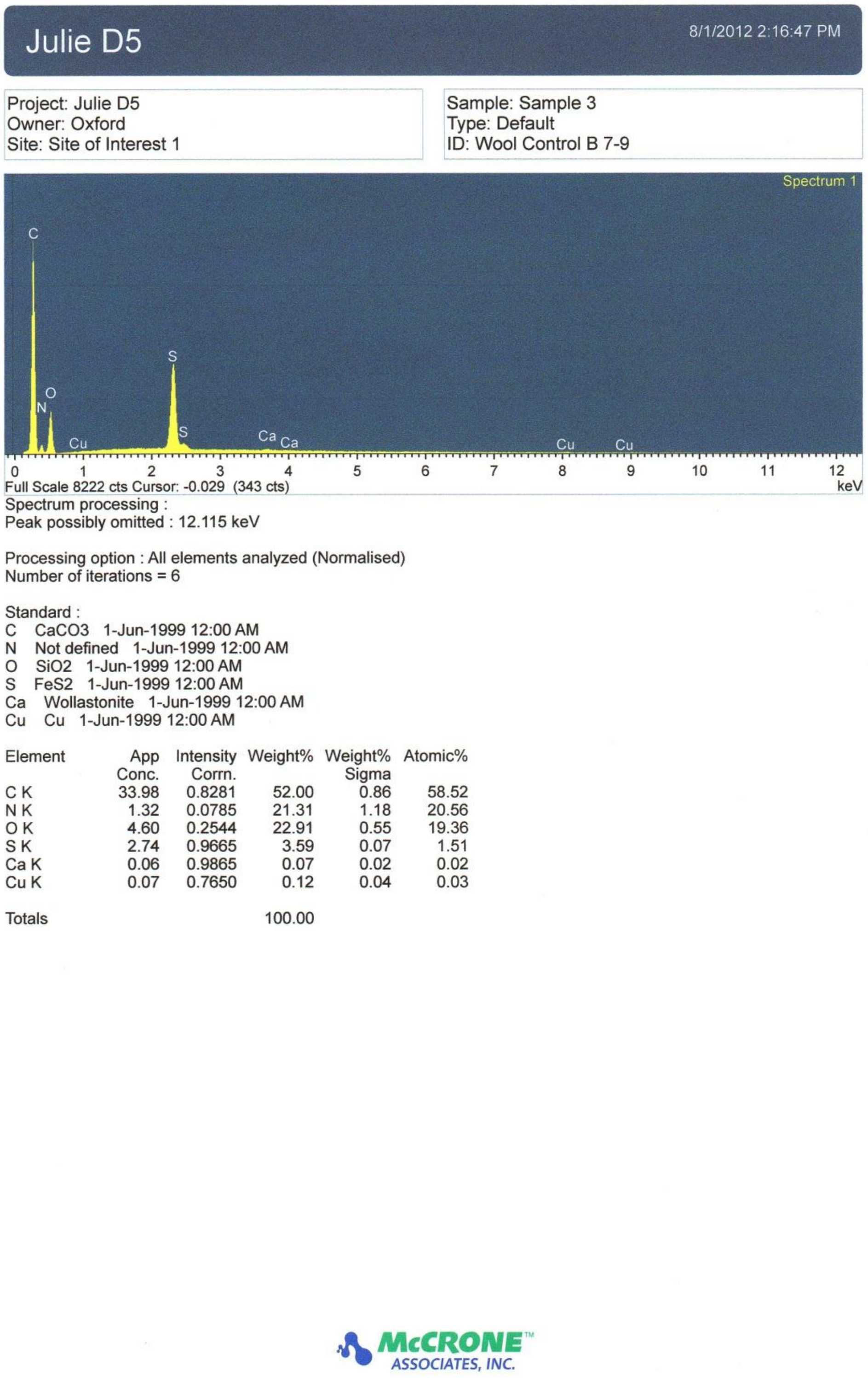

Julie D5

8/1/2012 2:16:47 PM

Project: Julie D5
Owner: Oxford
Site: Site of Interest 1

Sample: Sample 3
Type: Default
ID: Wool Control B 7-9

Full Scale 8222 cts Cursor: -0.029 (343 cts)

Spectrum processing :
Peak possibly omitted : 12.115 keV

Processing option : All elements analyzed (Normalised)
Number of iterations = 6

Standard :
C CaCO3 1-Jun-1999 12:00 AM
N Not defined 1-Jun-1999 12:00 AM
O SiO2 1-Jun-1999 12:00 AM
S FeS2 1-Jun-1999 12:00 AM
Ca Wollastonite 1-Jun-1999 12:00 AM
Cu Cu 1-Jun-1999 12:00 AM

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	33.98	0.8281	52.00	0.86	58.52
N K	1.32	0.0785	21.31	1.18	20.56
O K	4.60	0.2544	22.91	0.55	19.36
S K	2.74	0.9665	3.59	0.07	1.51
Ca K	0.06	0.9865	0.07	0.02	0.02
Cu K	0.07	0.7650	0.12	0.04	0.03
Totals			100.00		

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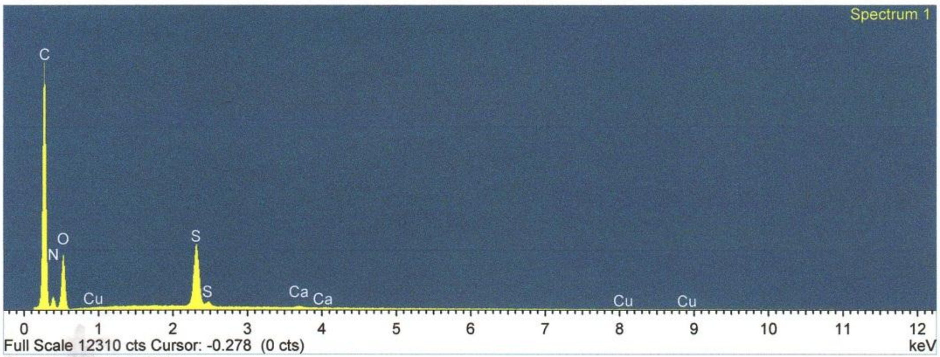
Julie D5

8/1/2012 2:31:27 PM

Project: Julie D5
Owner: Oxford
Site: Site of Interest 1

Sample: Sample 4
Type: Default
ID: Wool Treated B 10-12



Full Scale 12310 cts Cursor: -0.278 (0 cts)

Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 6

Standard :
C CaCO3 1-Jun-1999 12:00 AM
N Not defined 1-Jun-1999 12:00 AM
O SiO2 1-Jun-1999 12:00 AM
S FeS2 1-Jun-1999 12:00 AM
Ca Wollastonite 1-Jun-1999 12:00 AM
Cu Cu 1-Jun-1999 12:00 AM

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	54.55	0.9273	47.56	0.61	53.74
N K	2.56	0.0875	23.61	0.89	22.88
O K	8.55	0.2620	26.35	0.45	22.36
S K	2.76	0.9586	2.33	0.04	0.99
Ca K	0.09	0.9919	0.07	0.01	0.02
Cu K	0.08	0.7656	0.09	0.02	0.02
Totals			100.00		

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