

Farmer, Beatrice (2012) Friendly Fungicides: testing fungicides containing tea tree oil (melaleuca alternifolia) to determine their safety for use on historic cellulosic textiles. [MPhil]

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Friendly Fungicides: Testing fungicides containing tea tree oil (melaleuca alternifolia) to determine their safety for use on historic cellulosic textiles

by

Beatrice Farmer

Supervisor: Dr Anita Quye

Submitted in partial fulfilment of the requirements for the Master of Philosophy in Textile Conservation in the School of Culture and Creative Arts,

University of Glasgow, 23rd August, 2012.

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Abstract

This research explores new fungicide treatments containing tea tree oil (Melaleuca alternifolia) and will use a comparison with the product Preventol[®] ON-S. The effect on the condition of both a new cotton fabric and a cotton textile from the Second World War British Utility Scheme has been determined using colourimeter, FTIR-ATR, surface pH, and tensile strength testing. More recent understanding of the types of fungi found on cellulosic textiles and their growth mechanisms has been amalgamated, though the fungicides have not been tested directly on fungi. An overview of current practices used in textile conservation has informed both the theoretical principles and experimental design. Two treatment methods were used, immersion and spot cleaning suing a vacuum suction table. The spot cleaning treatments for both fungicides were found to be unviable due to the vacuum causing undesirable staining and build up on the samples. The Preventol[®] ON-S was found to have an overall negative effect on the condition of the textiles, but the tea tree oil with emulsifier was determined an effective treatment, causing neutral or beneficial effects on the condition, except on the tensile strength for the new cotton samples.

List of Abbreviations

| ATR | Attenuated Total Reflectance |
|--------|---|
| BS | British Standards |
| CC41 | Civilian Clothing Act of 1941 |
| CFU | Colony Forming Unit |
| cm | Centimetre |
| c.m.c | Critical Micelle Concentration |
| COSHH | Control of Substances Hazardous to Health |
| DP | Degree of Polymerisation |
| EEC | European Economic Community |
| FTIR | Fourier Transform Infrared Spectroscopy |
| HEPA | High-Efficiency Particulate Air (filter) |
| ICBCP | International Conference on Biodeterioration of Cultural Property |
| IMS | Industrial Methylated Spirits |
| ISO | International Organisation for Standardisation |
| MFC | Minimum Fungicidal Concentrations |
| ml | Millilitre |
| mm | Millimetre |
| MSDS | Material Safety Data Sheet |
| Ν | Newtons |
| Na-OPP | Sodium o-phenyl phenol |
| рН | Power of Hydrogen |
| PPE | Personal Protective Equipment |
| QAC | Quarternary Ammonium Compounds |
| RH | Relative Humidity |
| SCCP | European Commission Scientific Committee on Consumer Products |
| SD | Standard Deviation |
| тто | Tea Tree Oil |
| UV | Ultraviolet (light) |

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CHAPTER ONE

Introduction and Rational for Research

The reason for identifying the topic of mould removal for research was primarily due to the prevalence of fungi encountered on historic textiles and a lack of extensive recent literature on the topic. Biodeterioration of textiles has not been widely studied within the field of conservation and methods of targeting this source of degradation have been hindered due to the damaging effects of the antifungal agents to textiles. A personal curiosity in fungi, their growth cycles and the source of the coloured staining also drove the desire to pursue this research topic. Fungi are most harmful to cellulosic textiles, as some species are able to break down the cellulose molecules and use the textile as a food source. It was determined therefore that research into cellulosic textiles would be of greatest significance.

The primary method of preventing fungal growth lies with the control of environmental conditions and this should be considered when caring for a collection of heritage objects. It was however beyond the scope and intentions of this work to reevaluate preventive measures, disaster planning and methods of dealing with large outbreaks. A treatment was sought to prevent further fungal growth on textile objects and remove fungal remnants with the inactivation or destruction of the fungi.

It could be argued that the most common method of removal used by conservators, vacuum suction, may be sufficient. It does remove a considerable amount of the fungi present and with correct Personal Protective Equipment (PPE) and appropriate precautions with ventilation it is an effective method. However, vacuum suction is not entirely effective in removing particulates held within the weave structure. Suction may not remove all fungal deposits, both with the active fungal colonies and the characteristic staining that is a result of these colonies. It cannot penetrate within the cotton fibre itself although the fungi can. Any fungi remaining within the textile could become active and develop new colonies. Therefore a treatment method which inhibits the growth of fungi and removes the persistent remnants could be a great advantage to practicing conservators.

Chemicals can cause the activation of dormant fungi and many of these are likely to be used in a conservation treatment. Detergents, organic acids, alcohols, and other solvents could cause rapid colonisation as a result of contamination before or during treatment.¹ Therefore, if a safe fungal treatment could be found it could be useful for prevention or in response to a known outbreak. The fungicidal treatment would need to be safe for regular

¹ G. Caneva et al., *Plant Biology for Cultural Heritage: Biodeterioration and Conservation*, (Santa Monica: Getty Conservation Institute, 2009), 68.

use by conservators without expensive or unmanageable Health and Safety precautions. The treatment should also be safe to use on a historic textile. Bleaching treatments have also been used previously but they cannot be carried out repeatedly due to the damage and yellowing caused over time. This is the drawback of current fungicides, such as Preventol[®] ON-S, which have been found to be effective on fungi, but harmful to textile condition and of high toxicity. Preventol[®] ON-S contains sodium o-phenyl phenol which is effective in promoting dormancy in fungi,² but can catalyse degradation reactions leading to changes in colour and brightness.^{3,4}

During research of the fungicides currently available commercially, a formulation using tea tree oil was found. Unfortunately it is not available for sale in the UK, meaning it would not be easily accessible to practicing conservators in this country and any other constituents in the product were also unknown. Although this product was not available for testing, it initiated the investigation into the potential of tea tree oil in this context. Research into the effectiveness of tea tree oil against fungi was deemed promising,⁵ but no information on the effect of the oil on the condition of textiles could be found. Therefore an initial enquiry into the impact of tea tree oil on textiles has been carried out, which indicated whether further testing would be necessary with fungi itself. In order to evaluate the effect of the oil another fungicide already used in textile conservation, Preventol[®] ON-S, will be used as a comparison in a series of condition tests. Two types of cotton were used, a new cotton lawn and a cotton produced under the British Utility Scheme (1941-1952). This will allow an analysis of the effect on both a new and aged cellulosic textile.

Literature Review

² S. Bertalan (ed.), '12. Mold/Fungi', *Paper Conservation Catalog: The American Institute for Conservation of Historic and Artisitic Works*, (1994), 17.

³ O. Abdel-Kareem, 'The Long-Term Effect of Selected Conservation Materials Used in the Treatment of Museum Artefacts on some Properties of Textiles', *Polymer Degradation and Stability*, Vol. 87, (2005), 121-130.

⁴ Á. Tímár-Balázsy & D. Eastop, *Chemical Principles of Textile* Conservation, (Oxford: Butterworth-Heinemann, 2007), 299.

⁵ K.A. Hammer et al., 'Antifungal Activity of the Components of *Melaleuca alternifolia* (tea tree) oil', *Journal of Applied Microbiology*, Vol. 95, (2003), 853-860.

The literature has been analysed in relation to the fungi associated with cellulosic textiles and the use of fungicides on textiles, particularly the specific fungicides used in this study. It was intended that an overview of these subject areas would provide a context for the study and a restricted reference list for ease of referral.

Fungi

Caneva, G. et al., *Plant Biology for Cultural Heritage: Biodeterioration and Conservation*, (Santa Monica: Getty Conservation Institute, 2009).

This work is a highly useful and in depth examination of the subject. It covers different types of organisms encompassing fungi, bacteria, lichens and bryophytes and links their structure and modes of growth to their impact on heritage objects. Many materials are examined, including textile, paper, stone, metal and paint. It provides a detailed and clearly understandable description of the growth of the fungi found on heritage objects and the consequences of colonisation. The sections directly related to fungi and cellulosic textiles are limited in length but contain much information and proteinaceous fibres are also included. Therefore this work supplies an extremely useful and comprehensive introduction to the subject.

Florian, M.-L., Fungal Facts: Solving Fungal Problems in Heritage Collections, (London: Archetype, 2002).

A more detailed and minute description of fungi, their categorisation and life cycles is included in Florian's work, who is one of the foremost authors in the field. A previous understanding of the topic and a working knowledge of biological terms would be of advantage as some of the language is complex for a non-specialist. The effects of fungi on different heritage materials is then developed into more practical ideas on modes of collection recovery and future monitoring methods. This culminates in a chapter on prevention and disaster preparedness. The work as a whole supplies a exhaustive examination of the issues of fungi in relation to heritage objects and collections as a whole. A theoretical description of fungi then leads on to a more practical action plan that could be significantly beneficial if implemented correctly.

Montegut, D., Indictor, N. and Koestler, R.J., 'Fungal Deterioration of Cellulosic Textiles: A Review' International Biodeterioration Bulletin, (Barking: Elsevier Science Publishers Ltd., 1991), pp. 209-226.

This paper contributes a literature review from 1940 to the time of writing in 1991 and includes a great number of sources. It looks at the structure of cellulosic fibres and links this to the degradation of textiles by fungi. The current thinking on the modes of cellulolytic attack are described and chemical protectants and biocide testing is utilised to review the effectiveness of contemporary methods. Sodium o-phenylphenol is listed as well as another effective type of fungicide, quarternary ammonium compounds (QAC). The relevant sources are included and the factors controlling the effectiveness of these treatments are also investigated. Useful guidance on appropriate environmental controls includes the effects of varying RH, such as the minimum safe level that does not harm cellulosic textiles at 0.4a_w. The full literature review shows the development of the field and thought on the efficacy of different measures to counteract fungal attack.

Fungicides

Nugari, M. and Priori, G., 'Fungicides for Use on Textile Employed during the Restoration of Works of Art', *International Biodeterioration*, Vol. 23, (1987), pp. 295-306.

The purpose of this research was to find suitable fungicides that could be used in the restoration of works of art both as an immediate treatment against active growth and as a preventative measure. Eight fungicides were tested that included sodium o-phenylphenol on six fungal species based on the British Standard BS 6085:1981. Species of fungi were chosen based on those known to have cellulolytic activity and cultivated in agar suspensions. There are no instructions of the specific parameters used during the fungicidal treatments, including concentrations and lengths of immersion, which prevents replication. The efficacy of the treatments are listed and reviewed. It is stated that Na-OPP initially discoloured the textile and yellowed after ageing in the dark but retained the highest brightness values after UV ageing. Due to the low toxicity compared to the other biocides it was recommended for use with silk, but p-chloro-m-cresol was determined the most effective. However, it has been determined as highly toxic with the acute oral LD50 in rats at 5129 mg/kg,⁶ compared with 2500 LD₅₀ oral in rats for Preventol[®] ON-S. Therefore the research provides a good scientific analysis of the effects but does not elucidate the ways each fungicide acts on fungi or the testing methods.

⁶ United States Environmental Protection Agency, *Reregistration Eligibility Decision (RED) p-Chloro-m-cresol*, www.epa.gov/oppsrrd1/REDs/3046red.pdf, (1997). Accessed 7/8/2012, 6.

Abdel-Kareem, O., 'Evaluating the Combined Efficacy of Polymers with Fungicides for Protection of Museum Textiles against Fungal Deterioration in Egypt', Polish Journal of Microbiology, Vol. 59, no. 4, (2010), pp. 271-280.

Fungi were isolated from the collection of historical textiles in the Egyptian Museum and the Coptic museum in Cairo, where Abdel-Kareem is based in the Conservation Department of Cairo University. He has carried out numerous studies on this topic which have been published widely, and form the current research on fungicides for use in textile conservation. Twenty-two species were found using textile samples and sterilised cotton swab techniques. They are primarily of the Aspergillus and Penicillium species due to improper storage conditions and the highly humid climate. Two fungicides were tested, Preventol[®] O-Na produced by Bayer that is now available as Preventol[®] ON-S by Lanxess, and Neo-Desogen, a guarternary ammonium compound. The two fungicides are known to catalyse degradation reactions, so they were tested with adhesives and consolidants using the impregnation method and exposed to the isolated fungal species. Condition testing was then employed consisting of colourmetric measurements, tensile strength and elongation to determine the effect of the treatments. Results were compared to untreated samples and found that Klucel G and 2% Neo-Desogen was the most effective at preventing fungal attack and strengthened the textiles. This could be a useful preventative measure on a textile that warrants an adhesive treatment where there is a probability of humid storage conditions as it allows the fungicide to be held within the adhesive rather than oxidising or evaporating.

Gatenby, S. and Townley, P., 'Preliminary Research into the Use of the Essential Oil of *Melaleuca alternifolia* (tea tree oil) in Museum Conservation', *The 5th International Conference on Biodeterioration of Cultural Property (ICBCP-5): 12-14 November 2001, Sydney ,Preprints, lucite.org/lucite/archive/art_-_painting/ icbcp5_papers.pdf.* Accessed 31/07/2012. pp. 134-139.

This paper explains the research carried out at the Museum of Applied Arts and Sciences in Sydney, Australia. It is the primary application of tea tree oil in conservation using a Bactigas[®] aerial spray to control a large outbreak of mould in a museum store. The history and use of tea tree oil and previous scientific research is listed followed by the influence of environmental factors on the outbreak in the store. Initial trials by Bioassay were carried out using a Bactigas[®] concentrate (10% tea tree oil in ethanol) that achieved a reduction in 10³ of mould over seven days, which shows the effectiveness of the essential oil. A less concentrated solution was found to be more miscible though at the time of publishing the full treatment had not been carried out. The purpose of the aerial spray was to kill the active mould so that the remnants could be cleaned without immediate environmental controls being possible. This work forms the notable implementation of tea tree oil as a fungicide for use with heritage objects, and initiated the pursuance of this dissertation research.

Research Questions

- 1 Literature
- What are the categories and growth cycles of the fungi affecting cotton?
- · What is the effect of fungi on the condition of cotton?
- What prior research exists on Preventol® ON-S and Tea Tree Oil?
- 2 Experimental Design
- What is the effect on the condition of cotton of the fungicides Preventol[®] ON-S and Tea Tree Oil?
- Does the method of application affect the condition of the samples? (Immersion, spot cleaning using a vacuum suction table)
- 2 Condition Assessments
- Do the products produce a colour change in the samples?
- Is the pH of the textile affected by the treatment?
- · Are any residues left by the fungicide treatments?
- Is the tensile strength reduced after treatment?

Objectives

- 1 Literature
- Examine the literature for information on fungi in relation to cellulosic textiles.
- Determine the effect of fungi on the condition of cotton.
- Investigate the chemical properties of Preventol[®] ON-S and tea tree oil to analyse their strengths and potential risks.
- 2 Experimental Design
- Carry out treatments on the samples using the specified fungicides.
- Use varying application methods- immersion and spot cleaning. Visual and physical observations will be made during treatments. Repeat on 3 samples.
- **3** Condition Assessments
- Test the effect of these fungicides on the condition of the fibres using: colourimeter, FTIR, surface pH, tensile strength and elongation testing. Repeat readings 3 times.

CHAPTER TWO

Fungi

Categorisation and Growth Cycles of Fungi

Identification and culturing of fungi has not formed part of the experimental study, but is intended to provide context and useful information. It is advantageous to determine the exact species that is present so that precise growth patterns of the colony forming unit (CFU) can be predicted. Different species can also require widely varied minimum fungicidal concentrations (MFC) to work effectively. With a large outbreak it may also be useful to determine the toxicological potential of the species in order that adequate health and safety precautions can be made, for example ventilation with HEPA filters.⁷ Methods of identification have been described extensively using low- and high-tech approaches and could be used in practice by many conservators.^{8,9}

Since the 1960s the categorisation of fungi has greatly evolved and now delineates the type of fungi affecting historic objects as the *Eumycota* kingdom. They are thus classified due to their multicellular state and are further divided into the phylum *Zygomycota* (subphylum conjugation fungi) and phylum *Dikaryomycota* (subphylums *Ascomycotina* and *Basidiomycotina*). It is the latter phylum that attacks cellulosic materials as the former only digests starch and sugars. Therefore the *Zygomycota* could degrade the stains or finishes found on textiles but the *Dikarymycota* could grow and feed on cellulosic substrates.

Spores are heterotrophic, meaning they require nutrients from external organic compounds excepting the conidia, which have a reserve to enable the primary stage of activation. Spores originate through sexual (teleomorphic) or asexual (anamorphic) reproduction and have the function of dispersal and perpetuation of the species.¹⁰ Some species have both sexual and asexual states, which can complicate classifications and cause confusion. For the purpose of this study, the most common method for each species will be used and only those fungi affecting cellulosic textiles will be included, as listed in Fig. 1.

⁷ S. Guild & M. MacDonald, *CCI Technical Bulletin No. 26 Mould Prevention and Collection Recovery: Guidelines for Heritage Collections*, (Ottawa: Canadian Conservation Institute: 2007), 1-29.

⁸ O. Abdel-Kareem, 'Monitoring, Controlling and Prevention of the Fungal Deterioration of Textile Artifacts in the Museum of Jordanian Heritage', *Mediterranean Archaeology and Archaeometry*, Vol. 10, no. 2, (2010), 85-96.

⁹ Bertalan (ed.), 1994, 15-17.

¹⁰ Caneva et al., 2009, 68.

Fungi have a rigid cell wall made primarily of polysaccharides such as chitin, a polymer of N-acetylglucosamine, and β -D-glucan, with associated lipids, amino sugars, proteins, and glycoproteins. The cell wall of pigmented mycelium also contains melanines that aid resistance against agents causing chemical and physical degradation such as UV radiation, y- and x-rays, and attack by enzymes. Melanin consists of quinones and hydroquinones that restrict damage by free radicals, but also cause discolouration of textiles.¹¹ Fungi also have the capacity to produce enzymes, which are chains of amino acids that can aid degradation.

| Phylum | Subphylum | Order | Sexual Reproduction | Asexual Reproduction |
|---|---|---|---|---|
| <i>Zygomycota</i> - consume starch and sugars - nonseptate hyphae (no cell walls) | Zygomycota | Mucorales | Zygospores Genus: <i>Rhizopus</i> <i>Mucor</i> | Spores Genus: <i>Rhizopus</i> |
| Dikaryomycota | <i>mycota</i> me cellulose e hyphae | Asci with ascospores Genus: <i>Alternaria</i> <i>Chaetomium</i> | Conidia Genus: Aspergillus Eurotium Penicillium | |
| - consume cellulose - septate hyphae (separate cells) | | Sordariales | | Cladosporium Trichoderma Fusarium Stachybotrys Memnoniella Scopulariopsis Neurospora Myrothecium |

Figure 1: The Fungi Affecting Cellulosic Textiles

Sexual Reproduction

Fungi that reproduce sexually are known as zygospores and ascospores. The genus *Chaetomium* of the order *Sordariales* is particularly cellulolytic and is found in dirty water and soil, so it commonly affects burial textiles. Sexual reproduction creating ascospores takes place through the formation of a hook on the hypha, which ensures correct cell division. An ascus is then developed within an ascocarp, or emanating body. Nuclear fusion or karyogamy in the ascus then leads to meiosis with the production of ascospores. The ascopores then germinate to form mycelium.

¹¹ Caneva et al., 2009, 66.

Asexual Reproduction

Most of the fungi that are found on heritage objects are of a subdivision of *Dikarymycota* commonly known as conidial fungi (but also *Deuteromycotina*, *Deuteromycetes*, or fungi imperfect) due to their asexual reproduction. Asexual reproduction occurs through specialised hyphae that produce spores called conidia by mitosis, which can arise through multi- or uni-cellular fungi. The species *Aspergillus* and Penicillium of the order *Eurotiales* are the more common blue and green moulds and are transmitted through airborne conidia (see Fig. 2).¹²

Figure 2: Conidium¹³

Germination of Conidia and Environmental Catalysts

Spores, whether produced by sexual or asexual reproduction, can be unicellular or multicellular. They may also have smooth or elaborate surfaces, and be immobile or mobile through use of flagella, or tails. They have a thick and sometimes pigmented cell wall and usually become dormant before activation and germination which occurs when the environmental conditions are favourable.¹⁴

¹² M.-L. Florian, *Fungal Facts: Solving Fungal Problems in Heritage Collections*, (London: Archetype, 2002), 31-39.

¹³ Welcome Images, http://www.flickr.com/photos/wellcomeimages/5987578301/sizes/z/in/ photostream/. Accessed 27/4/12.

¹⁴ Caneva et al., 2009, 68.

The conidium may be dormant moving in the air or on a substrate until the required levels of water, pH, temperature and nutrients are present. The activation of conidia involves the adsorption of water and the swelling and rupturing of the cell walls with an increase in metabolic activity requiring further nutrients than those already held within the conidia. This leads to germination and once the conidium is germinated it cannot revert to the dormant state. The development of new materials for the cell wall allows the emergence of the germination tube, which will create the hypha (Fig. 3). Hyphae, or vegetative growth, then establish colonies known as mycelia (Fig. 4). This can be stalled through refrigeration, low or high pH or temperatures, or dehydration, and they can survive dormant for up to ten years under laboratory conditions. This inactive state will be swiftly reversed, often in under one hour, once the fungi are returned to more conducive environments.¹⁵

The difficulty in determining these causative environmental factors is that the various fungal species require opposing conditions, meaning textiles may always be susceptible to infection despite controls. Some can survive dormant below 0°C to 70°C, and favour an RH of above 60%.¹⁶ It has been found that if the water activity (a_w) is lower than 0.61 fungal development cannot take place.¹⁷ A pH of 4-6.5 has been found to produce cellulolytic fungi, meaning that acidic conditions should be avoided, which would be aided by the removal of the acidic products of cellulose degradation.¹⁸

Figure 3: Hyphae¹⁹

Figure 4: Mycelia (colony)²⁰

¹⁵ Florian, 2002, 31-39.

¹⁶ D. Montegut, N. Indictor & R.J. Koestler, 'Fungal Deterioration of Cellulosic Textiles: A Review' in *International Biodeterioration Bulletin*, (Barking: Elsevier Science Publishers Ltd., 1991), 217-220.

¹⁷ Caneva et al., 2009, 115-116.

¹⁸ Montegut et al., 1991, 217-220.

¹⁹ Wikipedia, http://upload.wikimedia.org/wikipedia/commons/7/74/Aspergillus_niger_01.jpg. Accessed 27/4/12.

²⁰ Wikipedia, http://upload.wikimedia.org/wikipedia/commons/e/eb/20100815_1818_Mold.jpg. Accessed 27/4/12.

Properties of Historical Cotton

Appearance

The structure of cotton must be first understood before mechanisms of degradation can be examined, which will then inform the effect of fungi and fungicides. Cotton textile fragments have been found dating from 5000 BC in Pakistan and Mexico and it is thought that the cotton *Gossypium* plants were cultivated for clothing from around this time. Cotton is a seed hair taken from the boll of the plant, with between 4000 fibres and 250,000 fibres obtained from one boll. They are staple fibres, 0.32-6.35cm in length, with most used for spinning 2.22-3.18cm. Cotton has one of the narrowest diameters compared to other natural fibres, with a relatively uniform width to more mature fibres. More immature fibres have a greater irregularity and are thinner in the middle than the ends. Mature cotton appears to be a flat twisted ribbon, with the twist being the most easily identifiable characteristic. If a cross-section is taken, it appears in the shape of a kidney bean.²¹ The cross section and longitudinal features are apparent in the images of Fig. 5.

Figure 5: Cross section and Longitudinal Views of Fibres Under Magnification²²

Physical Structure

The physical structure is illustrated in Fig. 6. It reveals the hollow lumen that is surrounded by primary and secondary cell walls with the slightly different construction of the winding layer separating the two. These layers are made up of elementary fibrils of around 100 polymer chains, and 15 elementary fibrils form a microfibril that are further organised into macrofibrils. Fibrils usually spiral around the longitudinal axis of the fibre or they can organise in parallel.²³ The primary cell wall is then covered by a waxy outer cuticle which is inert and designed to protect the inner structure from degradation. Manufacturing processes such as scouring and bleaching gradually remove this outer cuticle.²⁴

²¹ K. Hatch, *Textile Science*, (Apex: Tailored Text Custon Publishing, 2006), 163-164.

²² Cotton Fibres, http://www.freewebs.com/textile-technology/fibrescience.htm. Accessed 6/8/2012.

²³ Tímár-Balázsy & Eastop, 2007, 22-23.

²⁴ Hatch, 2006, 164.

Chemical Structure and Properties

Cotton is predominantly made of cellulose at 99% with the remaining material being hemicellulose and pectin. It has a high Degree of Polymerisation (DP) at 11,000 which makes the fibres mechanically strong. The crystallinity is 70% and due to this relatively high proportion of the ordered crystalline areas, there are less amorphous areas which are more prone to degradation. This is due to the linear structure of cellulose with the polysaccharide built up from β -D glucose units that are formed in ring shapes, with the chemical formula C₆H₁₂O₆. Two β -D glucose units form one cellobiose (Fig. 7), which is the monomer of cellulose, through a condensation reaction. The presence of small side groups allow many intermolecular hydrogen bonds, dipole and van der Waals forces leading to greater strength and a low %elongation of 7-3%.²⁶

Figure 7: Cellobiose Unit²⁷

²⁵ Structure of Cotton, http://textilefibre.blogspot.co.uk/2012/05/macro-structure-of-cotton-fiber.html. Accessed 6/8/2012.

²⁶ Tímár-Balázsy & Eastop, 2007, 10-24.

²⁷ Cellobiose, http://wolverton.owu.edu/plantphys/cell-wall.html. Accessed 6/8/2012.

Degradation Mechanisms of Cotton

Degradation may be caused by fungal attack or the fungicides used to inhibit the fungi. It is therefore important to identify the causes which can then be linked to fungi and fungicides. Degradation begins in the amorphous regions and is caused by high temperatures, extremes in RH (outside the acceptable 45-65%), light, soiling, and acid or alkaline conditions. Biodeterioration also occurs through bacteria, enzymes and fungal attack, which will be examined following the chemical and physical mechanisms of degradation.

Oxidation is a primary mechanism of degradation and can be caused by exposure to light, particularly Ultraviolet (UV) light, and is catalysed by higher temperatures. Every 10° rise above 20°C doubles the rate of degradation reactions. Oxidation first occurs to the hydroxyl side groups of cellulose, leading to free radicals that attack the polymer and cause chain scission. This results in a decrease in the DP and lower mechanical strength. Chain fragments are evident in the yellow acidic products of degradation that increase the acidity of the textile and perpetuate deterioration, though they are water soluble.

The other main mechanism of degradation is hydrolysis. It takes place through the donation of a hydrogen atom from either an acid present or excess water. The addition of the hydrogen breaks the chain of β -D glucose rings and creates hydrocellulose. These shorter chain fragments also decrease the DP and increase crystallinity through intermolecular hydrogen bonds. However, rather than making the fibres stronger, they are more rigid and brittle. Alkalis attack the last unit of the cellulose chain, due to the presence of an aldehyde group, and gradually peel away the end of the chain. It forms a complex set of reactions known as alkaline depolymerisation.²⁸

²⁸ Tímár-Balázsy & Eastop, 2007, 25-31.

Fungal Degradation of Cotton

Fungi cause degradation through cellulolytic attack by enzymes produced by fungi. The enzymes produced are classified as hydrolases because they speed up hydrolytic reactions. They are twisted chains of amino acids which catalyse this reaction by fitting particular substrates into pockets within molecules and create easily digestible simple sugars. These sugars provide nutrients and allow the fungi to reproduce further, self perpetuating the growth of the mycelia and deterioration of the textile.

Firstly the amorphous regions are attacked before the more resistant crystalline regions are targeted. Degradation occurs in the extra-cellular cellulase, which acts directly on the substrate, and also through the action of intra-cellular enzymes. Fungi can penetrate deeply into the fibres. Evidence of fungi reaching the internal structure has been found with hyphae being seen in the lumen of cotton. It is visible under magnification due to the coloured nature of some fungal species. Blackish-brown spots are probably fungal species with melanin contained in the cell wall. Reddish-purple tones are the product of the release of indelible exopigments, for example *Fusarium* and *Aspergillus* or the ascomycete *Chaetomium*. Coloured staining on a textile is evident in Fig. 8.

Figure 8: Fungal Staining on Fabric²⁹

Biodegradability is increased by previous degradation reactions. If textiles are already more degraded they have a lower DP or reduced crystallinity and are more susceptible to biological attack.³⁰ The presence of non-cellulosic materials like pectin and hemicellulose makes the fibre structure more prone to fungal degradation due to their more amorphous arrangement.³¹ The water solubility of degraded cellulose chain fragments also makes

²⁹ ehow, http://www.ehow.co.uk/how_7767454_remove-black-mold-spots-fabric.html. Accessed 27/4/12.

³⁰ Caneva et al., 2009, 115-116.

³¹ Montegut et al., 1991, 211-212.

them more easily metabolised, which is comparable with starch. Starch may be found on cotton in food soiling or through the historic use of starch to aid pressing. Organic acids released as metabolites also induce a reduction in elasticity and strength.

The weave structure has an impact and fungi can develop on the surface of a textile. Open weave fabrics allow more dust and biological contaminants to gather, with the type of weave structure and thread count also influencing this. Tighter weave structures with a lower thread count have greater resistance to penetration by fungi. Other components often found on textiles such as metal threads or leather elements will also effect the susceptibility to fungal attack.³² The breakdown of the cellulose structure engenders mechanical damage and if untreated will lead to the partial or complete loss of the object.

Health and Safety

Different species can infect cellulose or amino acids, human respiratory systems and skin conditions.³³ The species *Stachybotrys*, also known as black rot, feeds on cellulose and is particularly harmful to the health of those exposed. It produces mycotoxins than if inhaled or ingested can induce chronic fatigue, headaches, fever, irritation to the respiratory system and in extreme reactions vomiting and bleeding in the lungs. This is why when dealing with large mould outbreaks it can be useful to identify the species to aid assessment of the risks.

Health and Safety guidelines have also been set up to aid personal protection and prevent harm to individuals dealing with fungal problems. PPE of goggles, respiratory mask, gloves and lab coat with preferably a suit covering the body, must be worn to ensure that fungi harmful to humans does not come into contact with the skin or is inhaled. Depending on the scale of contamination various measures should be taken, from cleaning infected areas with 70% ethyl alcohol to using fume extraction fitted with a HEPA filter.³⁴

³² Caneva et al., 2009, 115-116.

³³ Florian, 2002, 7-11.

³⁴ Guild & MacDonald, 2004, 21-25.

Fungicides

Preventol® ON-S

One of the fungicides under investigation is a commercial product, Preventol[®] ON-S, that contains minimum 94% sodium o-phenyl phenol (Na-OPP) and 1-2% sodium hydroxide.³⁵ The chemical structure is shown below in Fig. 9. The product is a finely ground powder which also contains amounts of mineral oil, and has a broad spectrum of activity against bacteria, fungi, yeasts and lipophile viruses. It is soluble in water and has a high pH of 11.5 (1% in deionised water). If stored correctly in the original sealed container, it should have a shelf life of 18 months but it may discolour when exposed to air, light, moisture or heat, which has no adverse effect on the quality. Preventol[®] ON extra contains the same active ingredients but comes in flake form which may be too large for limited applications.³⁶

Figure 9: Chemical Structure of sodium o-phenyl phenol³⁷

Commercial Application

Lanxess, the current supplier of Preventol[®] ON-S, advertises the biocide for preservation and finishing in the glue and adhesive, paper and textile industries. It is also used for the manufacture of powdery medical instrument disinfectants and for the manufacture of powdery wash and cleaning agents with antimicrobial properties. It is possible to mix the product directly into other powders to dilute it.³⁸

³⁵ MSDS Preventol ON-S, Lanxess, (2011). Provided by suppliers via email, 3/05/2012, 2.

³⁶ Lanxess, *Product Information: Preventol*[®] ON-S, (2012), 2-5.

³⁷ Preventol[®] ON-S, ChemExper, www.chemexper.com/chemicals/supplier/cas/132-27-4.html. Accessed 25/06/2012.

³⁸ Lanxess, 2012, 2-3.

Use in Textile Conservation

A 1% solution in deionised water was chosen based on the manufacturers' information as well as previous use in textile conservation studies.^{39,40} It acts by rendering the fungi dormant, which ensures that fungal growth will not continue if the objects are then kept in a sufficiently low RH.⁴¹ This means that with a large mould outbreak textiles could be treated with the fungicide and then wet cleaning or vacuum suction used later to remove the dormant structures. However, Na-OPP can catalyse degradation reactions such as oxidation leading to changes in colour and brightness. After treatment with 1% Na-OPP textile samples were found to be more yellow after ageing than non-treated samples.^{42,43}

Toxicity and Health and Safety Precautions

In terms of toxicity 1% Na-OPP has been found to contain 2500 LD_{50} oral (mg/kg rat) compared with 1900 LD_{50} oral (mg/kg rat).⁴⁴ The MSDS for Preventol[®] ON-S states the following hazards listed in Fig. 10, which require considerable care to be taken when using this product.

| R22 | Harmful if swallowed |
|-----------|--|
| R34 | Causes burns |
| R37 | Irritating to respiratory system |
| R50 | Very toxic to aquatic organisms |
| S26 | In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. |
| S36/37/39 | Wear suitable protective clothing, gloves and eye/face protection |
| S45 | In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible) |
| S60 | The material and its container must be disposed of as hazardous waste |
| S61 | Avoid release to the environment. Refer to special instructions/ safety data sheet. |

Figure 10: Hazards Identification for Preventol® ON-S⁴⁵

³⁹ O. Abdel-Kareem, 'Evaluating the Combined Efficacy of Polymers with Fungicides for Protection of Museum Textiles against Fungal Deterioration in Egypt', *Polish Journal of Microbiology*, Vol. 59, no. 4, (2010), pp. 271-280.

⁴⁰ M. Nugari and G. Priori, 'Fungicides for Use on Textile Employed during the Restoration of Works of Art', *International Biodeterioration*, Vol. 23, (1987), 295-306.

⁴¹ S. Bertalan (ed.), 1994, 17.

⁴² Abdel-Kareem, 2005, 121-130.

⁴³ Tímár-Balázsy & Eastop, 2007, 299.

⁴⁴ MSDS Organic Tea Tree Essential Oil, www.nhrorganicoils.com/.../

²⁰¹¹⁰²⁰⁸¹⁷⁵⁴³⁰e_tea_tree_msds.pdf. Accessed 11/05/2012.

⁴⁵ MSDS Preventol ON-S, Lanxess, (2011). Provided by suppliers via email, 3/05/2012, 1-2.

These hazards and the toxicity of the fungicide require the use of PPE involving a lab coat, gloves, goggles and face mask, and to carry out tests under adequate fume extraction such as a fume cupboard. This should avoid personal exposure but the risks associated with this product are acute, meaning that large-scale and frequent use would not be straightforward or personally comfortable to perform. This is one of the disadvantages of Na-OPP and why an alternative would be beneficial.

Tea tree oil

Tea Tree oil (hereafter referred to as TTO) has been traditionally used in Australia by Aborigines to treat skin infections by crushing the leaves. It was then commercially cultivated in Australia with the first publications promoting its benefits originating in the 1920s and sales of the oil beginning in Europe in the 1930s.⁴⁶ It is a colourless to pale yellow essential oil with a spicy smell.⁴⁷ The plant itself is shown in Fig. 11. It is obtained by, "steam distillation of the foliage and terminal branchlets of Melaleuca alternifolia, Melaleuca linarifolia and Melaleuca dissitiflora as well as other species of Melaleuca provided that the oil obtained conforms to the requirements given in the International Standard (ISO 4730-2004)."⁴⁸ The need for testing has been identified by textile researchers interested in commercial textile finishing who state, "its novel medicinal activity is yet to be explored on textile substrates".⁴⁹ Its anti-inflammatory and antimicrobial properties are supported by a growing body of clinical data. However, its further use as a fungicide is particularly interesting as fungal infestations are a common problem encountered in historic textiles.



Figure 11: Tea Tree Plant

⁴⁶ Australian Tea Tree Oil Association, http://www.attia.asn.au/teatree_about.php. Accessed 17/04/2012.

⁴⁷ MSDS Tea Tree Oil (Melaleuca Alternifolia) Australian, http://www.sciencelab.com. Accessed 16/04/2012.

⁴⁸European Commission SCCP (Scientific Committee on Consumer Products), *Opinion on Tea Tree Oil, 16 December 2008*, ec.europa.eu/health/ph_risk/committees/04_sccp/.../sccp_o_160.pdf. Accessed 07/04/2012, 6.

⁴⁹ M. Joshi et. al, 'Ecofriendly antimicrobial finishing of textiles using bioactive agents based on natural products', *Indian Journal of Fibre and Textile Research*, Vol. 34, (September 2009), 295-304.

Over 100 constituents of the oil were found in 1986 through Gas Chromatography and Gas Chromatography Mass Spectrometry and the main constituents are shown in Fig. 12 below, with Terpinen-4-ol (Fig. 13) being the most concentrated. They are mostly monoterpenes, sequiterpenes and their related alcohols, and the designated maximum and minimum percentages are those stated in the ISO 4730-2004. The opinion of the European Commission Scientific Committee on Consumer Products (SCCP) stated that the composition of the essential oil is altered particularly with exposure to light, higher temperatures and levels of oxygen. This leads to an increase in p-cymene and oxidation reactions cause the formation of peroxides, endoperoxides and epoxides.⁵⁰ These cause the bleaching of textiles, which would lead to the weakening and yellowing of cellulose treated with these substances. It has been found that after as short a period of 21 months p-cymene can increase to 20-40%.⁵¹ It has also been found that it can react with polystyrene,⁵² and has an extremely low flash point of 57°C. Therefore it must be noted that the oil should be kept in a sealed non-plastic container and in cool, dark conditions to avoid oxidation, which should be prevented for 2-3 years.⁵³

| Constituent | Minimum (%) | Maximum (%) |
|-----------------------------|-------------|-------------|
| Terpinolene | 1.5 | 5 |
| 1,8-Cineole (eucalyptol) | Trace | 15 |
| α-Terpinene | 5 | 13 |
| y-Terpinene | 10 | 28 |
| p-Cymene | 0.5 | 8 |
| Terpinen-4-ol | 30 | 48 |
| α-Terpineol | 1.5 | 8 |
| Limonene | 0.5 | 1.5 |
| Sabinene | Trace | 3.5 |
| Aromadendrene | Trace | 3 |
| δ-Cadinene | Trace | 3 |
| Globulol | Trace | 1 |
| Viridiflorol | Trace | 1 |
| α-Pinene | 1 | 6 |
| Ledene (syn. viridiflorene) | Trace | 3 |

Figure 12: Main Constituents of TTO (from ISO 4730-2004)⁵⁴

⁵⁰ European Commission SCCP. Accessed 07/04/2012, 6.

⁵¹ J. J. Brophy et al., 'Gas Chromatographic Quality Control for Oil of *Melaleuca* Terpinen-4-ol Type (Australian Tea Tree), *Journal of Agricultural and Food Chemistry*, Vol. 37, (1989), 1332.

⁵² K.A. Hammer et al., 'Antifungal Activity of the Components of *Melaleuca alternifolia* (tea tree) oil', *Journal of Applied Microbiology*, Vol. 95, (2003), 853-860.

⁵³ European Commission SCCP. Accessed 07/04/2012, 12.

⁵⁴ European Commission SCCP. Accessed 07/04/2012, 6-7.

Fungicidal Properties of TTO

TTO acts by rupturing the cell wall of fungi, but has varying effects depending on the stage of growth. The potential as a fungicide has been tested in relation to its use in cosmetics and medicinal treatments, with studies carried out in vitro, on laboratory organisms and human test subjects. A comprehensive analysis of the efficacy against 24 different species of fungi, some of which are prevalent on cellulosic textiles, such as Aspergillus and Penicillium was compiled by Carson et. al in 2006. These studies show that minimum fungicidal concentrations (MFC) of TTO vary between the species from 0.12-2%, excepting Aspergillus niger which has had an MFC of 8%. It has also been noted that the unbroken cell wall of the conidium requires a higher concentration of fungicide whereas germinated conidia are more susceptible. Inhibition of the formation of germination tubes and hyphal colonies has been shown but further clinical studies are necessary to show the safety and efficacy in treating skin conditions, which could affect the H&S precautions taken by textile conservators to avoid contact with the skin.⁵⁶ It would be noted, therefore, that although there are strong indications that it could act as a useful fungicide, further tests on textiles following this study would be required.

The antimicrobial and fungicidal properties of TTO have been tested by textile manufacturers and there is now greater interest with the increasing resistance of microbes to antibiotics. This has promoted the testing of alternatives to antibiotics including the antifungal properties of TTO, which could also inform textile conservators. There are some known drawbacks to using TTO on textiles, such as the smell and danger of oxidation.

 ⁵⁵ Terpinen-4-ol, Wikipedia, http://en.wikipedia.org/wiki/Terpinen-4-ol, Accessed 25/06/2012.
 ⁵⁶ C. F. Carson et al., '*Melaleuca alternifolia* (Tea Tree) Oil: A Review of Antimicrobial and Other Medicinal Properties', *Clinical Microbial Reviews*, Vol. 19, no. 1, (2006), 54-56.

New Technologies

New technologies have created microcapsules containing a non-ionic product to provide dispersion and yeast microcapsules to minimise evaporation.^{57,58} Microencapsulation of TTO in cyclodextrin has also been formulated, which has the additional benefits of minimising evaporation, preventing oxidation and the pervasive smell.⁵⁹ These innovations could be used in the future to extend the usefulness of the oil in the treatment of historic textiles. Instead of a timed application of TTO to textiles, the microcapsules could be applied to an object for long-term anti-fungal properties. This may be useful for an object in an environment vulnerable to mould outbreak, such as a private stately home or a country with high humidity, where ideal environmental controls are not always sustainable. However, prior to the introduction and application of the microcapsules, the effect of TTO on the condition of textiles must be ascertained.

Application to Conservation

TTO has been used in conservation practice by the Sydney Technological Museum, now the Museum of Applied Arts and Sciences in Australia. A product called Bactigas[®] was used through an aerial spray to control a large outbreak of mould in an object store initially consisting of Bactigas[®] (0.3% tea tree oil, 97% liquid carbon dioxide and 2.7% ethanol).⁶⁰ A Bactigas[®] concentrate (10% tea tree oil in ethanol) was trialled by Bioassay which showed a microbial reduction of 10³ over 7 days. A further test found that Bactigas[®] (3% concentrate and 97% liquid carbon dioxide) had greater microbial reduction, as the dilute concentrate is more miscible and better absorbed. Florian's directions on the Aseptic Technique were then followed to remove the dead mould,⁶¹ which remains harmful to humans due to the antigenic and mycotoxic nature of the fungal structures.⁶² This case study formed an initial inquiry into the properties of tea tree oil in relation to the treatment of heritage objects.

⁵⁷ I. Holme, 'Innovative Technologies for High Performance Textiles', *Society of Dyers and Colourists, Colouration Technology*, (2007), 68.

⁵⁸ Microencapsulation Innovations, http://microencapsulationinnovations.com/index.html. Accessed 19/06/2012.

⁵⁹ Wacker Chemie, 'New Applications for the Aboriginal Remedy: Tea Tree Oil - Wrapped up in Sugar Molecules', No. 4, (April 2009), *www.wacker.com/cms/media/en/documents/...pdf/ Teebaumoel.pdf.* Accessed, 1-10.

⁶⁰ S. Gatenby & P. Townley, 'Preliminary Research into the Use of the Essential Oil of *Melaleuca Alternifolia* (Tea Tree Oil) in Museum Conditions', *AICCM Bulletin*, (2003), 67-70.

⁶¹ M-L. Florian, 'Aseptic Technique: A Goal To Strive For in Collection Recovery of Moldly Archival Materials and Artefacts', *Journal of the American Institute for Conservation*, Vol. 39, (2000), 107-115.

⁶² S. Gatenby & P. Townley, 'Preliminary Research into the Use of the Essential Oil of *Melaleuca alternifolia* (tea tree oil) in Museum Conservation', *The 5th International Conference on Biodeterioration of Cultural Property (ICBCP-5): 12-14 November 2001, Sydney ,Preprints, lucite.org/lucite/archive/art_-_painting/icbcp5_papers.pdf*. Accessed 31/07/2012, 137-138.

CHAPTER THREE

Fungicide Experiments

Experimental Design Summary

The Cotton and Utility textile samples have been treated using the 2 fungicides: TTO and Preventol[®] ON-S to test the residual effect of the treatments on the fabric samples.

Two treatment methods have been used:

- 1. Immersion for 30 minutes and a rinsing in softened and deionised water soak.
- 2. Spot cleaning using a vacuum suction table for 10 minutes followed by rinsing with deionised water.

It was intended that each test would use three samples of each fabric, as shown in Fig. 14. However, there was some variation due to the need to vary the treatment method. This is explained in the more detailed examination of each test following this summary.

| Fungicide | Treatment Method (abbreviation) | Cotton Sample Numbers | Utility Sample Numbers | |
|-----------------------------|------------------------------------|--------------------------|------------------------|--|
| Preventol [®] ON-S | Immersion | 3 3 | | |
| Fleventor On-S | Spot cleaning | 3 | 3 | |
| тто | Immersion | 3 | 3 | |
| | Spot cleaning | 3 | 3 | |

| Figure 14 | 4: Table | of Test | Samples |
|-----------|----------|---------|---------|
|-----------|----------|---------|---------|

Each sample then underwent condition testing with three readings taken to allow averages:

- 1. Colourimeter
- 2. Fourier Transform Infrared Spectroscopy (FTIR)
- 3. Surface pH
- 4. Tensile Strength

Selection and Preparation of Textile Samples

Fabric One: New Cotton

A new cotton lawn fabric will be used as it is lightweight and has a plain weave, and a high S-twist thread count of 40/cm, with a semi-transparent appearance. This will allow close visual examination aided by the appearance of the fabric and will contrast with Fabric Two. The white fabric has been scoured to remove any impurities remaining from the manufacturing process. This fabric will hereafter be referred to as 'Cotton'.

Fabric Two: British Utility Scheme cotton

During the Second World War rationing regulated the production and purchase of fabrics and a points plan was established under the British Utility Scheme (1941-1952).⁶³ The purpose was to limit the consumption of raw materials and standardise designs for domestic uses. The Utility mark CC41 (Fig. 15 below) is an abbreviation of the Civilian Clothing Act of 1941 and identifies the object as being produced within this period.⁶⁴ The use of this fabric for experimentation presents an example of a historic textile with a clearly defined period of production of 60 to 70 years ago. The fabric used has been identified through a microscopic study of the fibres, which showed the twisted characteristics of cotton. The object was kindly donated from a private collection and has been used previously for at least half of its lifetime as a bed sheet and dust cover. It is heavier in weight than the new fabric and has a low thread count of 25/cm in both warp and weft directions with a relatively uneven yarn thickness in S-twist. The fabric exhibits small holes throughout and several light brown stains. The samples were taken at random but avoiding the damage and staining. This fabric will hereafter be referred to as 'Utility'.



Figure 15: The Utility mark

⁶³ Imperial War Museums, http://www.iwm.org.uk/history/clothes-rationing-in-britain-during-the-second-world-war#. Accessed 8/4/2012.

⁶⁴ V. Margolin, 'Review', *Studies in the Decorative Arts*, Vol. 8, no. 2, (2001).

Size of Sample Fabrics

The size of the sample fabrics has been determined based on the requirements of the tensile strength testing. The BS ISO 13934-2:1999 states that the gauge length size should be 200mm (the distance between the tensile jaws) and 50mm wide. However, this would be too large for the purposes of this experiment so half the standard size was chosen, at 100x25mm for the purposes of the tensile testing. The gauge length requires extra to ensure the fabric is held firmly between the jaws, and to allow for any fraying or shrinking of the samples during treatment the samples were cut at 150x80mm (Figs. 16 and 17). This allowed up to 2.5mm to be cut and discarded from either width, and 3 strips could be tested with the tensile tester from each sample. The samples were taken from both the warp and weft directions to ensure accuracy of results.

Figure 16: Cotton Sample

Figure 16: Utility Sample

Immersion Test Method

The immersion tests will be carried out by placing the six fabric samples (3 Cotton, 3 Utility) into individual 250ml glass beakers and 50ml of the solution was added to each beaker. The samples were left in the solution for 30 minutes, which was thought to be the adequate treatment time required for the fungicide to penetrate the fibres and impact mould. The pH of the solution in each beaker was then tested using Fisherbrand pH indicator strips to determine any changes in acidity or alkalinity. The samples were then rinsed using a running rinse of softened water for 10 minutes and the pH was again tested using indicator strips to ensure a return to near neutral which would indicate the removal of the fungicide. A final 10 minute soak in deionised water was carried out to remove any remaining impurities left by the softened water as usual in textile conservation practice, with the pH again noted.

Spot Cleaning Test Method



Figure 18: Spot Cleaning

A vacuum suction table was used as this enables a small area of the sample to be treated in isolation in a controlled manner.⁶⁵ Melinex[®] was used to cover the size of the mesh to direct the vacuum. A rectangle was cut out of the centre to the size of 25x75mm and the placement of the sample was marked.

In textile conservation practice it is usual that the soiled area is placed downwards so that the soiling is drawn to the less visible side, and a piece of fabric is placed in between the vacuum and the object to draw the soiling away and protect the object from getting an imprint of the mesh. There was no soiling on the samples, so this was not considered a variable for the purposes of the tests, but a new piece of cotton lawn was placed over the vacuum to replicate normal practice.

⁶⁵ M. Ringgaard, 'The Use of the Vacuum or Suction Table in Textile Conservation', http:// independent.academia.edu/MajRinggaard/Papers/1090139/ THE_USE_OF_THE_VACUUM_OR_SUCTION_TABLE_IN_TEXTILE_CONSERVATION. Accessed 31/07/2012. The treatment time decided on was a 10 minute treatment followed by a 5 minute rinse with deionised water, with 1 drop from a glass pipette every 1-2 seconds. The treatment area would be covered systematically to allow full and even coverage. This is comparable to usual treatments undertaken using the vacuum suction table. A demonstration is shown in Fig. 18.

Preventol® ON-S Testing

The packaging of the product is seen in Fig. 19 and the supplier information for all of the chemicals used is listed in Appendix One. The 1% Preventol[®] ON-S solution was made up under extraction in a fume cupboard and wearing the PPE as dictated by the COSHH and Risk Assessment forms included in Appendix Three. The powder was weighed out using a microbalance and the deionised water measured in a graduated cylinder and both added to a volumetric flask at 1g/100ml. The mixture was then shaken in the stoppered flask for approximately 30 seconds to ensure complete dissolution and the pH was tested using indicator strips.





Preventol[®] ON-S Immersion

The samples were treated as described previously in the Immersion Test Method. The samples were placed in the individual beakers and 50ml of the solution was added to each. The samples were then left to soak for 30 minutes and the pH was measured. A 10 minute running rinse in softened tap water was completed and the pH again tested. The samples then received a final 10 minute soak in deionised water, with the pH measured again following this.

The pH was tested using pH indicator strips and the readings are shown below in Fig. 20:

| 1% | 1% Preventol in DI water | | | | | | |
|-----------------------|--------------------------------|----------|----------|----------|-----------|-----------|-----------|
| | | Cotton 1 | Cotton 2 | Cotton 3 | Utility 1 | Utility 2 | Utility 3 |
| pН | End of 30 min immersion | 11.5 | 12 | 11.5 | 11.5 | 11.5 | 11 |
| | End of 10 min rinse | 6.5 | 6.5 | 6.5 | 6.5 | 6.5 | 7 |
| | End of 10 min DI water soak | 6.5 | 6.5 | 6.5 | 6.5 | 6 | 6.5 |
| | | | | | | | |
| pH of tap water = 6.5 | | | | | | | |
| pH of DI water = 6.5 | | | | | | | |
| F | H of pH of 1% Preventol = 11.5 | | | | | | |

Figure 20: Preventol® ON-S Immersion Treatment pH

The fabric samples were then laid out to dry on absorbent blue paper. Although textile objects would usually be dried with blotting paper with the possible use of cool fans to speed up the process, this method would have been difficult to replicate for multiple samples. Laying out the samples negated introducing additional variables.

Preventol[®] ON-S Spot Cleaning

The 1% Preventol[®] ON-S solution was then used to commence the spot cleaning treatments using the method described previously.

Firstly a Cotton sample was set up for treatment. The drops of fungicide were dispensed from the pipette but after 3 minutes slight yellowing of the sample was noticed, which by 8 minutes had darkened to a noticeable brown tone and included tide marks of a darker brown. The rinsing allayed the tide marks slightly but there was a clear contrast in the white of the untreated area and the brown of the treated area, with a darker brown around the edges by the Melinex[®]. This test was repeated on another Cotton sample with the same results. The vacuum suction table was thoroughly cleaned and deionised water and different solvents (acetone and IMS) were used on a cotton sample with no browning effect, meaning that the table was not the cause. A scoured wool delaine was used without the cotton in between the vacuum mesh to ascertain whether the fungicide was reacting with the cotton. The same brown residue was again found on the wool so it was thought that the issue could have been caused by contamination of the fungicide.

A new solution of 1% Preventol[®] ON-S was made up and two more samples were treated, with the second at half the treatment time (5 minute treatment, 2.5 minute rinse). There was no improvement in the results and the samples after treatment can be seen in Fig. 21.

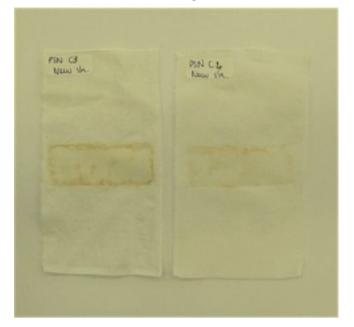


Figure 21: Samples After Preventol[®] ON-S Spot Cleaning (left= 10 minute treatment; right= 5 minute treatment)

Therefore, contamination of the vacuum suction table, contamination of the fungicide, and contamination of the cotton fabric samples were all ruled out as causative factors. Explanations were then considered including that the use of the vacuum catalysed oxidation reactions, and that the browning of the solution was advanced with the high levels of suction. The Preventol[®] ON-S solutions were then left for two weeks and it was noticed that they turned brown in this time (Fig. 22) and it was concluded that the solutions had oxidised. This was mentioned in the supplier's literature with the statement that it did not have a detrimental effect on performance. However, brown staining of the samples was having a detrimental effect. New solutions should therefore be made up on the day of treatment and the vacuum suction table was discounted as a valid tool. This may be a significant observation and prevent the browning of historic textile objects in the future.



Figure 22: Preventol[®] ON-S solutions made on 18/5/2012 and 12/6/2012

TTO Testing

The TTO was sourced from a company which guaranteed that the product was certified Organic by the Soil Association which should ensure it met strict, ethical production and processing criteria under the EEC Regulation No. 2092/91.⁶⁶ The oil was dispatched in a tin container with a sealed plastic lid, preventing exposure to light that could cause oxidation (Fig. 23).



Figure 23: TTO Product Packaging

TTO Immersion with Detergent

The primary issue with using the TTO was how to enable the oil to mix with water. A 5% solution was required which should have ensured that the required 0.12-2% MFC was in contact with the textile. Because there were no formulas available the first experiment used the non-ionic detergent Dehypon[®] LS45 as an emulsifier. The process was initiated by adding drops of the detergent to the 5% TTO in deionised water. The final solution necessitated a 30 x critical micelle concentration (c.m.c.) for most of the oil to mix with the water. This was an extremely concentrated level of detergent and the immersion test was carried out to determine the effect on the textile despite the knowledge that a more workable solution should be sought following this test. Due to the amount of detergent it involved a greatly extended rinsing period to remove the detergent from the samples. There was a strong TTO smell remaining on the samples which took several days to start evaporating. This indicates that there was TTO remaining on the textile.

⁶⁶ NHR Organics, http://www.nhrorganicoils.com/frame.php?page=info_20. Accessed 17/07/2012.

The pH readings for the tests were taken using pH indicator strips and are shown in the table of Fig. 24:

| 5% | TTO in DI water with 30 x c.m.c. D | Dehypon LS | 645 | | | | |
|----|------------------------------------|------------|----------|----------|-----------|-----------|-----------|
| | | Cotton 1 | Cotton 2 | Cotton 3 | Utility 1 | Utility 2 | Utility 3 |
| pН | End of 30 min immersion | 5.5 | 5.5 | 5.5 | 5.5 | 6 | 6 |
| | End of 45 min rinse | 6.5 | 6.5 | 6.5 | 6.5 | 6.5 | 6.5 |
| | End of 10 min DI water soak | 6.5 | 6.5 | 6.5 | 6.5 | 6.5 | 6.5 |
| | | | | | | | |
| | pH of tap water $= 6.5$ | | | | | | |
| | pH of DI water = 6.5 | | | | | | |
| | pH of TTO solution = 5.5 | | | | | | |

Figure 24: TTO and Detergent Immersion Treatment pH

Choice of Emulsifier

Research was carried out to find a suitable emulsifier. It needed to be easily removed with rinsing and have a neutral pH so that it would not harm the textile. It should also not produce any thickening of the solution, which is the effect of many emulsifiers. Emulsifiers are not currently used in conservation, so an alternative was sought from either the food or skincare industries. It was hoped that a natural substance could be found that was in keeping with the benefits of the TTO and not harmful to humans. Carrageenan, a sulphated polysaccharide extracted from red seaweed, is used in the food industry but was discounted due to its tendency to thicken solutions. Other emulsifiers are used in skincare products also have a thickening effect or have an unknown mixture of ingredients, some of which may be be negative when using on textiles.

The emulsifier chosen was Steareth-21, trade name Eumulgin S 21, which is a polyethoxylated fatty alcohol combined with a small percentage of detergent. It is a nonionic emulsifier derived from Stearic acid and the preference of non-ionic surfactants for slightly acidic conditions should make it effective for use with historic cellulosic textiles which are commonly acidic as well as the TTO. It also has greater surface activity in comparison with anionic surfactants due to the larger hydrophilic head, and therefore makes it a better wetting and emulsifying agent.

It can be bought as a food grade as well as a cosmetic grade product and therefore should be safe for use with textiles. It has no known toxicological dangers and has a pH of 6-7.5. The product was purchased in the form of waxy pellets and the product directions suggested using it at 2-6%.

Using the Emulsifier

To establish the concentration of Steareth-21 necessary for use with the 5% TTO, a 2% emulsifier was made initially. The pellets were weighed out and added to a bain-marie containing deionised water. The mixture was heated slowly on a Hotplate and magnetic stirrers were added at medium speed to preclude the need for constant stirring, as illustrated in Fig. 25. The temperature was raised to 45°C over 10 to 15 minutes. The mixture formed bubbles while the wax was melting before turning clear. The 2% emulsifier was then combined with the oil but small globules of oil were suspended in the solution, suggesting that a higher percentage of emulsifier was necessary. A 4% solution was made up which did effectively disperse the oil in the solution. When adding the TTO care was taken that the temperature was at 40°C and well below the flash point of the oil at 57°C.



Figure 25: Emulsifier on Hotplate

TTO Immersion with Emulsifier

Following the cooling of the solution the immersion test was carried out in the same manner used previously. The six samples were placed into separate beakers and 50ml of the solution added and left for 30 minutes. The pH readings were taken as before and are shown in the Fig. 26 below. There was a less noticeable TTO smell than with the previous Immersion test, and evaporated in a shorter period of 2 days, showing a smaller amount of TTO remaining.

| 5% | TTO in 4% emulsifier in DI water | | | | | | |
|----|----------------------------------|----------|----------|----------|-----------|-----------|-----------|
| | | Cotton 1 | Cotton 2 | Cotton 3 | Utility 1 | Utility 2 | Utility 3 |
| pН | End of 30 min immersion | 6 | 6 | 6.5 | 6 | 6 | 6 |
| | End of 20 min rinse | 7 | 6.5 | 6.5 | 6.5 | 6.5 | 6.5 |
| | End of 10 min DI water soak | 6.5 | 6.5 | 6.5 | 6.5 | 6.5 | 6.5 |
| | | | | | | | |
| | pH of tap water $= 6.5$ | | | | | | |
| | pH of DI water = 6.5 | | | | | | |
| | pH of TTO solution = 5.5 | | | | | | |

TTO with Emulsifier Spot Cleaning

Following the completion of the immersion tests, a new batch of the solution was tested using the vacuum suction table. A Melinex[®] sheet with the same sized rectangle was used and the 10 minute treatment, 5 minute rinse was to be repeated using the new fungicide. The first treatments were carried out on two Cotton samples but on examination of the samples after treatment, it was noticed that a white waxy deposit had built up on particularly the lower surface of the textile. It was also observed that detergent had gathered in between the vacuum mesh and Melinex[®], suggesting that the emulsifier had not been taken through the mesh with the suction, but had been collected and remained on the surface.

A new solution was made up to discount this variable. The third Cotton sample was treated at half the treatment times- 5 minute treatment, 2.5 minute rinse. There was a slight loss in flexibility indicating a much smaller amount of waxy build up. Any shorter treatment time than 5 minutes was considered too short to be effective in allowing the fungicide to act, so alternative treatment methods were sought.

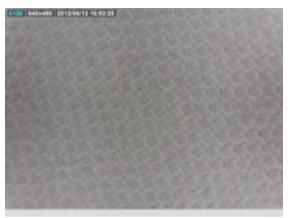
The fourth Cotton sample was treated for 10 minutes but with periodic drying intervals. A 2 minute drying phase every 2.5 minutes was used, as observations had shown the sample should be completely dry in 2 minutes. It was thought this may have allowed the emulsifier/detergent to be taken away by the vacuum. This was repeated up to the 10 minute treatment time, with the 5 minute rinse also broken up with a 2 minute drying period. There were some deposits but less than the initial test which suggested that the regular drying of the samples was of some benefit.

A fifth Cotton sample was then treated for 10 minutes with a 1 minute drying period every 2.5 minutes, which allowed slightly more build up than the previous method. It was observed that the receptacle used to hold the fungicide, a small conical flask, had a portion of scum floating at the meniscus level. This had previously been thought to be incomplete mixing of the solution after being left for over an hour, and the flask had been thoroughly shaken prior to use. However, this could in fact have been excess emulsifier, so subsequent tests were carried out placing the pipette below this scum so it could be avoided.

A sixth Cotton sample was then tried avoiding this scum, using half the treatment time at 5 minutes, with a 2 minute rinse. This still produced a waxy residue. Two Utility samples were then tested to ascertain whether the weave structure of the new cotton was the cause. The first Utility sample was treated for 10 minutes with a 2 minute drying period, and the second Utility sample was treated for 5 minutes with a 2 minute drying period.

The fabric samples were analysed using a Dino-Lite Digital Microscope. The photographs taken are shown in Figs. 27 and 28. The difference between the untreated and the sample treated first can clearly be seen with the weave structure obscured by the residues.

Figure 27: Untreated Cotton Sample





It was then concluded that the levels of emulsifier necessary to allow complete dispersion of the oil were too concentrated for use with the vacuum suction table.

CHAPTER FOUR

Condition Testing After Fungicidal Treatments

Condition Test 1: Colourimeter

Theory and Purpose of Test

To accurately measure the colour as perceived by the human eye, a colourimeter can be used. This is based on the fact that colours absorb and reflect the different wavelengths of light, and this can be quantified and calculated using a colourimeter. The physical occurrence of colour is represented as X, Y, and Z values, but CIE L*a*b* can more accurately represent the visible colour space. The L denotes lightness and darkness, with 0 being black and 100 being white. The 'a' denotes the red-green axis with positive values being red. The 'b' denotes the blue-yellow, with positive values being yellow. Zero is neutral for both axes.⁶⁷ From these values a three-dimensional rectangular colour space can be used to plot the Lab colours.

The colourimeter functions by using a pulsed xenon arc lamp inside a mixing chamber at a 0° viewing angle which produces diffuse, even lighting. This model of colourimeter uses a wide area illumination so that the 50mm diameter measuring area is evenly illuminated. The light reflected perpendicular to the specimen surface is collected by the optical fibre cable to obtain the colour measurement. The diagram in Fig. 29 shows the various parts of the instrument.

Figure 29: Colourimeter Measuring Head⁶⁸

⁶⁷ Hunter-Lab, 'Color Theory', http://www.hunterlab.com/pdf/color.pdf. Accessed 19/8/2012.

⁶⁸ Minolta, 'Chroma Meter Manual', www.konicaminolta.com/content/download/4728/34959/CR. Accessed 19/8/2012, 68.

Method of Test

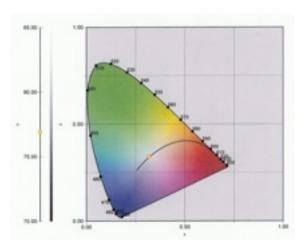
The instrument used was a Minolta Chroma Meter CR-210 (Fig. 30). It was firstly calibrated by using a white tile calibration plate and data is inputted to match the target colour. The colour space used, as described previously was L*a*b* because it most accurately measures the colours seen by normal human vision. Three measurements were taken for each sample to allow mean and standard deviations (SD) to be more precise. The data was then printed and inputted onto spreadsheets. For ease of interpretation the following test results have been transmitted into two-dimensional Yxy colour space graphs which show colour without the need to decipher a three-dimensional form. The software used to produce the graphs was the Konica Minolta Color Data Software SpectraMagic NX.



Figure 30: Minolta Chroma Meter CR-210 with Textile Sample

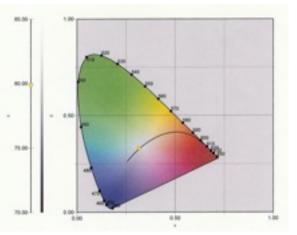
Control

| | с | otton | 1 | с | otton | 2 | с | otton | 3 | ι | Jtility | 1 | ι | Jtility | 2 | ι | Jtility | 3 |
|----------|-------|-------|-------|-------|-------|-------|-------|-------|-------|--------------|---------|-------|-------|---------|-------|-------|---------|-------|
| | L | а | b | L | a | b | L | а | b | L | a | b | L | а | b | L | a | b |
| 1 | 91.59 | +0.76 | +0.23 | 91.66 | +0.65 | +0.48 | 91.64 | +0.70 | +0.41 | 90.14 | +0.87 | +0.91 | 90.39 | +0.74 | +0.92 | 90.32 | +0.51 | +1.44 |
| 2 | 91.60 | +0.76 | +0.25 | 91.64 | +0.67 | +0.46 | 91.66 | +0.71 | +0.42 | 91.13 | +0.85 | +0.90 | 90.36 | +0.76 | +0.93 | 90.35 | +0.52 | +1.43 |
| 3 | 91.61 | +0.75 | +0.25 | 91.64 | +0.72 | +0.46 | 91.66 | +0.70 | +0.41 | 90.12 | +0.82 | +0.92 | 90.35 | +0.82 | +0.92 | 09.34 | +0.56 | +1.44 |
| Max | 91.61 | +0.76 | +0.25 | 91.66 | +0.72 | +0.46 | 91.66 | +0.71 | +0.42 | 90.14 | +0.87 | +0.92 | 90.39 | +0.82 | +0.93 | 90.35 | +0.56 | +1.44 |
| Min | 91.60 | +0.75 | +0.23 | 91.64 | +0.65 | +0.46 | 91.64 | +0.70 | +0.41 | 90.12 | +0.82 | +0.90 | 90.35 | +0.74 | +0.92 | 90.32 | +0.51 | +1.43 |
| Mea n | 91.60 | +0.76 | +0.24 | 91.65 | +0.68 | +0.47 | 91.65 | +0.70 | +0.41 | 90.13 | +0.85 | +0.91 | 90.37 | +0.77 | +0.92 | 90.34 | +0.53 | +1.44 |
| SD | 0.00 | 0.01 | 0.01 | 0.01 | 0.03 | 0.01 | 0.01 | 0.01 | 0.00 | 0.01 | 0.02 | 0.01 | 0.02 | 0.04 | 0.00 | 0.01 | 0.02 | 0.01 |
| | | | | | | | | | | | | | | | | | | |
| Mea | an Co | tton | | | 6 | a | ł | D | | Mean Utility | I | - | i | a | l | 0 | | |
| | | | 91 | .63 | +0 | .71 | +0 | .37 | | | 90 | .28 | +0 | .72 | +1 | .09 | | |



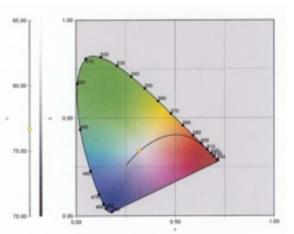
Control Cotton Mean

Control Utility Mean



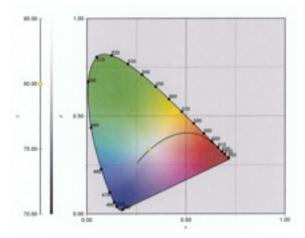
Preventol[®] ON-S Immersion

| | C | Cotton 1 | | с | otton | 2 | с | otton | 3 | U | ltility | 1 | u | Itility | 2 | ι | Jtility | 3 |
|------|-------------|----------|-------|-------|-------|-------|-------|-------|-------|--------|---------|-------|-------|---------|-------|-------|---------|-------|
| | L | а | b | L | а | b | L | а | b | L | а | b | L | а | b | L | а | b |
| 1 | 91.64 | +0.79 | +0.20 | 91.75 | +0.74 | +0.16 | 91.68 | +0.75 | +0.22 | 90.31 | +0.71 | +1.75 | 89.89 | +0.56 | +2.28 | 90.39 | +0.98 | +0.6 |
| 2 | 91.64 | +0.76 | +0.20 | 91.72 | +0.83 | +0.14 | 91.67 | +0.76 | +0.22 | 90.30 | +0.67 | +1.79 | 89.89 | +0.59 | +2.27 | 90.39 | +1.00 | +0.68 |
| 3 | 91.66 | +0.76 | +0.19 | 91.72 | +0.77 | +0.16 | 91.67 | +0.73 | +0.23 | 90.30 | +0.67 | +1.78 | 89.92 | +0.59 | +2.28 | 90.34 | +0.97 | +0.7 |
| Мах | 91.66 | +0.79 | +0.20 | 91.75 | +0.83 | +0.16 | 91.68 | +0.76 | +0.23 | 90.31 | +0.71 | +1.79 | 89.92 | +0.59 | +2.28 | 90.39 | +1.00 | +0.7 |
| Min | 91.64 | +0.76 | +0.19 | 91.72 | +0.74 | +0.14 | 91.67 | +0.73 | +0.22 | 90.30 | +0.67 | +1.78 | 89.89 | +0.56 | +2.27 | 90.34 | +0.97 | +0.67 |
| Mean | 91.65 | +0.77 | +0.20 | 91.73 | +0.78 | +0.15 | 91.67 | +0.75 | +0.22 | 90.30 | +0.68 | +1.77 | 89.90 | +0.58 | +2.28 | 90.37 | +0.98 | +0.6 |
| SD | 0.01 | 0.01 | 0.01 | 0.01 | 0.04 | 0.01 | 0.01 | 0.02 | 0.01 | 0.01 | 0.02 | 0.02 | 0.01 | 0.01 | 0.01 | 0.02 | 0.01 | 0.02 |
| | | | | | 1 | | | | 1 | | | | | | | | | |
| Mea | Mean Cotton | | L | á | a | | 0 | | Me | an Uti | ilitv | l | - | | a | ł | 0 | |
| inou | | 91 | .68 | +0 | .77 | +0 | .19 | | | | | 90 | .19 | +0 | .75 | +1 | .58 | |



Mean Utility

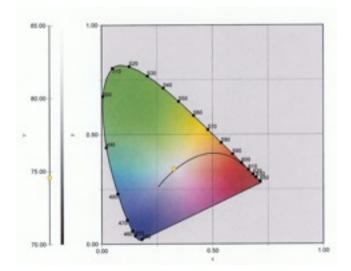
Mean Cotton



Preventol[®] ON-S Spot Cleaning

| | С | otton | 1 | ι | Jnder | 1 | c | otton | 2 | ι | Inder | 2 | С | otton | 3 | с | otton | 4 |
|------|--------|-------|-------|-------|-------|--------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| | L | а | b | L | а | b | L | a | b | L | a | b | L | а | b | L | а | b |
| 1 | 89.81 | +0.28 | +4.30 | 87.93 | +0.07 | +8.59 | 89.90 | +0.35 | +4.45 | 90.20 | +0.51 | +3.20 | 88.99 | +0.61 | +5.35 | 90.25 | +0.42 | +3.70 |
| 2 | 89.17 | +0.20 | +5.60 | 86.30 | +0.11 | +11.86 | 89.67 | +0.26 | +5.15 | 90.30 | +0.50 | +3.15 | 88.40 | +0.67 | +6.07 | 89.65 | +0.28 | +4.85 |
| 3 | 88.93 | +0.12 | +6.28 | 86.85 | +0.04 | +10.90 | 89.14 | +0.30 | +6.26 | 90.51 | +0.59 | +2.50 | 88.77 | +0.63 | +5.51 | 90.06 | +0.26 | +4.37 |
| Max | 89.81 | +0.28 | +6.28 | 87.93 | +0.11 | +11.86 | 89.90 | +0.35 | +6.26 | 90.51 | +0.59 | +3.20 | 88.99 | +0.67 | +6.07 | 90.25 | +0.42 | +4.85 |
| Min | 88.93 | +0.12 | +4.30 | 87.30 | +0.04 | +8.59 | 89.14 | +0.26 | +4.45 | 90.20 | +0.50 | +2.50 | 88.40 | +0.61 | +5.35 | 89.65 | +0.26 | +3.70 |
| Mean | 89.30 | +0.20 | +5.39 | 87.03 | +0.07 | +10.45 | 89.57 | +0.30 | +5.29 | 90.34 | +0.53 | +2.95 | 88.72 | +0.64 | +5.64 | 89.99 | +0.32 | +4.31 |
| SD | 0.45 | 0.08 | 1.00 | 0.82 | 0.03 | 1.68 | 0.38 | 0.04 | 0.91 | 0.15 | 0.04 | 0.39 | 0.29 | 0.03 | 0.37 | 0.03 | 0.08 | 0.57 |
| | | | | | | | | | | | | | | | | | | |
| Mea | an Col | tton | l | _ | | a | | b | | | | | | | | | | |
| | | | 89 | .20 | +0 | .34 | +5 | .67 | | | | | | | | | | |

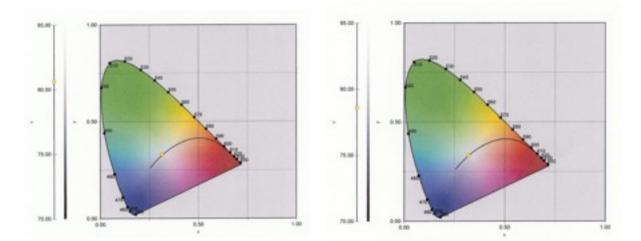
Mean Cotton



TTO with Detergent Immersion

| | с | otton | 1 | с | otton | 2 | с | otton | 3 | L | Jtility | 1 | ι | Jtility | 2 | ι | Jtility | 3 |
|------|-------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|---------|-------|-------|---------|-------|-------|---------|--------|
| | L | а | b | L | а | b | L | а | b | L | а | b | L | a | b | L | a | b |
| 1 | 91.81 | +0.86 | -0.28 | 92.04 | +0.77 | -0.15 | 92.03 | +0.87 | -0.24 | 90.65 | +0.64 | +1.30 | 90.75 | +0.54 | +1.52 | 90.74 | +0.65 | +0.72 |
| 2 | 91.88 | +0.84 | -0.29 | 92.03 | +0.78 | -0.12 | 92.01 | +0.80 | -0.21 | 90.69 | +0.63 | +0.29 | 90.77 | +0.52 | +1.48 | 90.69 | +0.70 |)+0.71 |
| 3 | 91.89 | +0.85 | -0.27 | 92.02 | +0.81 | -0.16 | 92.00 | +0.86 | -0.23 | 90.61 | +0.70 | +1.33 | 90.75 | +0.58 | +1.53 | 90.76 | +0.61 | +0.73 |
| Max | 91.89 | +0.86 | -0.27 | 92.04 | +0.81 | -0.12 | 92.03 | +0.87 | -0.21 | 90.69 | +0.70 | +1.33 | 90.77 | +0.58 | +1.53 | 90.76 | +0.70 |)+0.73 |
| Min | 91.81 | +0.84 | -0.29 | 92.02 | +0.77 | -0.16 | 92.00 | +0.80 | -0.24 | 90.61 | +0.63 | +1.29 | 90.75 | +0.52 | +1.48 | 90.69 | +0.61 | +0.71 |
| Mean | 91.86 | +0.85 | -0.28 | 92.03 | +0.79 | -0.14 | 92.01 | +0.84 | -0.23 | 90.65 | +0.66 | +1.31 | 90.76 | +0.55 | +1.51 | 90.73 | +0.65 | +0.72 |
| SD | 0.04 | 0.01 | 0.01 | 0.01 | 0.02 | 0.02 | 0.01 | 0.03 | 0.01 | 0.04 | 0.04 | 0.02 | 0.01 | 0.03 | 0.02 | 0.03 | 0.04 | 0.01 |
| | | | | | | | | | | | | | | | | | | |
| Mea | Mean Cotton | ton | l | - | á | a | ł | D | | Me | an Uti | litv | | _ | i | a | I | b |
| mea | | | 91 | .97 | +0. | .83 | -0. | 22 | | | | | 91 | .05 | +0 | .62 | +1 | .18 |

Mean Cotton



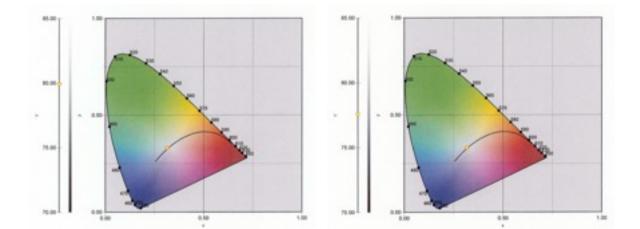
Mean Utility

TTO with Emulsifier Immersion

| | с | otton | 1 | с | otton | 2 | с | otton | 3 | ι | Jtility | 1 | ι | Jtility | 2 | ι | Jtility | 3 |
|------|-------|------------|-------|-------|-------|-------|-------|-------|-------|-------|---------|-------|-------|---------|-------|-------|---------|-------|
| | L | а | b | L | а | b | L | а | b | L | а | b | L | а | b | L | а | b |
| 1 | 92.02 | +0.69 | +0.01 | 91.97 | +0.71 | -0.12 | 91.81 | +0.07 | +0.27 | 90.64 | +1.07 | +0.05 | 90.62 | +0.77 | +0.88 | 90.46 | +0.55 | +1.75 |
| 2 | 92.04 | +0.68 | +0.05 | 92.00 | +0.70 | -0.07 | 91.87 | +0.07 | +0.24 | 90.69 | +1.04 | +0.21 | 90.54 | +0.83 | +0.74 | 90.55 | +0.65 | +1.47 |
| 3 | 92.05 | +0.71 | -0.01 | 91.94 | +0.76 | -0.08 | 91.90 | +0.69 | +0.24 | 90.61 | +1.03 | +0.20 | 90.66 | +0.90 | +0.37 | 90.53 | +0.62 | +1.64 |
| Max | 92.05 | +0.71 | +0.05 | 92.00 | +0.76 | -0.07 | 91.90 | +0.70 | +0.27 | 90.69 | +1.07 | +0.21 | 90.66 | +0.90 | +0.88 | 90.55 | +0.65 | +1.75 |
| Min | 92.02 | +0.68 | -0.01 | 91.94 | +0.70 | -0.12 | 91.81 | +0.69 | +0.24 | 90.61 | +1.03 | +0.05 | 90.54 | +0.77 | +0.37 | 90.46 | +0.55 | +1.47 |
| Mean | 92.04 | +0.69 | +0.02 | 91.97 | +0.72 | -0.09 | 91.86 | +0.70 | +0.25 | 90.65 | +1.05 | +0.15 | 90.61 | +0.83 | +0.66 | 90.51 | +0.61 | +1.62 |
| SD | 0.01 | 0.01 | 0.03 | 0.03 | 0.03 | 0.02 | 0.04 | 0.01 | 0.01 | 0.04 | 0.02 | 0.09 | 0.06 | 0.06 | 0.26 | 0.04 | 0.05 | 0.14 |
| | | | | | | | | | | | | | | | | | | |
| Mea | n Cot | n Cotton – | L | _ | á | 3 | ł | 0 | | Me | an Uti | ilitv | | _ | | a | | 0 |
| | | | 91 | .96 | +0. | .70 | +0 | .06 | | | | | 90 | .59 | +0 | .83 | +0 | .81 |

Mean Utility

Mean Cotton



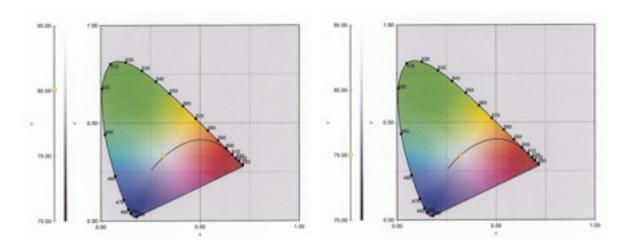
TTO with Emulsifier Spot Cleaning

| | | С | otton | 1 | С | otton | 2 | с | otton | 3 | С | otton | 4 | С | otton | 5 | С | otton | 6 |
|------|-----|-----|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| | | L | а | b | L | а | b | L | а | b | L | а | b | L | а | b | L | а | b |
| 1 | 91 | .70 | +0.71 | +0.46 | 91.63 | +0.71 | +0.59 | 91.89 | +0.76 | +0.08 | 91.77 | +0.62 | +0.64 | 91.90 | +0.73 | +0.27 | 91.80 | +0.72 | +0.33 |
| 2 | 91 | .68 | +0.66 | +0.62 | 91.48 | +0.72 | +0.66 | 91.79 | +0.76 | +0.24 | 91.78 | +0.62 | +0.54 | 91.80 | +0.27 | +0.27 | 91.74 | +0.79 | +0.30 |
| 3 | 91 | .68 | +0.68 | +0.59 | 91.44 | +0.71 | +0.69 | 91.84 | +0.76 | +0.16 | 91.71 | +0.52 | +0.96 | 91.84 | +0.25 | +0.25 | 91.78 | +0.77 | +0.32 |
| Мах | 91 | .70 | +0.71 | +0.62 | 91.63 | +0.72 | +0.69 | 91.89 | +0.76 | +0.24 | 91.78 | +0.62 | +0.96 | 91.90 | +0.27 | +0.27 | 91.80 | +0.79 | +0.33 |
| Min | 91 | .68 | +0.66 | +0.46 | 91.44 | +0.71 | +0.59 | 91.79 | +0.76 | +0.08 | 91.71 | +0.52 | +0.54 | 91.80 | +0.25 | +0.25 | 91.74 | +0.72 | +0.30 |
| Mear | 191 | .69 | +0.68 | +0.56 | 91.52 | +0.71 | +0.65 | 91.84 | +0.76 | +0.16 | 91.75 | +0.71 | +0.71 | 91.85 | +0.26 | +0.26 | 91.77 | +0.76 | +0.32 |
| SD | 0. | .01 | 0.02 | 0.08 | 0.10 | 0.00 | 0.05 | 0.05 | 0.00 | 0.08 | 0.03 | 0.05 | 0.21 | 0.05 | 0.01 | 0.01 | 0.03 | 0.03 | 0.01 |
| | | | | | | | | | · | | | | | | | | | | |
| Me | an | Col | tton | L | - | ä | a | ł |) | | | | | | | | | | |
| | | | | 91. | .74 | +0 | .65 | +0 | .44 | | | | | | | | | | |

| | l | Utility 1 | | | Utility 2 | 2 |
|---------|-------|-----------|-------|-------|-----------|-------|
| | L | а | b | L | а | b |
| 1 | 89.50 | +0.82 | +2.07 | 89.36 | +0.84 | +1.67 |
| 2 | 89.46 | +0.76 | +2.05 | 89.33 | +0.88 | +1.65 |
| 3 | 89.44 | +0.78 | +2.03 | 89.43 | +0.89 | +1.68 |
| Max | 89.50 | +0.82 | +2.07 | 89.43 | +0.89 | +1.68 |
| Min | 89.44 | +0.76 | +2.03 | 89.33 | +0.84 | +1.64 |
| Mean | 89.47 | +0.79 | +2.05 | 89.37 | +0.87 | +1.66 |
| SD | 0.03 | 0.03 | 0.02 | 0.05 | 0.02 | 0.02 |
| | | | | | | |
| Mean | L | - | á | 9 | | b |
| Utility | 89. | .42 | +0 | .83 | +1 | .86 |

Mean Cotton

Mean Utility



Summary of Mean Values

| Test | | Cotton | | | Utility | |
|---|-------|--------|-------|-------|---------|-------|
| lest | L | а | b | L | а | b |
| Control- Untreated | 91.63 | +0.71 | +0.37 | 90.28 | +0.72 | +1.09 |
| Preventol [®] ON-S Immersion | 91.68 | +0.77 | +0.19 | 90.19 | +0.75 | +1.58 |
| Preventol [®] ON-S Spot Cleaning | 89.20 | +0.34 | +5.67 | - | - | - |
| TTO Immersion with Detergent | 91.97 | +0.83 | -0.22 | 91.05 | +0.62 | +1.18 |
| TTO Immersion with Emulsifier | 91.65 | +0.70 | +0.06 | 90.59 | +0.83 | +0.81 |
| TTO Spot Cleaning with Emulsifier | 91.74 | +0.65 | +0.44 | 89.42 | +0.83 | +1.86 |

Analysis of Results

The Control Utility fabric was slightly darker and more yellow, as shown by the +1.09 b value compared to the Control Cotton +0.37. This could have been caused by the presence of yellow cellulose degradation products. The other results were then compared to the untreated controls to assess the effect of the treatments.

The Preventol[®] ON-S Immersion Utility test showed an increase in this yellowing, which has been noted in previous trials and could have been caused by the damaging effect of the treatment on the more vulnerable aged textile which could have promoted chain scission. However, this was not supported by the Cotton sample, which had a reduction in yellowing. The greatest difference was in the Preventol[®] ON-S Spot Cleaning. As noticed during the treatment there was a great deal of brown staining produced with tide marks, which could not be rinsed away. There was a darker tone than the other samples and an increase to +5.67 showing it was much more yellow, with a decrease in red to +0.34.

The TTO with Detergent Immersion values were contrasting for the Cotton and Utility fabrics. The Cotton was less yellow and slightly to a blue tone whereas the Utility was brighter but slightly more yellow. The TTO with Emulsifier Immersion test resulted in both fabric types having a reduction in the yellow colouration. A notable difference in the colour values was found in the TTO with Emulsifier Spot Cleaning test. The Cotton exhibited a slight increase in brightness, less red and more yellow. The Utility fabric showed a darkening and increase in yellow. This was probably due to the residue remaining on the surface obscuring the textile colour itself.

Therefore there were some changes evident between the untreated Control samples and those samples treated with the fungicides. In the Cotton samples all except the Preventol[®] ON-S Spot Cleaning reduced the yellowing, and this was the only test that greatly altered and lowered the red. For the Utility fabric all of the treatments except the TTO with Emulsifier Immersion had a yellowing effect, suggesting that the aged fibres were more prone to the damaging effect of the treatments, despite the probable reduction in the water-soluble degradation products of cellulose. All of the figures measured do not show a difference in colours visible to the human eye, but they were able to be detected by the colourimeter. The colours may be more changeable if the samples underwent accelerated light and heat ageing, which should be identified for further study.

Condition Test 2: FTIR

Theory and Purpose of Test

FTIR (Fourier Transform Infrared Spectrometry) analyses the molecular structure of organic materials using infrared radiation. This test will be used to confirm the fabric samples as being cotton, and primarily aims to detect any residues left following the fungicide treatments.

By exposing the molecule to infrared radiation the bonds between carbon and other atoms, such as hydrogen, nitrogen and oxygen will vibrate, bend and stretch. The frequency at which the bonds absorb the radiation and vibrate indicates the energy required. This allows the type of bonds present, or functional groups with specific configurations of atoms and bonds, to be identified due to the frequency of radiation required. A beam of infrared radiation is sent from the spectrometer and part of the beam interacts with the sample and advances to the detector. The other part of the beam moves directly to the detector. The two beams are then compared to determine which frequencies have been absorbed by the sample.⁶⁹

The wavenumbers range from 4000cm⁻¹ to 400 cm⁻¹ and from this range the molecular structure can be ascertained on the spectrum produced by the software connected to the FTIR spectrometer. The sample can then be compared to a database of known samples to correlate the spectrum. In order to test textile samples without causing damage through the extraction of specimens, an ATR (attenuated total reflectance) accessory can be used. Small points of contact are analysed by placing the sample above a highly refractive crystal which produces an evanescent wave that is altered as the sample absorbs the energy and is reflected back to the beam before being passed to the detector in the spectrometer. (Fig. 31). There must be good contact with the sample and crystal, which is ensured by clamping the sample from above.⁷⁰ The ATR accessory used for these tests is shown in Fig. 32.

 ⁶⁹ A. Quye, *Analytical Techniques Handout*, Unpublished Lecture Notes, (2011), 2.
 ⁷⁰ Perkin Elmer, Technical Note: FTIR Spectroscopy Attenuate Total Reflectance (ATR), shop.perkinelmer.com/content/technicalinfo/tch_ftiratr.pdf. Accessed 10/8/2012, 2-3.

Figure 31: Diagram Showing Infrared Beam and ATR Crystal⁷¹



Figure 32: The ATR accessory with Textile Sample

Method of Test

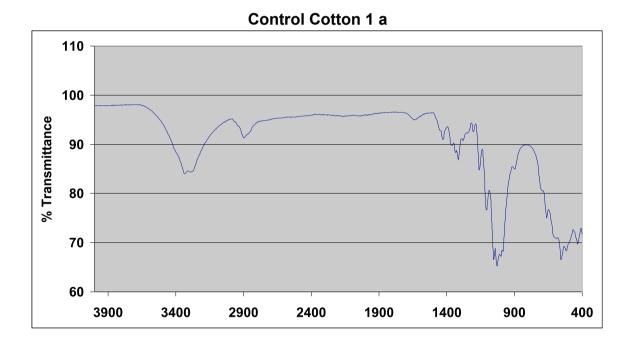
The instrument used for these tests was the Perkin Elmer Spectrum One FTIR Spectrometer with a Perkin Elmer Universal ATR Sampling Accessory. Each of the textile samples were placed onto the crystal and the clamp screwed down to achieve a Force Gauge of 50 as shown on the computer monitor. The spectrometer took an average of 16 readings per spectrometer to ensure as accurate a measurement as possible. Three areas on each textile were tested at random in this way, excepting the spot cleaning samples which were tested at random but within the treated area.

⁷¹ Perkin Elmer. Accessed 10/8/2012, 1.

Results

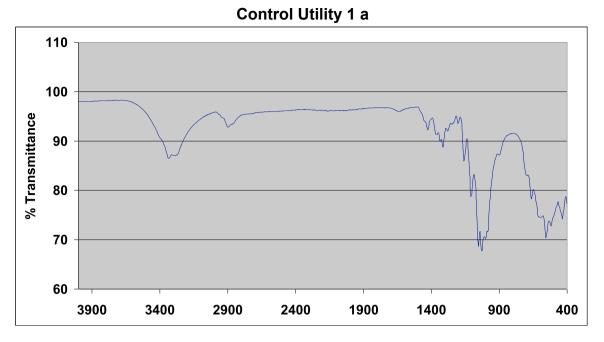
The results below show one of the spectra for each different treatment type, beginning with the untreated controls. After examination of all of the spectra it was determined that there was no discernible difference between the results for each repetition, making it unnecessary to present the repeats. The spectra are titled with the Treatment Type, Fabric Type and 1 a (each repetition for a sample was labelled a, b, or c; sample 2 or sample 3 etc. was labelled likewise).

The spectra are discussed both individually and examined as a whole in the analysis following the spectra.



This spectra shows the standard appearance of cotton when compared to a known sample from a database. The first peak at around 3300cm⁻¹ shows the O-H stretch, the peak around 2900cm⁻¹ marks the C-H stretch and the peak around 1000cm⁻¹ is the C-O stretch.⁷² All of these bonds are found in the cellulose molecule and can be used to identify the spectrum as cotton.

⁷² C. Chung et. al, 'Characterisation of Cotton Fabric Scouring by FT-IR ATR Spectroscopy', *Carbohydrate Polymers*, Vol. 58, (2004), 419.



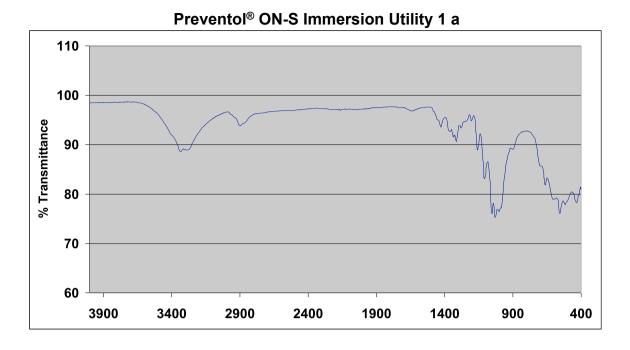
The spectrum for the Utility cotton fabric shows the same peaks as for the previous new cotton. This confirms the microscopic identification of the Utility fabric as cotton.



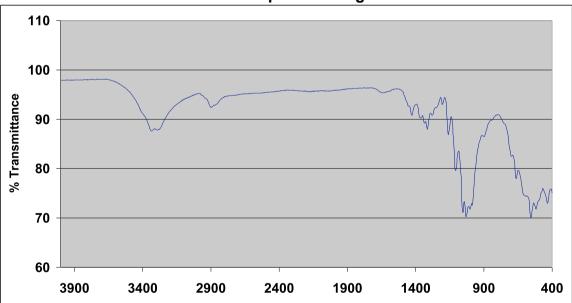
Preventol® ON-S Immersion Cotton 1 a

This spectrum is identical to the peaks of the previous untreated control samples. It could indicate that there were no residues remaining on the textile, but they could in fact be below detectable levels. It does show that there was not a large amount of the Preventol[®] ON-S present. This spectrum could have showed a larger peak for the aromatic, cyclic structure of alternating C-C single and double bonds contained in the o-phenyl phenol molecule at 1500cm⁻¹ and for the aromatic C-H stretch at 3000-3100cm⁻¹.⁷³ However, there is no peak above 3000cm⁻¹ showing it has not been detected.

⁷³ W. Volland, 'Organic Compund Identification Using Intrared Spectroscopy', http://www. 800mainstreet.com/irsp/eir.html. Accessed 10/8/2012.

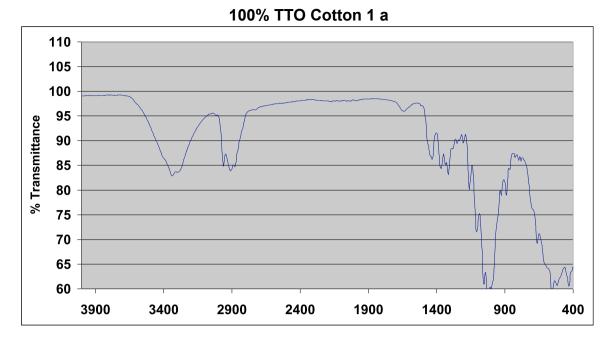


For this sample there are also no other peaks than for the cotton, indicating any residues are not above detectable levels.

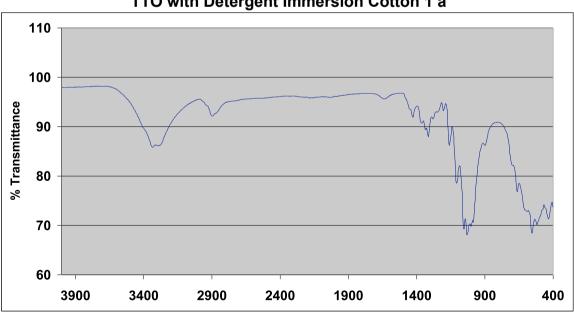


Preventol® ON-S Spot Cleaning Cotton 1 a

There is no apparent difference in this spectrum to the untreated control and only cotton is shown.



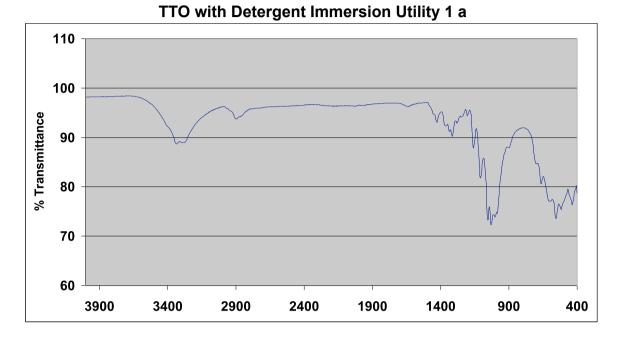
In order to establish a known sample of TTTO on cotton and to determine the presence of molecules that could could oxidation, two drops of 100% TTO were placed on a cotton sample and the spectrum was detected. There are multiple peaks around 2900cm⁻¹ showing the C-H stretch and O-H stretch which would be greater considering the presence of the terpinen-4-ol, as well as the peaks at 1000cm⁻¹ showing C-O bonds.⁷⁴ The aromatic benzene ring could have been present from the p-cymene contained in TTO. This catalyses oxidation and is damaging to textiles, but fortunately this TTO has been protected from oxidation and there is p-cymene detectable.



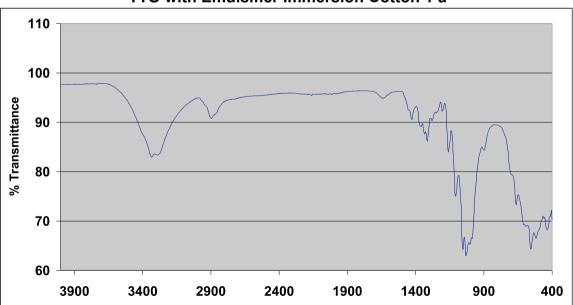
TTO with Detergent Immersion Cotton 1 a

⁷⁴ W. Volland. Accessed 10/8/2012.

The peaks seen with the 100% TTO are not evident here. This may indicate there is no TTO remaining on the textile, but it can only certainly state that it is not above detectable levels.

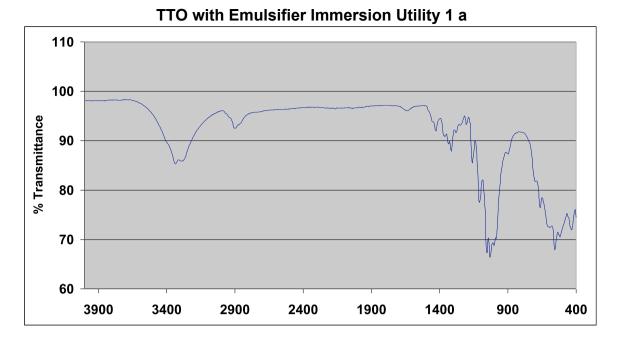


There is no apparent difference in this spectrum to the untreated control and only cotton is shown.

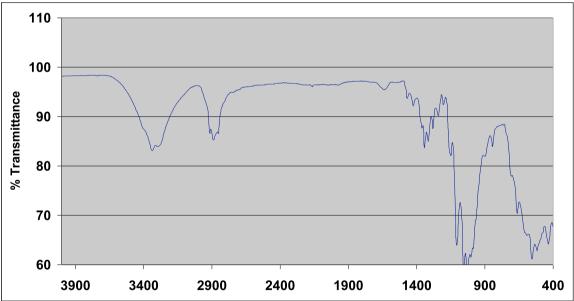


TTO with Emulsifier Immersion Cotton 1 a

There is no apparent difference in this spectrum to the untreated control and only cotton is shown.

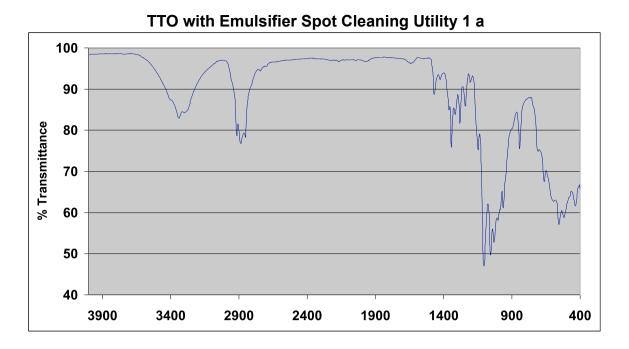


There is no apparent difference in this spectrum to the untreated control and only cotton is shown.



TTO with Emulsifier Spot Cleaning Cotton 1 a

This spectrum is comparable to the 100% TTO which could indicate the presence of TTO remaining on the textile. The peaks are at the same wavenumbers but it could also indicate the presence of the emulsifier which was a polyethoxylated fatty alcohol combined with a small percentage of detergent. This would show the same C-H and O-H stretches around 2900cm⁻¹ and the C-O at 1000cm⁻¹. Therefore although it cannot here be established that only the TTO is represented, it does confirm the presence of residues from the treatment which were identified earlier using visually analysis.



There is a more notable peak at 1500cm⁻¹ which shows the presence of aromatic C=C bonds, that are present in some of the more damaging compounds that make up TTO. They are are included in the Utility samples but not the cotton samples. However, this reason for this cannot currently be determined.

Analysis of Results

The FTIR-ATR analysis confirmed the presence of cotton for the two sample fabrics. The Preventol[®] ON-S tests did not detect any other groups other than those present in cotton, suggesting that there were no residues. This was the same for the TTO immersion tests. However, there could have been lower levels that were unable to be detected by the analysis.

The 100% TTO sample indicated that there were no aromatic compounds present which could show that the TTO had not undergone oxidation resulting in higher levels of p-cymene. The TTO Spot Cleaning tests confirmed what was made through visual identification that either the TTO or emulsifier was remaining on the textile, and was comparable to the 100% TTO spectrum. The difference in the TTO with Emulsifier Spot Cleaning Cotton and Utility samples could suggest that different residues had remained which may have been due to the difference in thread count. This would require further analysis and could be developed in future work. Therefore only the final tests were able to identify residues following the fungicide treatments.

Condition Test 3: Surface pH

Theory and Purpose of Test

pH is an abbreviation of 'power of hydrogen'. It is a scale of measurement determining the acidity or alkalinity of solutions. The scale is from 0 to 14, with 0-6 being acidic and 8-14 alkaline, while 7 is neutral. The test "records the concentration of free hydrogen ions (or more accurately the concentration of hydroxonium ions (H₃O⁺)) in an aqueous solution".⁷⁵ This is due to the ionization or dissociation of a water molecule, through the breaking of the covalent bond, resulting in a hydrogen ion (H⁺) and a hydroxyl ion (OH⁻). Testing the pH of a textile can establish the presence of acidic degradation products and show any acidic/alkaline residues remaining after a treatment. It is desirable that the pH of a textile is close to neutral, as this helps to prevent acid hydrolysis and alkaline depolymerisation.

The measurement of pH can be carried out using different methods, namely indicator solutions, indicator papers, surface pH meters and meters designed to measure the pH of solutions. The similarity lies in the fact that water must be present with all these methods.

Method of Test

The instrument used for this test was a Fisherbrand FB68797 Flat Surface pH, and read through a HANNA Instruments HI9024 Microcomputer (Fig. 33). The deionised water used had a pH of 6.6.





⁷⁵ Tímár-Balázsy & Eastop, 2007, 215.

The small area of the textile to be measured was dampened with a drop of deionised water and the flat-head probe was placed on top. Three areas of the textile were tested at random, except for the spot cleaning samples which were tested at random but only within the treated area. The three areas are listed under 1, 2, and 3 in the tables below. The monitor was read at 15, 30 and 45 seconds after contact using a stop watch due to the way in which the pH can change over the course of contact. These pH measurements allowed 9 readings to be taken for each fabric sample and a mean value to be determined. The readings were noted down and rounded to one decimal place, with the mean values having two decimal places.

The results tables for the untreated controls and each fungicidal treatment are below. They are followed by an analysis and evaluation of the results.

Results

| Surface pH | Cotton 1 | Cotton 2 | Cotton 3 | Utility 1 | Utility 2 | Utility 3 |
|------------|----------|-------------|----------|-----------|---------------|-----------|
| 1 | | | | | | |
| 15 secs | 6.5 | 6.3 | 6.8 | 6.5 | 6.4 | 6.6 |
| 30 secs | 6.4 | 6.4 | 6.7 | 6.5 | 6.4 | 6.6 |
| 45 secs | 6.4 | 6.4 | 6.7 | 6.5 | 6.4 | 6.6 |
| 2 | | | | | | |
| 15 secs | 6.3 | 6.3 | 6.7 | 6.8 | 6.6 | 6.6 |
| 30 secs | 6.3 | 6.3 | 6.7 | 6.7 | 6.6 | 6.6 |
| 45 secs | 6.3 | 6.3 | 6.7 | 6.7 | 6.7 | 6.6 |
| 3 | | | | | | |
| 15 secs | 6.4 | 6.6 | 6.7 | 6.5 | 6.7 | 6.7 |
| 30 secs | 6.4 | 6.5 | 6.7 | 6.5 | 6.7 | 6.7 |
| 45 secs | 6.4 | 6.5 | 6.7 | 6.5 | 6.6 | 6.6 |
| | | | | | · | |
| Мах | 6.5 | 6.6 | 6.8 | 6.8 | 6.7 | 6.7 |
| Min | 6.3 | 6.3 | 6.7 | 6.5 | 6.4 | 6.6 |
| Mean | 6.38 | 6.40 | 6.71 | 6.58 | 6.57 | 6.62 |
| SD | 0.06 | 0.11 | 0.03 | 0.11 | 0.12 | 0.04 |
| Mean | | Cotton= 6.5 | | | Utility= 6.59 | |
| SD | | 0.17 | | | 0.11 | |

Control

| Surface pH | Cotton 1 | Cotton 2 | Cotton 3 | Utility 1 | Utility 2 | Utility 3 |
|------------|--------------|----------|----------|-----------|---------------|-----------|
| 1 | | | | | | |
| 15 secs | 6.6 | 7.1 | 6.6 | 6.7 | 7.4 | 7.5 |
| 30 secs | 6.6 | 6.9 | 6.4 | 6.7 | 7.3 | 7.2 |
| 45 secs | 6.6 | 6.8 | 6.4 | 6.8 | 7.3 | 7.1 |
| 2 | | | | | | |
| 15 secs | 7.1 | 6.9 | 6.7 | 7 | 7.2 | 7.2 |
| 30 secs | 7 | 6.8 | 6.6 | 7 | 7.1 | 7.3 |
| 45 secs | 6.9 | 6.8 | 6.6 | 7.1 | 7 | 7.3 |
| 3 | | | | | | |
| 15 secs | 6.5 | 6.7 | 6.7 | 6.9 | 7.3 | 7.3 |
| 30 secs | 6.5 | 6.7 | 6.7 | 6.8 | 7.2 | 7.3 |
| 45 secs | 6.5 | 6.7 | 6.7 | 6.8 | 7.2 | 7.1 |
| | | | | | | |
| Мах | 7.1 | 7.1 | 6.7 | 7.1 | 7.4 | 7.5 |
| Min | 6.5 | 6.7 | 6.4 | 6.7 | 7 | 7.1 |
| Mean | 6.70 | 6.82 | 6.60 | 6.87 | 7.22 | 7.26 |
| SD | 0.22 | 0.12 | 0.12 | 0.13 | 0.11 | 0.12 |
| Mean | Cotton= 6.71 | | | | Utility= 7.11 | |
| SD | 0.18 | | | | 0.21 | |

Preventol[®] ON-S Immersion

Preventol[®] ON-S Spot Cleaning

| Surface pH | Cotton 1 | Under 1 | Cotton 2 | Under 2 | Cotton 3 | Cotton 4 | Wool 1 |
|------------|---------------|---------|----------|---------|----------|----------|--------|
| 1 | | | | | | | |
| 15 secs | 9.9 | 10.2 | 10 | 10.1 | 10.5 | 10 | 10.3 |
| 30 secs | 9.9 | 10.3 | 10 | 10.2 | 10.6 | 10 | 10.3 |
| 45 secs | 9.9 | 10.3 | 10 | 10.1 | 10.6 | 10 | 10.3 |
| 2 | | | | | | | |
| 15 secs | 10.1 | 10.4 | 10.2 | 10.4 | 10.5 | 10.5 | 10.6 |
| 30 secs | 10 | 10.3 | 10.2 | 10.4 | 10.5 | 10.5 | 10.6 |
| 45 secs | 10 | 10.3 | 10.2 | 10.3 | 10.5 | 10.5 | 10.5 |
| 3 | | | | | | | |
| 15 secs | 10.1 | 10.2 | 10 | 10.3 | 10.1 | 10.1 | 10.6 |
| 30 secs | 10.1 | 10.2 | 10 | 10.2 | 10 | 10.1 | 10.6 |
| 45 secs | 10.1 | 10.1 | 9.9 | 10.2 | 10 | 10 | 10.6 |
| | | | | | | | |
| Max | 10.1 | 10.4 | 10.2 | 10.4 | 10.6 | 10.5 | 10.6 |
| Min | 9.9 | 10.1 | 9.9 | 10.1 | 10 | 10 | 10.3 |
| Mean | 10.01 | 10.26 | 10.06 | 10.24 | 10.37 | 10.19 | 10.49 |
| SD | 0.09 | 0.08 | 0.11 | 0.11 | 0.24 | 0.22 | 0.14 |
| Mean | Cotton= 10.19 | | | | | | |
| SD | 0.20 | | | | | | |

| Surface pH | Cotton 1 | Cotton 2 | Cotton 3 | Utility 1 | Utility 2 | Utility 3 |
|------------|--------------|----------|----------|---------------|-----------|-----------|
| 1 | | | | | - | |
| 15 secs | 6.6 | 6.4 | 6.2 | 6.4 | 6.5 | 6.2 |
| 30 secs | 6.5 | 6.2 | 6.1 | 6.4 | 6.4 | 6.2 |
| 45 secs | 6.4 | 6.2 | 6.1 | 6.4 | 6.4 | 6.1 |
| 2 | | | | | | |
| 15 secs | 6.2 | 6.3 | 6.7 | 6.2 | 6.4 | 6.2 |
| 30 secs | 6.1 | 6.1 | 6.6 | 6.2 | 6.4 | 6.1 |
| 45 secs | 6 | 6.2 | 6.5 | 6.2 | 6.3 | 6.2 |
| 3 | | | | | | |
| 15 secs | 6.1 | 6.1 | 6.4 | 6.4 | 6.4 | 6.1 |
| 30 secs | 6 | 6.1 | 6.3 | 6.3 | 6.3 | 6.1 |
| 45 secs | 6 | 6 | 6.2 | 6.3 | 6.3 | 6.2 |
| | | | | | | |
| Max | 6.6 | 6.4 | 6.7 | 6.4 | 6.5 | 6.2 |
| Min | 6 | 6 | 6.1 | 6.2 | 6.3 | 6.1 |
| Mean | 6.21 | 6.18 | 6.34 | 6.31 | 6.38 | 6.16 |
| SD | 0.22 | 0.11 | 0.21 | 0.09 | 0.06 | 0.05 |
| Mean | Cotton= 6.24 | | | Utility= 6.28 | | |
| SD | | 0.20 | | | 0.12 | |

TTO with Detergent Immersion

TTO with Emulsifier Immersion

| Surface pH | Cotton 1 | Cotton 2 | Cotton 3 | Utility 1 | Utility 2 | Utility 3 |
|------------|--------------|----------|----------|-----------|---------------|-----------|
| 1 | | | | | | |
| 15 secs | 5.8 | 6.5 | 6.3 | 6.3 | 6.5 | 6.2 |
| 30 secs | 5.8 | 6.3 | 6.2 | 6.3 | 6.5 | 6.2 |
| 45 secs | 5.8 | 6.3 | 6.2 | 6.3 | 6.5 | 6.2 |
| 2 | | | | | | |
| 15 secs | 6.3 | 6.4 | 5.8 | 6.8 | 6.7 | 6.4 |
| 30 secs | 6.3 | 5.8 | 5.9 | 6.8 | 6.7 | 6.4 |
| 45 secs | 6.2 | 5.6 | 5.9 | 6.7 | 6.6 | 6.4 |
| 3 | | | | | | |
| 15 secs | 6.2 | 6.3 | 5.5 | 6.1 | 6.6 | 6.4 |
| 30 secs | 6.1 | 6.3 | 5.5 | 6.2 | 6.5 | 6.4 |
| 45 secs | 6 | 6.2 | 5.6 | 6.2 | 6.5 | 6.4 |
| | | - | - | - | | - |
| Max | 6.3 | 6.5 | 6.3 | 6.8 | 6.7 | 6.4 |
| Min | 5.8 | 5.6 | 5.5 | 6.1 | 6.5 | 6.2 |
| Mean | 6.06 | 6.19 | 5.88 | 6.41 | 6.57 | 6.33 |
| SD | 0.20 | 0.28 | 0.29 | 0.26 | 0.08 | 0.09 |
| Mean | Cotton= 6.04 | | | | Utility= 6.44 | |
| SD | | 0.29 | | | 0.19 | |

| Surface p⊦ | Cotton 1 | Cotton 2 | Cotton 3 | Cotton 4 | Cotton 5 | Cotton 6 | Utility 1 | Utility 2 |
|------------|--------------|----------|----------|----------|----------|----------|-----------|-----------|
| 1 | | | | | | | | |
| 15 secs | 6.3 | 6 | 7 | 6.7 | 5.9 | 6 | 6.3 | 6.3 |
| 30 secs | 6.2 | 6 | 6.6 | 6.5 | 6 | 6.1 | 6.2 | 6.4 |
| 45 secs | 6.1 | 6 | 6.4 | 6.3 | 6 | 6.1 | 6.3 | 6.4 |
| 2 | | | | | | | | |
| 15 secs | 6.3 | 6.1 | 6.2 | 6.4 | 5.9 | 6.1 | 6.1 | 6.6 |
| 30 secs | 6.2 | 6.1 | 6 | 6.4 | 5.9 | 6.1 | 5.7 | 6.6 |
| 45 secs | 6.2 | 6.1 | 6 | 6.4 | 5.9 | 6.2 | 6.4 | 6.5 |
| 3 | | | | | | | | |
| 15 secs | 6.1 | 6.1 | 6.1 | 5.9 | 5.9 | 6 | 6.4 | 6.4 |
| 30 secs | 6.1 | 6.1 | 6.1 | 5.8 | 5.9 | 6 | 6.3 | 6.3 |
| 45 secs | 6.1 | 6 | 6.2 | 5.8 | 5.9 | 6 | 6.3 | 6.3 |
| | | | | | | | | |
| Max | 6.3 | 6.1 | 7 | 6.7 | 6 | 6.2 | 6.4 | 6.6 |
| Min | 6.1 | 6 | 6 | 5.8 | 5.9 | 6 | 5.7 | 6.3 |
| Mean | 6.18 | 6.06 | 6.29 | 6.24 | 5.92 | 6.07 | 6.22 | 6.42 |
| SD | 0.08 | 0.05 | 0.31 | 0.31 | 0.04 | 0.07 | 0.20 | 0.11 |
| Mean | Cotton= 6.13 | | | | | Utility | = 6.32 | |
| SD | 0.22 | | | | | | 0. | 19 |

TTO with Emulsifier Spot Cleaning

Summary of Mean Surface pH Readings

| Test | Cotton | Utility |
|---|--------|---------|
| Control- Untreated | 6.5 | 6.59 |
| Preventol [®] ON-S Immersion | 6.71 | 6.11 |
| Preventol [®] ON-S Spot Cleaning | 10.19 | - |
| TTO with Detergent Immersion | 6.24 | 6.28 |
| TTO with Emulsifier Immersion | 6.04 | 6.44 |
| TTO with Emulsifier Spot Cleaning | 6.13 | 6.32 |

Analysis of Results

The pH was not seen to deviate dramatically between each of the readings taken for each sample, as calculated with the Standard Deviation (SD) for each mean. No results needed to be discounted and the mean values calculated were considered representative. The untreated Control samples were expected to differ between the aged and new fabric, with the Utility fabric more acidic due to degradation. However, the Utility fabric was in fact slightly closer to neutral.

The alkalinity of the Preventol[®] ON-S could have rendered the textiles more alkaline, but it was found that there was not much difference for the Immersion tests, suggesting that rinsing had been adequate and no alkaline residues were remaining on the samples. However, as noted through visual observations during the Preventol[®] ON-S Spot Cleaning tests there was a great deal of residue on the textile that was not rinsed away, as shown by the pH mean of 10.19. Leaving the textile at this pH would catalyse alkaline depolymerisation and cause severe degradation to the cellulose. Therefore as well as the visible brown staining this test was also chemically damaging to the fibres.

All of the TTO tests had a slightly acidic effect on the samples, caused by the low pH of the solutions used. However, the fact that there is very little difference between the control samples and the samples treated with TTO means there is no concern for the effect on the fibre condition through acidity in the long term.

Therefore, only the Preventol[®] ON-S Spot Cleaning samples were found to have a negative impact on the pH of the textile following treatment, when compared to the untreated controls. The other fungicides had little lasting impact on the surface pH.

Condition Test 4: Tensile Strength

Theory and Purpose of Test

The final test carried out was Tensile Strength. This test was used to assess the mechanical strength and elongation of the samples to establish whether the treatments had a deleterious or positive effect on the strength of the fibres. This formed the final condition test and resulted in the destruction of the samples. The sample is placed under an axial stretch and from this the breaking load and elongation (extension and stretch) can be ascertained. The factors affecting the results are fibre type, thread count and weave structure. There are two types of break modes that can be initiated by a tensile tester, but for these trials it was pull to break, which produces the graph in Fig. 34. The initial modulus, or Young's modulus is a measure of the original deformation caused by low levels of stress. This is related to fabric handle, with stiffer fabrics having a higher initial modulus. The extent of degradation, DP and cross-linking also impact on the amount of load required to break the textile. The number of strips tested, gauge length, extension rate, RH and type of jaws all affect the tensile strength results.⁷⁶ It is important therefore to maintain the same test parameters to ensure precise results.

Figure 34: Tensile Strength Test Curve (Pull to Break)77

Method of Test

The tensile tester used was the Instron[®] 5544 (Fig. 35) connected to Bluehill 2 application software. Each sample was cut into three strips 2.5mm in width, with 2.5mm from each side discarded due to fraying following the treatments. The gauge length (the gap between the jaws in which the sample was held) was set at 100mm to follow half the size

⁷⁶ X. Wang et. al, 'Physical and Mechanical Testing of Textiles', in J. Hu (ed.), *Textile Testing*,

⁽London: Woodhead Publishing, 2008), 92-96.

⁷⁷ X. Wang et. al, 2008, 93.

of BS ISO 13934-2:1999. The sample was placed between the jaws, which were activated using the foot operated suction pump shown in Fig. 36, with care taken to ensure the sample was vertical and not at an angle. The upper jaw was then raised by the load cell, with the ramp rate at a constant rate of extension of 1:10 mm/minute. The source for tensile strain was extension and was measured in mm to show the elasticity of the sample. The strength was measured through the force exerted by the load cell in newtons (N). The force was then exerted until destruction of the sample, as evident in Fig. 37.

Figure 35: Instron 5544 Tensile Tester

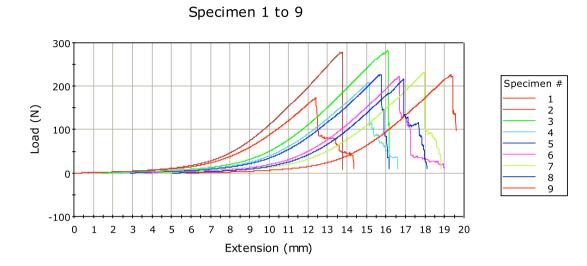


Figure 36: Tensile Tester Pump



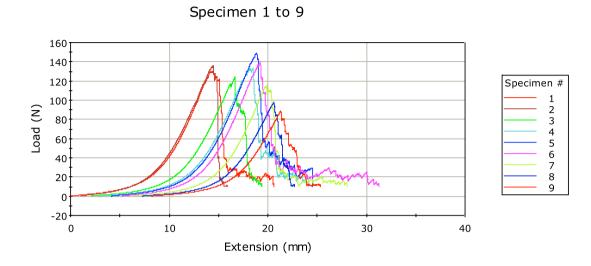
Figure 37: Strip in Jaws After Break



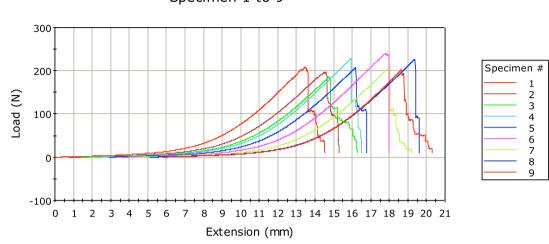


| | Maximum Load (N) | Tensile extension at Maximum Load (mm) |
|--------------------|---------------------|--|
| 1 | > 173.71 | > 12.41 |
| 2 | > 278.55 | > 13.05 |
| 3 | > 281.14 | > 14.65 |
| 4 | > 209.04 | > 12.94 |
| 5 | > 226.39 | > 12.84 |
| 6 | > 222.04 | > 13.08 |
| 7 | > 231.61 | > 13.64 |
| 8 | > 216.26 | > 11.87 |
| 9 | > 227.10 | > 13.58 |
| Mean | 229.54 | 13.12 |
| Standard Deviation | 33.27027 | 0.78997 |

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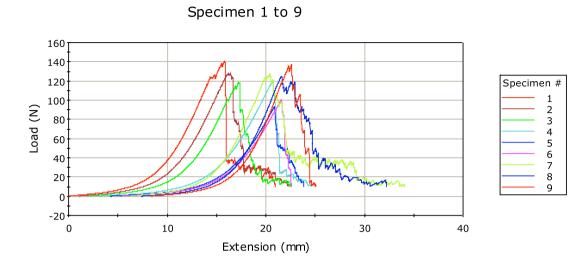


| | Maximum Load (N) | Tensile extension at Maximum Load (mm) |
|--------------------|---------------------|--|
| 1 | > 130.59 | > 14.42 |
| 2 | > 136.08 | > 13.43 |
| 3 | > 124.49 | > 14.62 |
| 4 | > 133.36 | > 15.04 |
| 5 | > 149.14 | > 14.77 |
| 6 | > 139.97 | > 14.07 |
| 7 | > 115.91 | > 13.73 |
| 8 | > 97.87 | > 13.42 |
| 9 | > 88.43 | > 13.06 |
| Mean | 123.98 | 14.06 |
| Standard Deviation | 19.93126 | 0.68978 |



| | Maximum Load (N) | Tensile extension at Maximum Load (mm) |
|--------------------|---------------------|--|
| 1 | > 209.02 | > 13.47 |
| 2 | > 197.09 | > 13.77 |
| 3 | > 185.87 | > 13.31 |
| 4 | > 229.06 | > 13.76 |
| 5 | > 208.32 | > 13.29 |
| 6 | > 240.41 | > 14.17 |
| 7 | > 210.00 | > 13.67 |
| 8 | > 226.15 | > 14.27 |
| 9 | > 201.54 | > 12.84 |
| Mean | 211.94 | 13.62 |
| Standard Deviation | 17.10104 | 0.44901 |

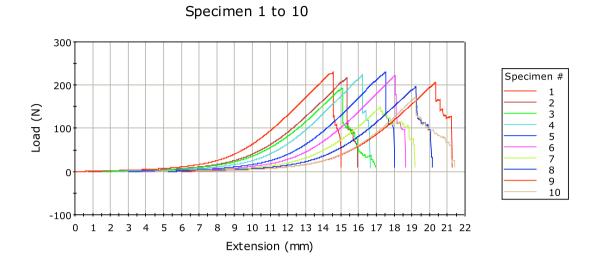
Standard Deviation



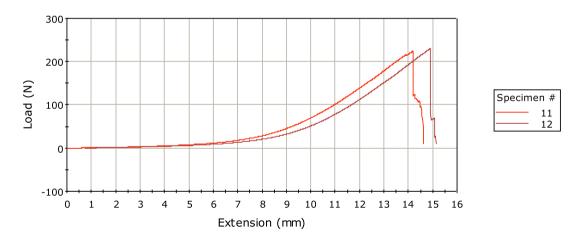
| | Maximum Load (N) | Tensile extension at Maximum Load (mm) |
|------|---------------------|--|
| 1 | > 209.02 | > 13.47 |
| 2 | > 197.09 | > 13.77 |
| 3 | > 185.87 | > 13.31 |
| 4 | > 229.06 | > 13.76 |
| 5 | > 208.32 | > 13.29 |
| 6 | > 240.41 | > 14.17 |
| 7 | > 210.00 | > 13.67 |
| 8 | > 226.15 | > 14.27 |
| 9 | > 201.54 | > 12.84 |
| Mean | 211.94 | 13.62 |

0.44901

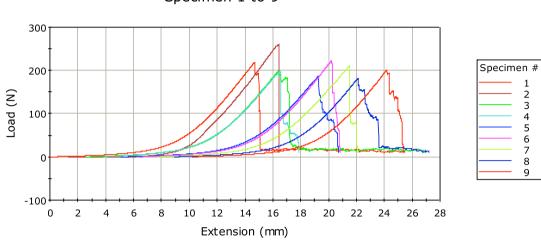
17.10104



Specimen 11 to 12

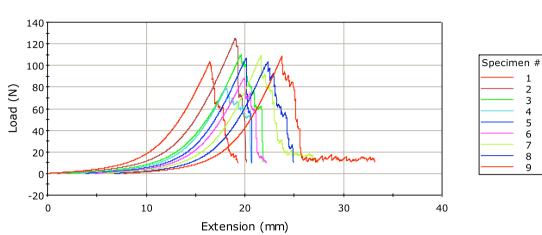


| | Maximum Load (N) | Tensile extension at Maximum Load (mm) |
|--------------------|---------------------|--|
| 1 | > 229.78 | > 14.56 |
| 2 | > 217.91 | > 14.60 |
| 3 | > 192.81 | > 13.55 |
| 4 | > 223.89 | > 13.96 |
| 5 | > 230.68 | > 14.52 |
| 6 | > 223.09 | > 14.30 |
| 7 | > 148.68 | > 12.73 |
| 8 | > 197.33 | > 13.99 |
| 9 | > 206.93 | > 14.33 |
| 10 | > 171.75 | > 12.42 |
| 11 | > 223.74 | > 14.17 |
| 12 | > 229.66 | > 14.17 |
| Mean | 208.02 | 13.94 |
| Standard Deviation | 26.04566 | 0.70504 |



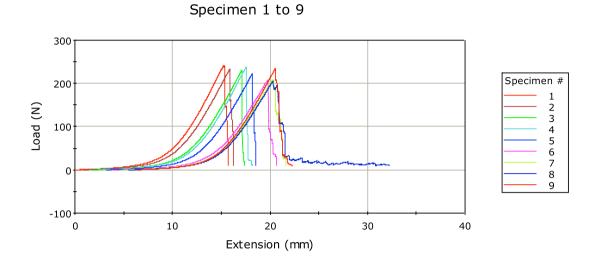
| | Maximum Load (N) | Tensile extension at Maximum Load (mm) |
|--------------------|---------------------|--|
| 1 | > 218.34 | > 14.68 |
| 2 | > 259.38 | > 15.10 |
| 3 | > 199.19 | > 13.87 |
| 4 | > 197.82 | > 12.67 |
| 5 | > 187.54 | > 14.20 |
| 6 | > 222.42 | > 13.83 |
| 7 | > 211.02 | > 13.87 |
| 8 | > 182.63 | > 13.23 |
| 9 | > 200.68 | > 14.01 |
| Mean | 208.78 | 13.94 |
| Standard Deviation | 23.06455 | 0.71804 |

Specimen 1 to 9

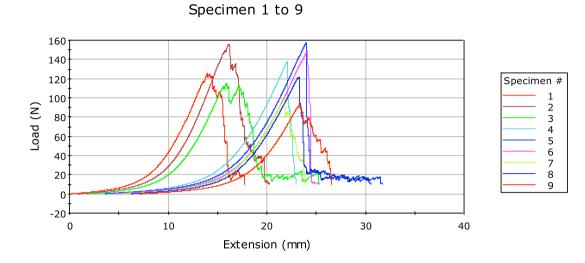


| | Maximum Load (N) | Tensile extension at Maximum Load (mm) |
|--------------------|---------------------|--|
| 1 | > 103.50 | > 16.45 |
| 2 | > 124.64 | > 18.03 |
| 3 | > 110.65 | > 17.74 |
| 4 | > 79.59 | > 15.31 |
| 5 | > 106.44 | > 16.30 |
| 6 | > 88.50 | > 15.11 |
| 7 | > 109.56 | > 15.91 |
| 8 | > 103.21 | > 15.58 |
| 9 | > 108.82 | > 16.06 |
| Mean | 103.88 | 16.28 |
| Standard Deviation | 13.07160 | 1.01430 |

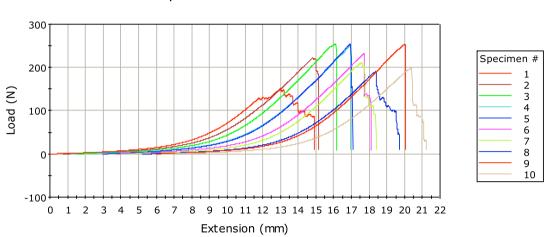
Specimen 1 to 9



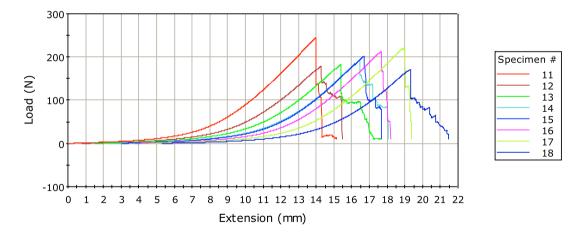
Tensile extension at Maximum Load Maximum Load (N) (mm) > 241.74 1 > 15.31 2 > 233.67 > 15.10 3 > 230.90 > 15.54 > 237.92 4 > 15.19 5 > 221.95 > 15.03 > 209.36 6 > 15.89 7 > 209.02 > 15.56 8 > 205.71 > 14.84 9 > 234.87 > 14.31 225.01 15.20 Mean Standard 13.85688 0.46085 Deviation



Tensile extension at Maximum Load Maximum Load (N) (mm) > 125.74 1 > 13.98 2 > 155.66 > 15.24 3 > 115.33 > 14.12 4 > 137.74 > 19.41 5 > 157.33 > 20.44 6 > 148.86 > 19.69 7 > 99.87 > 16.49 8 > 121.74 > 17.08 9 > 93.39 > 16.26 128.41 16.97 Mean Standard 23.31178 2.40193 Deviation

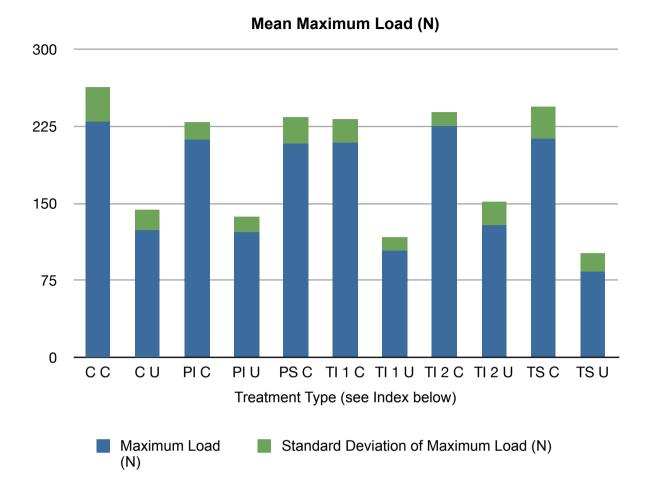


Specimen 11 to 18



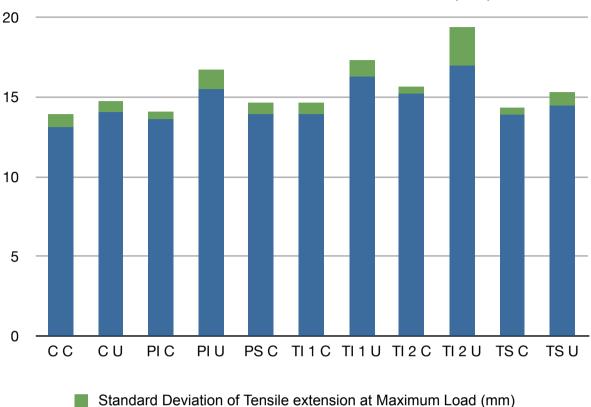
Specimen 1 to 10

| | Maximum Load (N) | Tensile extension at Maximum Load (mm) |
|--------------------|---------------------|--|
| 1 | > 152.79 | > 13.02 |
| 2 | > 222.45 | > 14.07 |
| 3 | > 255.13 | > 14.65 |
| 4 | > 250.83 | > 14.71 |
| 5 | > 254.75 | > 13.96 |
| 6 | > 232.83 | > 13.99 |
| 7 | > 212.02 | > 13.12 |
| 8 | > 191.28 | > 13.16 |
| 9 | > 253.47 | > 14.05 |
| 10 | > 199.82 | > 13.70 |
| 11 | > 244.48 | > 13.97 |
| 12 | > 178.12 | > 13.50 |
| 13 | > 182.96 | > 13.88 |
| 14 | > 192.97 | > 14.19 |
| 15 | > 201.86 | > 13.67 |
| 16 | > 213.32 | > 13.87 |
| 17 | > 220.03 | > 14.33 |
| 18 | > 171.50 | > 14.00 |
| Mean | 212.81 | 13.88 |
| Standard Deviation | 31.38639 | 0.46865 |



Index

| Bar Chart Notation | Treatment Type | |
|-----------------------|--|--|
| СС | Control Cotton | |
| CU | Control Utility | |
| PI C | Preventol [®] ON-S Immersion Cotton | |
| PI U | Preventol [®] ON-S Immersion Utility | |
| PS C | Preventol [®] ON-S Spot Cleaning Cotton | |
| TI 1 C | TTO with Detergent Immersion Cotton | |
| TI 1 U | TTO with Detergent Immersion Utility | |
| TI 2 C | TTO with Emulsifier Immersion Cotton | |
| TI 2 U | TTO with Emulsifier Immersion Utility | |
| TS C | TTO with Emulsifier Spot Cleaning Cotton | |
| TS C | TTO with Emulsifier Spot Cleaning Utility | |



Standard Deviation of Tensile extension at Maximum Load (mm) Tensile extension at Maximum Load (mm)

Index

| Bar Chart Notation | Treatment Type |
|-----------------------|--|
| CC | Control Cotton |
| CU | Control Utility |
| PI C | Preventol [®] ON-S Immersion Cotton |
| PI U | Preventol [®] ON-S Immersion Utility |
| PS C | Preventol [®] ON-S Spot Cleaning Cotton |
| TI 1 C | TTO with Detergent Immersion Cotton |
| TI 1 U | TTO with Detergent Immersion Utility |
| TI 2 C | TTO with Emulsifier Immersion Cotton |
| TI 2 U | TTO with Emulsifier Immersion Utility |
| TS C | TTO with Emulsifier Spot Cleaning Cotton |
| TS C | TTO with Emulsifier Spot Cleaning Utility |

Analysis of Results

<u>Control Cotton and Utility</u>: The treatments on Cotton samples all had a deleterious effect on the tensile strength when compared to the Control Cotton. The Control Utility samples had a much lower tensile strength than the Control Cotton samples. This was probably due to the lower thread count and looser weave structure, allowing the fabric to break at a lower force. The Utility samples generally had a much higher initial modulus, showing they were stiffer than the Cotton.

<u>Preventol[®] ON-S</u>: The Preventol[®] ON-S tests were all deleterious to the tensile strength, which was expected based on the previous literature and chemical composition of the fungicide. The greatest reduction in strength occurred with the Preventol[®] ON-S Immersion Cotton test.

<u>TTO with Detergent</u>: The Immersion tests on the Cotton and Utility fabrics had an adverse effect on the tensile strength but increased the extension. The weakening could have been due to the amount of detergent used or the extended rinsing, which also could have increased the bound water and allowed greater flexibility.

<u>TTO with Emulsifier Immersion</u>: The Cotton test had the least reduction in tensile strength. The Utility test did have a positive effect on the tensile strength, with more force required to break the sample. It also greatly increased the tensile extension. This could indicate a benefit in carrying out this treatment but more tests should be carried out to corroborate these findings. The initial modulus was much higher for these tests, suggesting there was an increase in stiffness, which should also be examined further.

<u>TTO with Emulsifier Spot Cleaning</u>: The least reduction occurred from the TTO with Emulsifier Spot Cleaning Cotton test, indicating that the TTO/Emulsifier residue was having a strengthening effect on the fibres, despite the other negative effects on surface texture and handle. The result of the Utility test was opposed to the Cotton, with a marked reduction in strength for the Utility fabric. This suggests that for the Utility fabric the residues were having a negative effect on fibre strength, which could be due to the difference in the weave structure and age.

Therefore the test with the least negative effect on tensile strength for both the Cotton and Utility fabrics was the TTO with Emulsifier Immersion test, with the Utility fabric being strengthened compared to the Control.

The tensile extension was not greatly different for most of the tests but the positive effect of all of the treatments was found. A marked improvement in extension was found following the TTO with Emulsifier Immersion Utility treatment, which was also most beneficial to the Cotton. The immersion treatments on the Utility samples showed that this textile particularly benefitted from exposure to water. The water could have removed some degradation products, soiling and impurities that were causing damage as well as reintroducing water as a plasticiser into the fibre structure and improving flexibility and stretch.

CHAPTER FIVE

Conclusions

The fungi affecting cellulosic textile have been examined including the structure of cotton, the degradation mechanisms and the effect of fungi on the condition of the fibre. Fungi and the enzymes they produce can infiltrate the fibre structure to the hollow lumen at the centre or can colonise the upper weave structure. Allowing fungal growth therefore can have an extremely deleterious effect upon the condition of the object. The process is initiated with the flow of air to transport the spores or conidia onto the surface of the textile. The conditions necessary for activation of fungi and the growth of hyphae into mycelia are adequate moisture, nutrients, temperature and pH.

The effect of the two fungicides tested on fungi has already been established in previous studies and is supported by an analysis of the chemistry of the two treatments. The Preventol[®] ON-S renders the fungi dormant whilst the TTO ruptures the cell membrane and prevents any future activation, making it a more effective fungicide of the two. The primary function of using Preventol[®] ON-S was to indicate a level of comparison by which to assess the TTO. This study and the series of experiments were designed to find an effective method of treatment for cotton and establish the effect of the treatment on the condition of the textiles through four sets of condition tests. Two types of cotton were used for testing, a new cotton lawn and a cotton fabric produced under the British Utility Scheme (1941-1952). This allowed comparison between new and aged cotton fabrics.

The treatment for the Preventol[®] ON-S samples was carried out using a 1% solution in deionised water. The solution was found to turn brown after a period of roughly two weeks, meaning if extended use of the fungicide was planned, new solutions should be regularly made up. The Preventol[®] ON-S Spot Cleaning tests had the most negative visual ramifications. The samples exhibited brown staining with tide marks which could not be removed through rinsing. This could have been due to increased oxidation under higher air pressure.

The TTO was first used with detergent as an emulsifier. However, to gain sufficient suspension of the oil in water a very high percentage of detergent was necessary which would be unacceptable for historic objects. The treatment also left a strong smell on the textile, which could be undesirable and indicates there was TTO remaining. An emulsifier was then found which allowed better suspension of the oil and was adequate for the Immersion treatment with only a faint smell lasting 2 days. However, the Spot Cleaning tests for this were negative. There was a noticeable build up on the surface of the textile

of a white waxy substance. It was probably the result of using the emulsifier under vacuum suction which held on the surface of the textile.

Therefore both of the Spot Cleaning treatments were determined inviable. The condition tests were carried out and evaluated but the results should be discounted at this stage due to the undesirable effect of the treatments.

The four condition tests were then carried out, comparing the results to untreated Controls. The first test assessed the impact of the treatments on colour change through use of the colourimeter. It was found that although there was not an extensive change, trends in the effect of the treatments could be identified. Both the Preventol[®] ON-S Immersion and TTO with Detergent Immersion tests increased the yellowing of the Utility sample whilst decreased the yellowing of the Cotton sample. This could be due to the already vulnerable, aged Utility cotton which was further damaged by the treatments through chain scission. The new Cotton fabric should have been less liable to chain scission and could have benefitted from the cleaning effect of the treatments. The TTO with Emulsifier Immersion had universally positive effect on the samples, with both Cotton and Utility showing a slight increase in brightness and a reduction in yellowing. Therefore the most positive effect was found through the TTO with Emulsifier Immersion.

The FTIR tests were useful in confirming the identification of both of the fabrics as cotton. For the majority of the results there was no difference found between the untreated Controls and treated samples. This could have proved that no residues were remaining but it could only be definitely stated that any residues were not above detectible levels. A 100% TTO on cotton sample was analysed which showed that the fungicide used had not undergone unwanted oxidation, which would have made it more damaging to the textiles. The residues from the TTO with Emulsifier Spot Cleaning were identifiable on the spectra, confirming visual observations.

The surface pH meter analysis of the textiles showed the rinsing for all of the treatments was adequate, with little effect on the final pH of the samples (except for the Preventol[®] ON-S Spot Cleaning). This demonstrates that despite being exposed to extremes in acidity or alkalinity during treatment this expose was not long lasting.

The tensile strength and elongation was only markedly improved for the TTO with Emulsifier Immersion test on the Utility fabric. All of the other tests were found to have reduced the tensile strength but improved the tensile extension. This was probably due to the damaging overall effect of the treatments, but with the introduction of water there was a greater potential for stretch due to the plasticising properties.

Therefore, the only test that was generally beneficial to the condition of the samples was the TTO with Emulsifier Immersion test. It increased brightness and reduced yellowing, did not leave any detectible residues during visual and FTIR analysis, and had little effect on the final surface pH. The tensile strength and extension results were also the most positive. Although the tensile strength of the Cotton was reduced when compared to the Control, the Utility was in fact stronger, and both were markedly more flexible despite the high initial modulus.

Therefore this treatment has found to have a neutral or positive effect on the condition on the textile samples and warrants further testing in order to corroborate these results. Although the anti-fungal properties of TTO are known it would be beneficial to test this solution directly on fungi, as this could show the effectiveness in the removal of staining as well as the ease of removal and treatment times required. In this way the method could be developed until practice on heritage objects became viable.

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APPENDIX ONE

List of Suppliers

Emulsifying Wax (Steareth-21)

Company name: The Soap Kitchen Ltd Address: 11a South Street Torrington Devon EX38 8AA UK Web: www.thesoapkitchen.co.uk Web: www.soapkitchenonline.co.uk/acatalog/index.html

Organic Tea Tree Essential Oil (Melaleuca alternifolia)

Company name: NHR Organic Oils Address: 24 Chatham Place Brighton BN1 3TN UK Web: www.nhrorganicoils.com

Preventol® ON-S

APPENDIX TWO

Conforms to Regulation (EC) No. 1907/2006 (REACH), Annex II - United Kingdom (UK)

SAFETY DATA SHEET

PREVENTOL ON-S

ANXESS Energizing Chemistry

00722650

SECTION 1: Identification of the substance/mixture and of the company/undertaking

| 1.1 Product identifier | |
|--|---|
| Product name | : PREVENTOL ON-S |
| 1.2 Relevant identified uses of | of the substance or mixture and uses advised against |
| Uses | : Biocide for industrial application |
| 1.3 Details of the supplier of the supplication of the supplic | the safety data sheet |
| Supplier | : LANXESS Deutschland GmbH, Industrial & Environmental Affairs 51369 Leverkusen, Germany, Telephone: +49 214 30 65109 E-mail: infosds@lanxess.com |
| 1.4 Emergency telephone number | : 0870 190 6777. National Chemical Emergency Centre |

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

 \square

| Classification according to D | Directive 1999/45/EC [DPD] | |
|-------------------------------|--|-----------------------------|
| Classification | : ₩n; R22 C; R34 Xi; R37 N; R50 | |
| Human health hazards | : Harmful if swallowed. Causes burns. Irritating to respi | iratory system. |
| Environmental hazards | : Very toxic to aquatic organisms. | |
| 2.2 Label elements | | |
| Hazard symbol or symbols | : | |
| | Contains | |
| | sodium 2-biphenylate sodium hydroxide | |
| Risk phrases | R22- Harmful if swallowed. R34- Causes burns. R37- Irritating to respiratory system. R50- Very toxic to aquatic organisms. | |
| Safety phrases | S26- In case of contact with eyes, rinse immediately wi water and seek medical advice. S36/37/39- Wear suitable protective clothing, gloves an protection. S45- In case of accident or if you feel unwell, seek mer immediately (show the label where possible). S60- This material and its container must be disposed hazardous waste. | nd eye/face dical advice |
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Contains

S61- Avoid release to the environment. Refer to special instructions/safety data sheet.

2.3 Other hazards

Other hazards which do not : Not available. result in classification

SECTION 3: Composition/information on ingredients

| Product definition (REACH) : Mixture | | | | |
|--------------------------------------|--|---------|---|---|
| | | | Classification | |
| Product/ingredient name | Identifiers | % | 67/548/EEC | Regulation (EC) No. 1272/2008 [CLP] |
| sodium 2- biphenylate | EC: 205-055-6 CAS: 132-27-4 Index: 604-021-00-1 | 70 - 80 | Xn; R22 Xi; R41, R37/38 N; R50 | Acute Tox. 4, H302 Skin Irrit. 2, H315 Eye Dam. 1, H318 STOT SE 3, H335 Aquatic Acute 1, H400 |
| sodium hydroxide | EC: 215-185-5 CAS: 1310-73-2 Index: 011-002-00-6 | 1 - 2 | C; R35 | Skin Corr. 1A, H314 |
| | | | See section 16 for the full text of the R- phrases declared above | See Section 16 for the full text of the H statements declared above. |

Occupational exposure limits, if available, are listed in Section 8.

SECTION 4: First aid measures 4.1 Description of first aid measures Inhalation : Cet medical attention immediately. Move exposed person to fresh air. If it is suspected that fumes are still present, the rescuer should wear an appropriate mask or self-contained breathing apparatus. Keep person warm and at rest. If not breathing, if breathing is irregular or if respiratory arrest occurs, provide artificial respiration or oxygen by trained personnel. It may be dangerous to the person providing aid to give mouth-to-mouth resuscitation. If unconscious, place in recovery position and get medical attention immediately. Maintain an open airway. Loosen tight clothing such as a collar, tie, belt or waistband. Ingestion Set medical attention immediately. Wash out mouth with water. : Move exposed person to fresh air. Keep person warm and at rest. If material has been swallowed and the exposed person is conscious, give small quantities of water to drink. Stop if the exposed person feels sick as vomiting may be dangerous. Do not induce vomiting unless directed to do so by medical personnel. If vomiting occurs, the head should be kept low so that vomit does not enter the lungs. Chemical burns must be treated promptly by a physician. Never give anything by mouth to an unconscious person. If unconscious, place Date of issue 2011-05-06 : Page: 2/11

| P REVENTOL ON-S | 00722650 / 1.01 |
|-------------------------|---|
| | in recovery position and get medical attention immediately. Maintain an open airway. Loosen tight clothing such as a collar, tie, belt or waistband. |
| Skin contact | : Wash skin immediately with plenty of water and soap. Remove contaminated clothing and shoes. Wash clothing before reuse. Clean shoes thoroughly before reuse. Get medical attention if irritation occurs. |
| Eye contact | : Set medical attention immediately. Immediately flush eyes with plenty of water, occasionally lifting the upper and lower eyelids. Check for and remove any contact lenses. Continue to rinse for at least 10 minutes. Chemical burns must be treated promptly by a physician. |
| 4.2 Most important symp | toms and effects, both acute and delayed |

See Section 11 for more detailed information on health effects and symptoms.

4.3 Indication of any immediate medical attention and special treatment needed

Notes to physician : Basic aid, decontamination, symptomatic treatment.

See Section 11 for more detailed information on health effects and symptoms.

SECTION 5: Firefighting measures

| 5.1 Extinguishing media Suitable extinguishing media | : | In case of fire, use water spray (fog), foam, dry chemical or CO_2 . | | | | | |
|--|---|--|--|--|--|--|--|
| Unsuitable extinguishing media | : | None known. | | | | | |
| 5.2 Special hazards arising from | 5.2 Special hazards arising from the substance or mixture | | | | | | |
| Hazards from the substance or mixture | : | No specific fire or explosion hazard. | | | | | |
| Hazardous combustion products | : | Decomposition products may include the following materials: carbon oxides | | | | | |
| 5.3 Advice for firefighters | | | | | | | |
| Special precautions for fire- fighters | : | Fromptly isolate the scene by removing all persons from the vicinity of the incident if there is a fire. No action shall be taken involving any personal risk or without suitable training. This material is very toxic to aquatic organisms. Fire water contaminated with this material must be contained and prevented from being discharged to any waterway, sewer or drain. | | | | | |
| Special protective equipment for fire-fighters | : | Fire-fighters should wear appropriate protective equipment and self- contained breathing apparatus (SCBA) with a full face-piece operated in positive pressure mode. | | | | | |

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SECTION 6: Accidental release measures

| 6.1 Personal precautions, protective equipment and emergency procedures | : No action shall be taken involving any personal risk or without suitable training. Keep unnecessary and unprotected personnel from entering. Do not touch or walk through spilt material. Provide adequate ventilation. Put on appropriate personal protective equipment (see Section 8). |
|---|---|
| 6.2 Environmental precautions | : Kvoid dispersal of spilt material and runoff and contact with soil, waterways, drains and sewers. Inform the relevant authorities if the product has caused environmental pollution (sewers, waterways, soil or air). Very toxic to aquatic organisms. |
| 6.3 Methods and materials fo | r containment and cleaning up |
| Small spill | : Move containers from spill area. Vacuum or sweep up material and place in a designated, labelled waste container. Dispose of via a licensed waste disposal contractor. |
| Large spill | : Move containers from spill area. Prevent entry into sewers, water courses, basements or confined areas. Vacuum or sweep up material and place in a designated, labelled waste container. Dispose of via a licensed waste disposal contractor. Note: see section 1 for emergency contact information and section 13 for waste disposal. |
| 6.4 Reference to other sections | : See Section 1 for emergency contact information. See Section 8 for information on appropriate personal protective equipment. See Section 13 for additional waste treatment information. |

SECTION 7: Handling and storage

| 7.1 Precautions for safe handling | : Put on appropriate personal protective equ Eating, drinking and smoking should be pro- material is handled, stored and processed. hands and face before eating, drinking and eyes or on skin or clothing. Do not ingest. A environment. Use only with adequate ventil respirator when ventilation is inadequate. K container or an approved alternative made material, kept tightly closed when not in us product residue and can be hazardous. | ohibited in areas where this Workers should wash I smoking. Do not get in Avoid release to the lation. Wear appropriate keep in the original from a compatible |
|--|--|--|
| 7.2 Conditions for safe storage, including any incompatibilities | : Store in accordance with local regulations. protected from direct sunlight in a dry, cool away from incompatible materials (see sec drink. Keep container tightly closed and se Containers that have been opened must be kept upright to prevent leakage. Do not sto containers. Use appropriate containment t contamination. | and well-ventilated area, tion 10) and food and ealed until ready for use. carefully resealed and ore in unlabelled |
| 7.3 Specific end use(s) | | |
| Recommendations | : Not available. | |
| Industrial sector specific solutions | : Not available. | |
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Remarks

: Take precautionary measures against electrostatic discharges. Vent waste air only via suitable separators or scrubbers. Ensure effective ventilation.

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

| Ingredient name | Occupational exposure limits |
|--|--|
| sodium hydroxide | EH40/2005 WELs (United Kingdom (UK), 8/2007). STEL: 2 mg/m ³ 15 minute(s). |
| Recommended monitoring procedures | : If this product contains ingredients with exposure limits, personal, workplace atmosphere or biological monitoring may be required to determine the effectiveness of the ventilation or other control measures and/or the necessity to use respiratory protective equipment. Reference should be made to European Standard EN 689 for methods for the assessment of exposure by inhalation to chemical agents and national guidance documents for methods for the determination of hazardous substances. |
| 8.2 Exposure controls <u>Risk management measures</u> | |
| Occupational exposure contr | <u>ols</u> |
| Technical measures | : Use only with adequate ventilation. If user operations generate dust, fumes, gas, vapour or mist, use process enclosures, local exhaust ventilation or other engineering controls to keep worker exposure to airborne contaminants below any recommended or statutory limits. |
| Personal protection measures | |
| Respiratory protection | : Use a properly fitted, air-purifying or air-fed respirator complying with an approved standard if a risk assessment indicates this is necessary. Respirator selection must be based on known or anticipated exposure levels, the hazards of the product and the safe working limits of the selected respirator. Recommended: In case of dust formation particle filter P2. |
| Hand protection | : Chemical-resistant, impervious gloves complying with an approved standard should be worn at all times when handling chemical products if a risk assessment indicates this is necessary. After contamination with product change the gloves immediately and dispose of them according to relevant national and local regulations Recommended: (< 1 hour) Polychloroprene - CR, Polyvinyl chloride - PVC. |
| Eye protection | : Safety eyewear complying with an approved standard should be used when a risk assessment indicates this is necessary to avoid exposure to liquid splashes, mists, gases or dusts. Recommended: Tightly fitting safety goggles. |
| Skin protection | : Fersonal protective equipment for the body should be selected based on the task being performed and the risks involved and should be approved by a specialist before handling this product. Recommended: Wear protective clothing. |
| | |

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| Ingredient name | Occupational exposure limits |
|---------------------------|---|
| Hygiene measures | : Wash hands, forearms and face thoroughly after handling chemical products, before eating, smoking and using the lavatory and at the end of the working period. Appropriate techniques should be used to remove potentially contaminated clothing. Wash contaminated clothing before reusing. Ensure that eyewash stations and safety showers are close to the workstation location. |
| Environmental exposure co | <u>ntrols</u> |
| Technical measures | : Emissions from ventilation or work process equipment should be checked to ensure they comply with the requirements of environmental protection legislation. In some cases, fume scrubbers, filters or engineering modifications to the process equipment will be necessary to reduce emissions to acceptable |

SECTION 9: Physical and chemical properties

levels.

9.1 Information on basic physical and chemical properties

| General information | | | | | | |
|--|--|--|--|--|--|--|
| <u>Appearance</u> | | | | | | |
| Physical state | : <mark>S</mark> olid. | | | | | |
| Colour | : White to yellowish. | | | | | |
| Odour | : Faint odour. | | | | | |
| Important health, safety and environmental information | | | | | | |
| рН | : 11,1 to 11,8 [Conc. (% w/w): 2%] | | | | | |
| Melting point | : 37°C - 120°C | | | | | |
| Density | : <mark>1∕,</mark> 36 kg/L (20 °C) | | | | | |
| Bulk density | : 🗚95 kg/m³ | | | | | |
| Solubility | : 1/200 g/l (water) | | | | | |
| Partition coefficient: n- octanol/water | : 2,95 (calculated) | | | | | |
| Combustibility at 20 °C | : BZ 2 = brief ignition and rapid extinction (VDI 2263). | | | | | |
| Ignition temperature | : >500°C | | | | | |
| 9.2 Other information | | | | | | |

No additional information.

SECTION 10: Stability and reactivity

| 10.1 Reactivity | : | No specific test data related to reactivity available for this its ingredients. | product or |
|--|---|---|------------|
| 10.2 Chemical stability | : | The product is stable. | |
| 10.3 Possibility of hazardous reactions | : | Under normal conditions of storage and use, hazardous will not occur. | reactions |
| 10.4 Conditions to avoid | : | If the case of dusty organic products the possibility of a cexplosion should always be considered. No hazardous r when used as directed. Avoid release to the environment | eactions |
| 10.5 Incompatible materials | : | No specific data. | |
| 10.6 Hazardous decomposition products | : | Under normal conditions of storage and use, hazardous decomposition products should not be produced. | |
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| SECTION 11: Toxicological information | | | | | | | | |
|--|--------------------------------------|------------------|------------|----------------|-----------|---|-------------|----------------------|
| 11.1 Information on toxic | cologica | l effect | s | | | | | |
| Potential acute health ef | fects | | | | | | | |
| Inhalation | : ritating to respiratory system. | | | | | | | |
| Ingestion | : | Harmfi stomad | | owed. Ma | ay caus | se burns to r | nouth, | throat and |
| Skin contact | : | Corros | ive to the | skin. Ca | uses b | ourns. | | |
| Eye contact | : | Corros | ive to eye | s. | | | | |
| Acute toxicity | | | | | | | | |
| Product/ingredient name | Resul | t : | Species | Dose | | Exposure | Tes | t |
| REVENTOL ON-S | LD50 Oral | - 1 | Rat | 1720 n | ng/kg | - | * | |
| REVENTOL ON-S | LC50 Inhalati Dusts a mists | ion | Rat | >1331 mg/m³ | | 1 hours | * | |
| Irritation/Corrosion | | | | | | | | |
| Skin | : | corrosi | ve (Rabbi | it) * | | | | |
| Eyes | : | corrosi | ve (Rabbi | it) * | | | | |
| <u>Sensitiser</u> | | | | | | | | |
| Product/ingredient | Route | | Species | S | Resu | ult | Test c | lescription |
| name s odium 2-biphenylate | expos skin | ure | Guinea p | ia | Note | sensitizing | | |
| Skin | - | odium | • | 0 | | • | - um sal | t·Non-sensitiser |
| Skin : sodium 2-biphenylate; 2-phenylphenol, sodium salt:Non-sensitiser. | | | | | | | | |
| Mutagenicity | 0110010 | | | | | | | |
| Product/ingredient nam | ıe | Test | | | Expe | riment | Re | esult |
| sodium hydroxide | | Ames | test | | Subj | eriment: In vi ect: Bacteria Ibolic activat | I | egative |
| | | Micron | ucleus as | say | Subj | eriment: In vi ect: Imalian-Anir | | egative |
| Teratogenicity | | | | | | | | |
| Product/ingredient nam | ne | Result | : | Spec | ies | Dose | | Exposure |
| sodium 2-biphenylate | | Positiv | e - Oral | Mou | se | <900 r NOAE | | 15 days Gestation |
| Remarks | : | * Test | results on | a produc | ct of sir | milar compo | sition | |

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SECTION 12: Ecological information

| 12.1 Toxicity | | | | | | | | |
|--|------|--|------------------------|------------|---------------------------------------|--------------|--|--|
| Product/ingredient name | | Test | Result | | Species | Exposure | | |
| REVENTOL ON-S | | * | Acute EC50 7,4 mg/l | - | Algae - Scenedesmus subspicatus | 72 hours | | |
| | | * | Acute EC50 3,8 mg/l | - | Daphnia | 48 hours | | |
| Conclusion/Summary | : | Not available. | | | | | | |
| 12.2 Persistence and degrad | lab | ility | | | | | | |
| Product/ingredient name | | Aquatic half-life | Photoly | <u>sis</u> | Biodegradability | | | |
| REVENTOL ON-S sodium 2-biphenylate | | - | - | | Readily Readily | | | |
| Conclusion/Summary | : | Not available. | | | Redding | 1 | | |
| 12.3 Bioaccumulative potent | tial | | | | | | | |
| Bioaccumulative potential | | | | | | | | |
| Product/ingredient name | | LogPow | BCF | | Potential | | | |
| sodium 2-biphenylate | | 0.6 | 22 | | low | | | |
| 12.4 Mobility in soil | | | | | | | | |
| Soil/water partition coefficient (K _{oc}) | : | Not available. | Not available. | | | | | |
| Mobility | - | Not available. | Not available. | | | | | |
| 12.5 Results of PBT and vPvB assessment | | | | | | | | |
| PBT | : | Not applicable. | | | | | | |
| vPvB | : | Not applicable. | | | | | | |
| 12.6 Other adverse effects | | | | | | T 1 : | | |
| Environmental effects | : | Very toxic to aquatic organisms. Readily biodegradable This product shows a low bioaccumulation potential. | | | | | | |
| Other adverse effects | : | Not available. | | | | | | |
| ΑΟΧ | : | The product does not contain organically bound halogens which could lead to an AOX value in waste water. | | | | | | |
| Biodegradability/Elimination | n fr | om the water: | | | | | | |
| Degradability | : | >80% | | | | | | |
| Acute bacterial toxicity (EC50) | : | ▶ 1000 mg/l (Pseudomonas fluorescens) | | | | | | |
| Remarks | : | * Test results on a product of similar composition | | | | | | |

SECTION 13: Disposal considerations

13.1 Waste treatment methods <u>Product</u>

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| P REVENTOL ON-S | 00722650/1.01 |
|------------------------|--|
| Methods of disposal | : Examine possibilities for re-utilisation. Product residues and uncleaned empty containers should be packaged, sealed, labelled, and disposed of or recycled according to relevant national and local regulations. Where large quantities are concerned, consult the supplier. When uncleaned empty containers are passed on, the recipient must be warned of any possible hazard that may be caused by residues. For disposal within the EC, the appropriate code according to the European Waste List (EWL) should be used. It is among the tasks of the polluter to assign the waste to waste codes specific to industrial sectors and processes according to the European Waste List (EWL). |
| Hazardous waste | : The classification of the product may meet the criteria for a hazardous waste. |
| Packaging | |
| Methods of disposal | : The generation of waste should be avoided or minimised wherever possible. Waste packaging should be recycled. Incineration or landfill should only be considered when recycling is not feasible. |
| Special precautions | : This material and its container must be disposed of in a safe way. Care should be taken when handling emptied containers that have not been cleaned or rinsed out. Empty containers or liners may retain some product residues. Avoid dispersal of spilt material and runoff and contact with soil, waterways, drains and sewers. |

SECTION 14: Transport information ADR/RID ADN/ADNR IMDG ΙΑΤΑ 14.1 UN number UN1759 UN1759 UN1759 UN1759 14.2 UN proper CORROSIVE CORROSIVE CORROSIVE SOLID, N.O.S. shipping name SOLID, N.O.S. SOLID, N.O.S. SOLID, N.O.S. (SODIUM (SODIUM (SODIUM (SODIUM HYDROXIDE, HYDROXIDE, HYDROXIDE, HYDROXIDE, SODIUM 2-SODIUM 2-SODIUM 2-SODIUM 2-**BIPHENYLATE**) **BIPHENYLATE**) **BIPHENYLATE**) BIPHENYLATE) 14.3 Transport hazard ¥⁄_ ¥ class(es)/Marks 14.4 Packing Ш Ш Ш Ш group 14.5 Yes. Yes. Yes. Yes. Environmental hazards 14.6 Special Emergency Passenger **Hazard** Hazard schedules (EmS) precautions for identification identification aircraft 860: 25 kg user/Additional <u>number</u> <u>number</u> F-A, S-B information 80 80 Cargo aircraft Limited quantity Limited quantity 864: 100 kg LQ24 LQ24

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|----------------------------------|
|----------------------------------|

14.7 Transport in bulk according to Annex II : Not available. of MARPOL 73/78 and the IBC Code

Hazard notes:

Slightly corrosive. Has a slight odour. Keep dry. Keep separated from foodstuffs.

SECTION 15: Regulatory information

15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture

EU Regulation (EC) No. 1907/2006 (REACH)

| Annex XVII - Restrictions on the manufacture, placing on the market and use of certain dangerous substances, mixtures and articles | : № ot applicable. | |
|---|---------------------------|--|
| | — | |

15.2 Chemical Safety: This product contains substances for which Chemical SafetyAssessmentAssessments are still required.

SECTION 16: Other information

| | | 2011-05-06 | Page: 10/11 |
|--|-----|--|-------------|
| Indicates information that h | nas | changed from previously issued version. | |
| Version | : | 1.01 | |
| Date of previous issue | : | 2008-12-19 | |
| <u>History</u> Date of issue | : | 2011-05-06 | |
| Full text of R-phrases referred to in sections 2 and 3 | : | H335 May cause respiratory irritation. H400 Very toxic to aquatic life. R22- Harmful if swallowed. R34- Causes burns. R35- Causes severe burns. R41- Risk of serious damage to eyes. R37- Irritating to respiratory system. R37/38- Irritating to respiratory system and skin. R50- Very toxic to aquatic organisms. | |
| Abbreviations and acronyms Full text of abbreviated H statements | | ATE = Acute Toxicity Estimate CLP = Classification, Labelling and Packaging Regulation [Regulat 1272/2008] DNEL = Derived No Effect Level EUH statement = CLP-specific Hazard statement PNEC = Predicted No Effect Concentration RRN = REACH Registration Number ✓302 Harmful if swallowed. H314 Causes severe skin burns and eye H315 Causes skin irritation. H318 Causes serious eye damage. | |

Notice to reader

The data given here is based on current knowledge and experience. The purpose of this Safety Data Sheet is to describe the products in terms of their safety requirements. The above details do not imply any guarantee concerning composition, properties or performance.

Date of issue

: 2011-05-06

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NHR ORGANIC OILS

24 CHATHAM PLACE, BRIGHTON BNI 3TN (UK) TEL.(UK) 0845 310 8066 FAX.(UK) 0845 310 8068 International Tel. +44 1273 746505 Int. Fax. +44 1273 202729 EMAIL: info@nhrorganicoils.com Web Site: www.nhrorganicoils.com

Material Safety Data Sheet ORGANIC TEA TREE - Australia Melaleuca alternifolia ESSENTIAL OIL

| ESSENTIAL OIL | | |
|---|---|--|
| Info | Identification | |
| Chemical Name | | |
| CAS No. | 68647-73-4 | |
| Health | Slightly hazardous. | |
| Flammability | Will ignite at most ambient conditions. | |
| Reactivity | Stable and not reactive with water. | |
| Info | Fire, Explosion & Reactivity | |
| Flash Point | 134 F | |
| Extinguishing Media | FOG-No CO2-Yes FOAM-Yes DRY-Yes | |
| Dot Classification | COMBUSTIBLE | |
| Stability | STABLE | |
| NFPA Classification | NF | |
| Fire Fighting | FIRE FIGHTERS SHOULD WEAR POSITIVE PRESSURE SELF- CONTAINED BREATHING APPARATUS. WATER IS UNSUITABLE FOR USE ON BURNING MATERIALS BUT MAY BE USED TO COOL CONTAINERS EXPOSED TO HEAT | |
| Unusual Fire Hazard | COMBUSTIBLE | |
| Hazardous Combustible Decomposition Products | NO HAZARDOUS POLYMERIZATION WILL OCCOUR | |
| Info | Physical Data | |
| Colour & Odour | PALE YELLOW LIQUID WITH AROMATIC SPICY MEDICINAL ODOUR. MOBILE. | |

| Boiling Point | 176C | |
|------------------------------|---|--|
| Melting Point | NOT FOUND | |
| Vapour Pressure | NOT FOUND | |
| Vapour Density | NOT FOUND | |
| Water Solubility | INSOLUBLE | |
| Info | Protection Data | |
| Respiratory | NO SPECIAL PROTECTION, ORGANIC VAPOR MASK OR RESPIRATOR SHOULD BE WORN IF VAPOURS ARE FOUND TO BE ANNOYING OR IRRITATING | |
| Ventilation | | |
| Skin | USE OIL/SOLVENT RESISTANT GLOVES | |
| Eye | USE CHEMICAL SPLASH GOGGLES OFR FACE SHIELD | |
| Other | USE PROTECTIVE CLOTHING AS NECESSARY | |
| Info | Occupational Exposure Limit | |
| Threshold Limit | | |
| OSHA Permissible Limit | NOT FOUND | |
| Carcinogen | NO | |
| NTP Limit | | |
| IARC Limit | | |
| OSHA Limit | | |
| Carcinogen Notes | | |
| Info | Health Hazards | |
| Material Type | NATURAL ESSENTIAL OIL | |
| Health Hazard | LIQUID MAY BE IRRITATING TO SKIN AND EYES LOW ACUTE TOXICITY, BUT CAN CAUSE BREATHING DIFFICULTIES IF SWALLOWED. | |
| Primary Entry Routes | INHALATION, EYE CONTACT-SKIN CONTACT, INGESTION | |
| Health Hazard Notes | REMOVE TO FRESH AIR, CALL PHYSICIAN, REMOVE CONTACT LENS FLUSH EYES WITH COOL WATER FOR AT LEAST 15 MIN,CALL PHYSICIAN IF IRRITATION PERSISTS, REMOVE CONTAMINATED CLOTHING AND WASH WITH MILD SOAP AND WATER, RINSE MOUTH AND CONSULT PHYSICIAN | |
| Info | Emergency First Aid | |
| Inhalation | REMOVE TO FRESH AIR , CONTACT PHYSICIAN | |
| Eye Contact | REMOVE CONTACT LENSES, FLUSH WITH COOL WATER FOR 15 MINUTESIF IRRITATION PERSISTS, CALL A | |

| | PHYSICIAN |
|--------------------------------------|--|
| Skin Contact | REMOVE CONTAMINATED CLOTHING. WASH WITH MILD SOAP AND WATER |
| Ingestion | RINSE MOUTH, CALL PHYSICIAN/ POISON CONTROL CENTER. |
| First Aid Notes | |
| Info | Spill, Leakage & Disposal Procedures |
| Spill Procedures | REMOVE SOURCES OF IGNITION, CONTAIN SPILL WITH INERT NON CONBUSTIBLE-ABSORBANT MATERIEL AND PLACE IN APPROVED CONTAINER-THIS OIL CAN BE CONTAINED BY SAND OR SOIL AND ABSORBED BY PAPER FROM WHICH-THE OIL WILL EVAPORATE. |
| Waste Disposal | DISPOSE OF IN ACCORDANCE WITH ALL REGULATIONS. |
| Info | Handling & Shipping Procedures |
| Handling & Shipping Procedures | STORE IN FULL SEALED CONTAINERS IN A COOL DRY PLACE AWAY FROM IGNITION, HEAT OR DIRECT SUNLIGHT. FOLLOW GOOD INDUSTRIAL SAFETY PRACTICES. |
| Info | SARA 313 Chemical Breakdown |
| SARA Chemical Name | NONE |
| SARA Concentration | NONE |

MATERIAL SAFETY DATA SHEET.

1. IDENTIFICATION OF SUBSTANCE/PREPARATION & COMPANY.

Product trade name: Eumulgin S 21. Product name: Emulsifying Wax. Synonyms: SUPPLIER: The Soap Kitchen, 11a South Street

SUPPLIER: The Soap Kitchen, 11a South Street, Torrington, Devon, EX38 8AA. Emergency Tel No: 01805 622944 Emergency Contact: Mr. Richard Phillips.

2. COMPOSITION / INFORMATION ON INGREDIENTS.

| DEFINITION/BOTANICAL ORIGIN: CHEMICAL: Polyethoxylated Alcohol . | | | |
|--|---------|---------|--------|
| CAS No: | Status: | CoE No: | FEMA: |
| Additives: | FDA: | IFRA:` | RTECS: |
| Formula: | RIFM: | EINECS: | |
| INCI name: Steareth-21 | FCCIV: | | |

3. HAZARDS IDENTIFICATION:

Contains no hazardous ingredients (93/112/EEC).

| INHALATION: | Not expected to be a hazard in normal conditions of use. |
|---------------|--|
| EYE CONTACT: | Not expected to be a hazard in normal conditions of use. |
| SKIN CONTACT: | Not expected to be a hazard in normal conditions of use. |
| INGESTION: | Low acute toxicity. |

4. FIRST-AID MEASURES:

| INHALATION: | Remove from exposure. Keep warm and at rest. Seek medical attention if you feel unwell |
|-----------------------|--|
| EYE CONTACT: | Wash out eyes with plenty of water. Obtain medical attention if soreness and redness persists. |
| SKIN CONTACT: | Wash skin with soap and water. Remove contaminated clothing. Obtain medical attention for thermal burns. Wash or dry-clean contaminated clothes before re-use. |
| INGESTION: | Wash out mouth with water. Give 200-300ml (half pint) of water to drink. Keep warm and at rest. Seek medical attention. |
| ADVICE TO PHYSICIANS: | Treat Symptomatically. |

5. FIRE FIGHTING MEASURES.

COMBUSTIBLE BUT NOT READILY IGNITED.

| EXTINGUISHING MEDIA : | Use water spray, carbon dioxide, foam or dry chemical |
|-----------------------|--|
| SPECIAL MEASURES: | Avoid inhalation of fumes. Use NIOSH approved and full protective |
| | equipment when fighting chemical fire. Use water spray to cool nearby exposed to fire. |

6. ACCIDENTAL RELEASE MEASURES.

| PERSONAL PRECAUTIONS: | Wear appropriate RPE. See section 8. |
|--------------------------|--|
| ENVIRONMENT PRECAUTIONS: | Advise authorities if product has entered water course or has |
| | contaminated soil or vegetation. |
| CLEAN UP PROCEDURE | Construct temporary dikes with any suitable inert material to prevent product spreading. Stop escape of material. Allow to solidify. Transfer to suitable containers for recovery or disposal. Wash spillage area with water. |

7. HANDLING & STORAGE.

| PRECAUTIONS IN HANDLING: | Avoid contact with eyes, skin and clothing. Keep container tightly closed when not in use. |
|-----------------------------|---|
| STORAGE CONDITIONS (SOLID): | Store in cool, dry conditions at a temperature well below melting point. |
| | Do not store near strong oxidising agents which could aggravate any fire situation. Keep away from sources of ignition. |
| SUITABLE CONTAINERS: | Plastic, plastic lined or stainless steel. |

8. EXPOSURE CONTROLS/PERSONAL PROTECTION.

| RESPIRATORY PROTECTION: | Not normally required. |
|-------------------------|--|
| SKIN PROTECTION: | Rubber or plastic gloves. Protective overalls. Safety shoes/boots. |
| EYE PROTECTION: | Use safety glasses/goggles. |
| WORK/HYGIENE PRACTICES: | Wash hands with soap & water after handling. Do not eat, drink or smoke around this product. |
| OCCUPATIONAL EXPOSURE: | No Occupational Exposure Limits Assigned. |

9. PHYSICAL & CHEMICAL PROPERTIES.

| APPEARANCE: | White waxy pellets, odourless. |
|-------------------------------|--|
| BOILING POINT [°] C: | > 100 |
| FLASH-POINT °C: | > 149 |
| SOLUBILITY: | Soluble in water. Soluble in ethanol and toluene |
| POUR POINT: | 38°C (aprox) |
| PH: | No data. |
| HLB: | 15.3 |

10. STABILITY & REACTIVITY.

| REACTIVITY: | May react with strong oxidants |
|----------------------|---|
| STABILITY: | Stable in normal conditions. |
| CONDITIONS TO AVOID: | Combustion will generate hazardous decomposition components (carbon dioxide and carbon monoxide). |

11. TOXICOLOGICAL INFORMATION.

| INHALATION: | Unlikely to be hazardous by inhalation unless present as a dust. |
|---------------|--|
| INGESTION: | Unlikely to be hazardous if swallowed. Oral median Lethal Dose |
| | 2.07g.Kg (rat). |
| EYE CONTACT: | Practically non-irritant. |
| SKIN CONTACT: | Unlikely to cause skin irritation in man. Not a skin sensitiser. |

12. ECOLOGICAL INFORMATION.

| ENVIRONMENTAL FATE & DISTRIBUTION: | Soluble in water. |
|------------------------------------|---|
| PERSISTENCE & DEGRADATION: | This surfactant complies with the minimum 80% |
| | biodegradability requirement of the EEC non-ionics directive |
| | 82/242. Chemical Oxygen Demand (COD) 2.23g02/g. |
| | Biological Oxygen Demand (BOD) 28 day/COD 30%. |
| | Dissolved Organic Carbon Removal (DOC removal) 72% |
| | Modified OECD 301C test. |
| EFFECT ON EFFLUENT TREATMENT: | This product is substantially removed in biological treatment |
| | Processes. |
| WGK NUMBER: | WGK2 |

13. DISPOSAL CONSIDERATIONS.

RECOMMENDATIONS: Do not wash into drains. Do not mix with other waste materials. Dispose in accordance with all applicable local and national regulations. If necessary seek advice from local waste authority.

14. TRANSPORT REGULATIONS.

| Road (ADR/RID): | n/a |
|-----------------|-----|
| AIR (IATA): | n/a |
| SEA (IMDG) | n/a |

15. REGULATORY INFORMATION.

| LABEL REQUIRED: | No. |
|-----------------|-----|
| LABEL NAME: | |
| HAZARDS: | n/a |
| SYMBOLS: | n/a |
| RISK PHRASES: | n/a |
| SAFETY PHRASES: | n/a |
| | |

16. OTHER INFORMATION.

SHELF-LIFE

Min 12 months when stored within advised conditions.

Q.C. REQUIREMENTS.

The data provided in this material safety data sheet is meant to represent typical data/analysis for this product and is correct to the best of our knowledge. The data was obtained from current and reliable sources, but is supplied without warranty, expressed or implied, regarding its' correctness or accuracy. It is the user's responsibility to determine safe conditions for the use of this product, and to assume liability for loss, injury, damage or expense arising from improper use of this product. The information provided does not constitute a contract to supply to any specification, or for any given application, and buyers should seek to verify their requirements and product use.

APPENDIX THREE



COSHH Assessment Form

| School: History of Art | | | | File ref: | |
|------------------------|----------------------|-------------------|---------------------------|-----------|------------|
| Title of Project: | Dissertation Testing | | | Date: | 16/05/2012 |
| Room No. | 309 A | | | | |
| Building: | Robertson | Persons involved: | Student supervised by the | utor | |

Description of Cleaning of textile samples using Preventol ON-S, through immersion and spot cleaning with a vacuum suction table.

| Substance used | Quantities used | Frequency of use | Hazards identified | Exposure route |
|----------------|-----------------|---|--|-------------------|
| Preventol ON-S | <50ml | For a conservation project <3 weeks | Harmful if swallowed, causes burns, irritating to respiratory system. Corrosive. Dangerous to the environment. | Liquid and vapour |

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

Justify not using it:

What measures have you taken to control risk?

Engineering controls:

To be carried out under fume extraction: elephant trunk or fume hood.

PPE:

Lab coat, goggles, gloves and mask to be worn.

Management measures:

Supervisors are aware of the activity and available if needed.

Checks on control measures:

| Is health | s health Training requirem | |
|---|----------------------------|---------------------------------|
| surveillance No required? | | |
| Emergency procedures: | | Waste disposal: |
| Soak up spillages with an inert absorbent material and allow to vapourise in the fume cupboard before disposal. | | Disposed of as hazardous waste. |
| Name and position of assessor: Beatrice Farmer, Student | | Signature: |
| Name of supervisor (student work only): | | Signature: |
| Name of Head of School or nominee: | | Signature: |

| Brief outline | Cleaning of textile samples using Preventol ON-S, through |
|---------------|---|
| of work/ | immersion and spot cleaning with a vacuum suction table. |
| activity : | |
| | |

| Location : | Textile Conservation lab, Robertson Building, University of Glasgow |
|------------|---|
| | |

| Significant | Exposure to Preventol ON-S |
|-------------|---|
| hazards : | Spillages of Preventol ON-S |
| | Glassware- risk of breaking and injury |
| | • The vacuum suction table emits a loud noise |
| | |
| | |

| Who might | Students, staff, supervised visitors |
|---------------|--------------------------------------|
| be exposed to | |
| the hazards : | |

| Existing | Completion of COSHH form |
|------------|--|
| control | • PPE: Lab coat, goggles and gloves, mask and ear protection are |
| measures : | worn |
| | Fume extraction used |
| | • Amount of Preventol ON-S used is no more than necessary |
| | • Spillages are dealt with as determined in the COSHH form |
| | • Broken glass is disposed of using a dustpan and brush in designated broken glass bins. |
| | |

| If NO, list | Additional controls : | Action by : |
|--------------|-----------------------|-------------|
| additional | | |
| controls and | | |
| actions | | |
| required : | | |

| Completed | | | |
|-----------|------|-----------|------|
| by : | Name | Signature | Date |
| | | | |

| Supervisor : | | | |
|--------------|------|-----------|------|
| Supervisor . | Name | Signature | Date |



COSHH Assessment Form

| School: History of Art | | | File ref: | | |
|------------------------|----------------------|-------------------|-----------------------------|-------|------------|
| Title of Project: | Dissertation Testing | | | Date: | 16/05/2012 |
| Room No. | 309 A | | Student supervised by tutor | | |
| Building: | Robertson | Persons involved: | | | |

Description of procedure: Cleaning of textile samples using tea tree oil, through immersion and spot cleaning with a vacuum suction table.

| Substance used | Quantities used | Frequency of use | Hazards identified | Exposure route |
|----------------|-----------------|---|--|-------------------|
| Tea tree oil | 250ml | For a conservation project <3 weeks | Harmful if inhaled, swallowed or on contact with the skin. Flammable with a low flash point (50°C) | Liquid and vapour |

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

Justify not using it:

What measures have you taken to control risk?

Engineering controls:

To be carried out under fume extraction: elephant trunk or fume hood.

PPE:

Lab coat, goggles, gloves and mask to be worn.

Management measures:

Supervisors are aware of the activity and available if needed.

Checks on control measures:

| Is health surveillance No | Training requirem | ents: |
|--|-----------------------------|--|
| required? Emergency procedures: | | Waste disposal: |
| Soak up spillages with an inert absorb to vapourise in the fume cupboard. | pent material and allow | Tea tree oil will be disposed of as stated by local legislation. |
| Name and position of assessor: | Beatrice Farmer, Student | Signature: |
| Name of supervisor (student work only): | | Signature: |
| Name of Head of School or nominee: | | Signature: |

| Brief outline | Cleaning of textile samples using tea tree oil, through immersion |
|---------------|---|
| of work/ | and spot cleaning with a vacuum suction table. |
| activity : | |
| | |

| Location : | Textile Conservation lab, Robertson Building, University of Glasgow |
|------------|---|
| | |

| Significant | Exposure to Tea tree oil | |
|-------------|---|--|
| hazards : | Spillages of Tea tree oil | |
| | Glassware- risk of breaking and injury | |
| | • The vacuum suction table emits a loud noise | |
| | | |

| Who might | Students, staff, supervised visitors |
|---------------|--------------------------------------|
| be exposed to | |
| the hazards : | |

| Existing | Completion of COSHH form | |
|------------|--|--|
| control | • PPE: Lab coat, goggles and gloves, mask and ear protection are | |
| measures : | worn | |
| | Fume extraction used | |
| | • Amount of tea tree oil used is no more than necessary | |
| | • Spillages are dealt with as determined in the COSHH form | |
| | • Broken glass is disposed of using a dustpan and brush in designated broken glass bins. | |
| | | |

| If NO, list | Additional controls : | Action by : |
|--------------|-----------------------|-------------|
| additional | | |
| controls and | | |
| actions | | |
| required : | | |

| Completed | | | |
|-----------|------|-----------|------|
| by: | Name | Signature | Date |

| Supervisor : | | | |
|--------------|------|-----------|------|
| Supervisor. | Name | Signature | Date |



COSHH Assessment Form

| School: History of Art | | |] | File ref: | |
|------------------------|----------------------|--|---|-----------|------------|
| Title of Project: | Dissertation Testing | |] | Date: | 16/05/2012 |
| Room No. | 309 A | | | | |
| Building: | Robertson | Persons involved: Student supervised by | | utor | |

Description of procedure: Heating an emulsifier solution using a hotplate to 45°C. Cleaning of textile samples using the emulsifier, through immersion and spot cleaning with a vacuum suction table.

| Substance used | Quantities used | Frequency of use | Hazards identified | Exposure route |
|----------------------------------|-----------------|---|--|-----------------------------|
| Emulsifying Wax Eumulgin S 21 | <10g | For a conservation project <3 weeks | Low acute toxicity No other hazards identified | Solid, liquid and vapour |

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

Justify not using it:

What measures have you taken to control risk?

Engineering controls:

No engineering controls are necessary

PPE:

Lab coat, goggles, gloves and mask to be worn.

Management measures:

Supervisors are aware of the activity and available if needed.

Checks on control measures:

| Is health Training requirem | | ents: |
|---|-----------------------------|---|
| surveillance No required? | | |
| Emergency procedures: | | Waste disposal: |
| Soak up spillages with an inert absorb to vapourise in the fume cupboard. | ent material and allow | Emulsifying wax will be disposed of as stated by local legislation. |
| Name and position of assessor: | Beatrice Farmer, Student | Signature: |
| Name of supervisor (student work only): | | Signature: |
| Name of Head of School or nominee: | | Signature: |

| Brief outline | Heating an emulsifier solution using a hotplate. Cleaning of textile |
|------------------------|--|
| of work/ activity : | samples using the emulsifier, through immersion and spot cleaning with a vacuum suction table. |
| | |

| Location : | Textile Conservation lab, Robertson Building, Univeristy of Glasgow |
|------------|---|
|------------|---|

| Significant | Exposure to Emulsifier | | | |
|-------------|---|--|--|--|
| hazards : | Spillages of Emulsifier | | | |
| | • Use of hotplate- the temperature will be raised to 45°C | | | |
| | Glassware- risk of breaking and injury | | | |
| | • The vacuum suction table emits a loud noise | | | |

| Who might | Students, staff, supervised visitors |
|---------------|--------------------------------------|
| be exposed to | |
| the hazards : | |

| Existing control | Completion of COSHH formPPE: Lab coat, goggles and gloves, mask and ear protection are |
|---------------------|---|
| measures : | worn Amount of emulsifier used is no more than necessary Care taken not to touch the hotplate when switched on Hotplate temperature is raised no higher than necessary and is switched off when not in use |
| | Spillages are dealt with as determined in the COSHH form Broken glass is disposed of using a dustpan and brush in designated broken glass bins. |

| If NO, list | Additional controls : | Action by : |
|--------------|-----------------------|-------------|
| additional | | |
| controls and | | |
| actions | | |
| required : | | |

| Completed | | | |
|--------------|------|-----------|------|
| by: | Name | Signature | Date |
| | | | |
| Suparvisor . | | | |
| Supervisor : | Name | Signature | Date |

| Brief outline | Textile samples are cut up and tested using a tensile strength tester, | | |
|------------------------|--|--|--|
| of work/ activity : | which involves use of a pump to open and close the jaws before placing in the sample in between. | | |

| Location : Textile Conservation lab, Robertson Building, University of Glasgo |
|---|
|---|

| Significant hazards : | Use of scissors to cut up samples Use of tensile tester with foot operated pump to activate the jaws. |
|--------------------------|--|
|--------------------------|--|

| Who might | Students, staff, supervised visitors |
|---------------|--------------------------------------|
| be exposed to | |
| the hazards : | |

| Existing | • PPE: Lab coat, goggles and gloves are worn | | | | |
|------------|---|--|--|--|--|
| control | • No more tests are done than necessary | | | | |
| measures : | • Care taken not to touch the jaws at all times | | | | |
| | Close location of emergancy stop button | | | | |
| | | | | | |
| | | | | | |

| If NO, list | Additional controls : | Action by : |
|--------------|-----------------------|-------------|
| additional | | |
| controls and | | |
| actions | | |
| required : | | |

| Completed | | | |
|-----------|------|-----------|------|
| by: | Name | Signature | Date |

| Supervisor : | | | |
|--------------|------|-----------|------|
| | Name | Signature | Date |