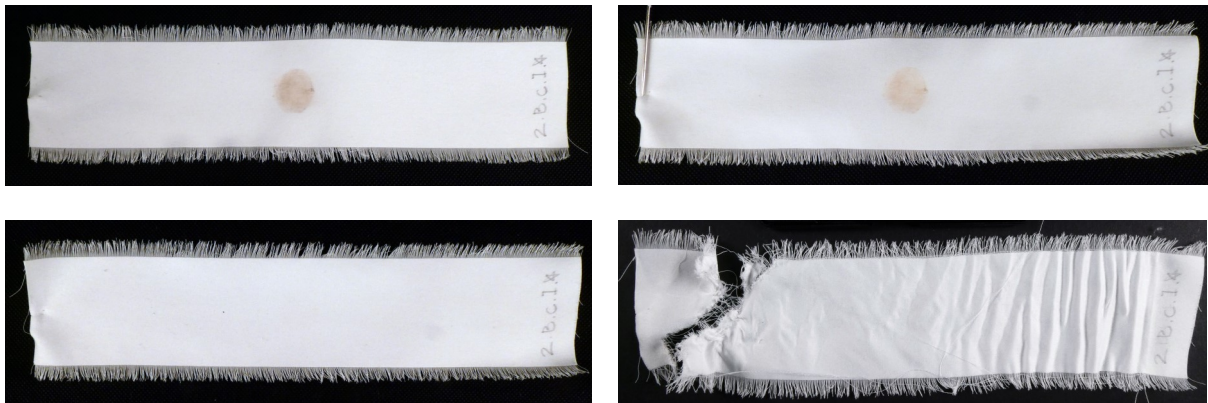




DEPARTMENT OF CONSERVATION

# A PILOT STUDY ON STAIN REDUCTION

Effects of Stains, Cleaning Agents, and Cleaning Methods  
on  
Polyester Textiles



**Karin Hindborg**

Degree project for Master of Science with a major in Conservation

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#### ABSTRACT

The thesis was an experimental pilot study that investigated efficiency and effects of stains and cleaning agents on polyester textiles. A literature review and interviews with textile conservators were used to understand the degradation of polyester fibres and to find suitable staining and cleaning agents to use in the experiments of the study. The experimental part contained aged and stained samples of polyester fabric. The samples were stained with acidic coffee, alkaline ashes and fatty synthetic sebum and cleaned with Orvus WA Paste, Dehypon LS54, Gall soap, triammonium citrate and ethanol. Two cleaning methods were used: cotton swabs dipped in cleaning agent and cleaning agent dripped through the sample on a suction table. All cleaning agents and cleaning methods were effective for stain reduction on some types of stains. No cohesive changes in degradation caused by the different staining agents or cleaning agents were shown in the results from tensile testing.

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# Table of Contents

ACKNOWLEDGEMENTS .....	11
1. INTRODUCTION .....	13
1.1 Background.....	13
1.2 Previous Research .....	14
1.3 Research Questions .....	15
1.4 Objective and Aim .....	15
1.5 Methodology .....	16
1.6 Delimitations .....	16
1.7 Theoretical Approach and Ethical Issues .....	17
2. LITERATURE REVIEW .....	19
2.1 Textile Fibres .....	19
2.2 Man-made Fibres .....	19
2.3 Polyester Fibres .....	20
2.3.1 Polyethylene Terephthalate (PET).....	20
2.3.2 Characteristic Properties of Polyester Fibres .....	21
2.3.3 Degradation of Polyester Fibres .....	22
2.4 Dirt and Staining .....	23
2.5 Cleaning Methods.....	24
2.6 Spot Cleaning.....	25
2.7 Cleaning Agents .....	27
2.7.1 Anionic surfactants.....	30
2.7.2 Non-ionic surfactants .....	31
2.7.3 Sequestering/Chelating Agents.....	31
2.7.4 Solvents.....	32
2.7.5 Enzymes .....	32
2.8 Analytical Methods .....	33
2.8.1 Colour Measurements.....	33
2.8.2 Infrared Spectroscopy .....	34
2.8.3 Tensile Testing.....	34
3. INTERVIEWS .....	37
3.1 Results from Interviews .....	38
4. SELECTED MATERIALS.....	39
4.1 Used Staining Agents .....	39
4.1.1 Coffee .....	39
4.1.2 Ashes.....	39
4.1.3 Sebum .....	40
4.2 Tested Cleaning Agents .....	40
4.2.1 Orvus WA Paste.....	40
4.2.2 Dehypon LS54.....	40

4.2.3 Gall Soap .....	41
4.2.4 Triammonium Citrate .....	41
4.2.5 Ethanol .....	41
5. MATERIALS AND METHODS OF EXPERIMENTS AND ANALYSES .....	42
5.1 Experimental Design .....	42
5.2 Test Fabrics .....	44
5.3 Sampling .....	44
5.4 Staining Agents .....	45
5.5 Staining .....	45
5.6 Accelerated Ageing .....	47
5.7 Cleaning agents .....	47
5.8 Stain Removal .....	48
5.9 Photography .....	50
5.10 Colour Measurement .....	50
5.11 Infrared Spectroscopy .....	51
5.12 Tensile Testing .....	51
6. RESULTS .....	53
6.1 Stain Removal .....	53
6.1.1 Cleaning of Un-aged Stains using Cotton Swabs .....	53
6.1.2 Cleaning of Un-aged Stains on a Suction Table .....	55
6.1.3 Cleaning of Aged Stains using Cotton Swabs .....	56
6.1.4 Cleaning of Aged Stains on a Suction Table .....	59
6.2 Colour Measurement .....	61
6.2.1 Un-aged Coffee Stains .....	61
6.2.2 Aged Coffee Stains .....	61
6.2.3 Un-aged Ash Stains .....	63
6.2.4 Aged Ash Stains .....	63
6.2.5 Un-aged Sebum Stains .....	65
6.2.6 Aged Sebum Stains .....	65
6.2.7 Aged Polyester .....	66
6.2.8 $\Delta E$ .....	66
6.3 Infrared Spectroscopy .....	68
6.4 Tensile Testing .....	69
6.4.1 Coffee .....	69
6.4.2 Ashes .....	71
6.4.3 Sebum .....	73
6.4.4 Aged Samples .....	74
6.5 Summary of Results .....	78
7. DISCUSSION .....	80
7.1 What causes degradation of polyester fibres and in what manner do they degrade? Do different types of stains degrade polyester further? .....	80
7.2 What will be the effect if a stain is left to age? .....	81
7.3 What cleaning agents are effective and suitable for cleaning polyester textiles? .....	82



7.3.1 Coffee .....	82
7.3.2 Ashes .....	83
7.3.3 Sebum .....	83
7.4 What cleaning methods are suitable for stain removal on polyester textiles? .....	85
8. CONCLUSIONS .....	88
8.1 Further Research .....	89
9. SUMMARY .....	91
10. LIST OF REFERENCES.....	93
11. LIST OF ILLUSTRATIONS .....	99
11.1 List of Figures.....	99
11.2 List of Tables .....	101
11.3 List of Abbreviations .....	101
APPENDICES.....	102
Appendix 1. Answers to Interview Questions.....	107
Appendix 2. Measurements of temperature and RH .....	104
Appendix 3. Tensile Testing Instrument Report .....	108
Appendix 4. Colour Measurements .....	117



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*There must be a beginning of any great matter,  
but the continuing unto the end until it be thoroughly finished  
yields the true glory.*

Francis Drake 1587

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# 1. Introduction

## 1.1 Background

Much of the Swedish textile cultural heritage can be found in the Swedish church. By law, the church has a responsibility to preserve and care for the textiles. This is most commonly done by textile conservators.

The textiles are often made of materials such as silk, wool or linen which are embroidered or decorated with symbolic images. As most textiles are in use it is not uncommon that they become stained and dirty. This research will investigate methods to perform localised stain removal to be used instead of a full wet cleaning. It is important to find methods that can be used for local stain removal, so that the textile does not need to be deconstructed before removing a stain. A deconstruction is very time consuming, which makes it costly, historical traces, such as the thread used for stitching, will be removed and the mechanical action might further degrade the textile. A deconstruction can also be considered unethical, as it is such a big intervention.

The thesis was a pilot study that investigated cleaning solutions for polyester fabric and a few types of stains. The conclusions from the study will be used to map out a model for further research about different textile fibres in combination with different types of stains and cleaning agents.

In textile conservation much research has been done on natural textile fibres, but not as much on man-made fibres. As man-made textile fibres are starting to be found in museums as well as in the Swedish churches, it is important that more research is performed so that we can find suitable ways to preserve those textiles as well.

One of the fibres that can be found in textiles in museums and in the Swedish church is polyester. How does polyester degrade? How does it behave in combination with different types of dirt? What types of cleaning agents work well with polyester? Those are questions that need to be investigated in order to conserve polyester textiles in a suitable way.

The research will start answering the question of how to treat polyester textiles with a conservation point of view. It will give some insight into how polyester relates to different types of stains as well as what cleaning agents might be suitable for treating polyester.

## 1.2 Previous Research

A big part of textile conservation concerns cleaning or not cleaning the textile in need of conservation. Much research has been done concerning suitable methods and cleaning agents for cleaning historical textiles. This study is focused on stain removal rather than full wet cleanings, and research concerning this question is not as extensive as research on full cleaning of an object. However, there are some research done on the matter.

In 1993 Tímár Balázs and Mátéfy published a paper called *Effect of Stains and Stain Removal on Historical Textiles*, where they investigated the relationship between stain removal methods and degraded fibres underneath the stains. They created mock-ups and applied different stain removal methods and measured colour change, whiteness and tensile strength to clarify how the different stain removal methods affected the stain as well as the textile.

France (2003) also writes about the importance of understanding the mechanical degradation of fibres, and not only use fading of colour as a measurement of degradation. She discusses the possibilities of using tensile strength measurements to understand the mechanical and physical properties of the textile. She describes the possibility to use loose, single threads from an actual object to obtain information of its current state.

In 2004 Liljedahl wrote a bachelor thesis called *Wine Staining on Ecclesiastic Textile*. She identified solvents that might be used in the removal of stains from wine on historical ecclesiastical textiles made from silk, rayon and cellulose acetate.

Kissa wrote an article called *Coffee Stain on Textiles. Mechanisms of Staining and Stain removal* in 1995 where he discusses coffee staining of different textile fibres as well as how to remove the stains.

Paper and textiles have many things in common as they can be based on the same/similar fibres. Dwan (2015) writes about the use of ammonium citrate to remove stains on paper and its benefits. She explains that ammonium citrate has a neutral pH and does not bleach or lighten the area around the removed stain.

During the last 100 years, many new textile materials have been developed. Those man-made fibres have started to be found in historical textiles, but they are not as common as natural fibres. Hence there has not been as much research conducted concerning their degradation and suitable conservation treatments as has been done with natural fibres.

Hunter, White, Cohen and Biermann wrote in 2000 *A Materials Ageing Problem in Theory and Practice* which discusses the mechanism of polyester ageing by hydrolysis. The article provides an approach for modelling the ageing behaviour of commercial polyester.

A study on degradation of polyester fibres was conducted by Cubric, Cubric, Krizmancic and

Kovacevic in 2022. The investigation was focused on topography, thickness, SEM analysis, moisture management, elongation at break, bursting force, and FTIR analysis.

Recently some research has been investigating man-made and synthetic fibres with a conservation perspective. In 2001 Rebecca Tinkham wrote a master thesis called *Soil Removal and Redeposition on Cotton, Nylon and Polyester Fabrics Wet-Cleaned with Anionic and Nonionic Surfactants*. The research examined conservation wet-cleaning of synthetic fabrics, where soil removal and redeposition were indicated by total colour change ( $\Delta E$ ).

### 1.3 Research Questions

Stains can often be found on historical textiles. Some of them should stay there as historical evidence, but others need to be removed if possible, as they might expedite the degradation of the textile as well as making it aesthetically unpleasing.

This thesis was investigating suitable cleaning agents and cleaning methods for stain removal on polyester fabric.

The following issues were addressed:

- What causes degradation of polyester fibres and in what manner do they degrade? Do different types of stains degrade polyester further?
- What will be the effects if a stain is left to age?
- What cleaning agents are effective and suitable for cleaning polyester textiles?
- What cleaning methods are suitable for stain removal on polyester textiles?

### 1.4 Objective and Aim

The objective of this thesis was to, through literature studies, interviews, experiments and analyses, gain an understanding of the degradation process of polyester and to find suitable cleaning methods and cleaning agents to use for stain removal on polyester fabric.

The aim was to create means to map out a model for further research of different textile fibres in combination with different types of stains, different cleaning agents and stain removal methods that can be used in a larger project about stain removal on textiles found in the Swedish church.

## 1.5 Methodology

The thesis contains four comprehensive methods to help answer the research questions in section 1.3. A literature review was used to gain a deeper understanding about the polyester fibre and its degradation, about different types of dirt and stains, different cleaning agents and cleaning methods for spot cleaning as well as about the different analytical tools used in the experiments.

Interviews with textile conservators from Sweden were used to find out how they work with spot cleaning and what cleaning agents they use.

Experiments were performed to investigate how different types of stains affect polyester fabric, the effect of different types of cleaning agents and the effect of different types of cleaning methods.

Analysis with infrared spectroscopy, colour measurements and testing of tensile strength were performed to help answer the research questions.

Experiments and most of the analysis were performed at the Department of Conservation at the University of Gothenburg. The tensile strength testing was performed at the Swedish National Heritage Board in Visby.

## 1.6 Delimitations

There are many types of textile fibres, of dirt, cleaning agents and spot cleaning methods. This study was limited to using the textile fibre polyester, as it is a modern material, where there is a need for conservation research. It is also a material that has started to be used more than earlier in ecclesiastic textiles in the Swedish churches, and can therefore be in need of conservation spot cleaning.

The study was limited to using three types of dirt. As different pH can have a large impact on textile fibres, acidic (coffee) and alkaline (ashes) dirt were chosen. Fatty and greasy stains from wear are very common on historical and ecclesiastic textiles. Fat is absorbed by the polyester fibre due to its hydrophobic character, hence it was decided to investigate a greasy (sebum) stain as well.

Five types of cleaning agents were used in the study. They were limited to one anionic (Orvus WA paste), one non-ionic (Dehypon LS54), one soap with ox-gall (Gall soap) and one sequestering agent (triammonium citrate). As it often is recommended to treat greasy stains with solvents, the sebum stains were treated with a solvent (ethanol) instead of a sequestering agent.



The study was limited to using two types of cleaning methods, one with cleaning agent and mechanical action through rolling of cotton swabs, and the other with cleaning agent dripped through the textile using a suction table.

The amount of replicas to each sample was considered. To have enough space to age all replicas at the same time, it was decided to limit the number of replicas to three of each sample.

The only available climate chamber for the quantities of samples examined in this study, had the ability to control heat and moisture, not UV-light. The ageing was limited to using the available equipment.

## **1.7 Theoretical Approach and Ethical Issues**

When considering cleaning historical textiles there are many aspects to take into account. Reversibility has for a long time been considered a key criteria in conservation treatments. The Burra Charter 2013 says:

15.1 *Changes may be necessary to retain cultural significance, but are undesirable where it reduces cultural significance.*

15.2 *Changes which reduce cultural significance should be reversible, and be reversed when circumstances permit.*

The Burra Charter

The Australia ICOMOS Charter for Places of Cultural Significance, 2013

Minimum intervention is a phrase that has been used over the last 60 years and was articulated by Cesare Brandi in 1963. In the 1980s minimum intervention became prominent as an ethical approach in conservation. The definition of minimal intervention has been debated and hard to decide upon as it is not a clear statement. 'The minimum intervention to achieve what?' Caple suggests that minimum intervention must be defined for a specific object over a specified time and at specified conditions (Caple 2000, p. 65). Munoz Vinas disagrees with using the phrase as a true minimal intervention would be to leave the object alone. The idea of minimum intervention can not help us determine the safe margins for a conservation treatment (Richmond & Bracker 2009, pp. 49-50). The Canadian Code of Ethics says:

- II. *In the conservation of cultural property, all actions of the conservation professional must be governed by an informed respect for the integrity of the property, including physical, conceptual, historical and aesthetic consideration.*
  
- IV. *The conservation professional shall seek to prevent damage and deterioration to a cultural property under his/her care...*

Code of Ethics

the Canadian Association for Conservation of Cultural Property  
and of the Canadian Association of Professional Conservators, 2009

The idea of minimum intervention springs from previous over cleaning, to invasive inpainting, restoration gone further than is now deemed necessary. As the Canadian Code of ethics expresses, all conservation action must be governed by an informed respect for the object, and the conservator should seek to prevent damage and deterioration of the object.

Ethical issues are very important to consider. The thoughts about reversibility being a key criteria is also debated, as many things you do as a conservator are irreversible. Cleaning is an irreversible process and should only be performed after weighing the benefits against the drawbacks. One reason not to clean textiles is that the cleaning will, or might be damaging to the object or to decorations attached. Eastop & Brooks (1996) explains that evidence of historical, cultural or technological value can be destroyed through cleaning of the specific object.

On the other hand, dirt can contribute to the degradation of textile fibres (Lundwall 2003, p. 110). The dirt can work as a catalyst and in that way hasten the degradation of the textile, hard and sharp particles can cut through weak fibres and some substances can destroy the area where they are deposited (Landi 1992, p. 79). Agnes Geijer explains that a water cleaning, done skilfully, can give extraordinary results as the cleaning removes dirt as well as swells the textile fibres so that they straighten, becomes more flexible and regains their natural sheen. Original shape and flattening can be regained without adding heat, if they are dried at a smooth surface (Geijer 2011, p. 80).

Ecclesiastical textiles in use are an important part of the sacred experience received during mass. Their appearance should be worthy and dignified. They are protected by the Historic Environment Act and should be properly stored and conserved (Historic Environment Act, Chapter 4, Section 6, SFS 2019:864 ). In this case, cleaning is often necessary to retain cultural significance and to prevent damage and deterioration. The cleaning is done with respect for the historical and aesthetic integrity of the ecclesiastic textile.

## 2. Literature Review

### 2.1 Textile fibres

Almost all textile fibres are polymers. The word polymer has its origin in the Greek words *poly*, meaning *many*, and *meros*, meaning *parts* which refers to the many monomer parts in a polymer (Jensen 2008). In nature there are many types of fibres, but only a few of those can be used to create yarn and textiles, due to its spinnability. All textile fibres are very long in comparison to their diameter, they are all anisotropic with properties of different value in width vs length (Wiklund 1984, p. 11).

Textile fibres can be classified as natural fibres and man-made fibres. In the classification natural fibres we find cellulose fibres divided in bast fibres (linen, hemp, etc), leaf fibres (sisal, banana, etc) and seed fibres (cotton, kapok, etc), and protein fibres (wool, silk, etc). Man-made fibres are classified as modified natural polymers/regenerated fibres based on cellulose (viscose, acetate, etc), protein (casein, zein and ardil) and natural rubber, as synthetic polymers (polyamide/nylon, polyester, etc) and as miscellaneous fibres such as asbestos, glass and metal fibres (Tímár-Balázs & Eastop 1998, p. 4).

### 2.2 Man made fibres

The idea of producing man-made fibres was introduced by Robert Hooke (1635-1703) with thoughts about the possibility to imitate the silkworm by making *an artificial glutinous composition [and] to find very quick ways of drawing it out into small wires for use* (Woodings 2001, p. 1). Most man-made fibres are produced as long, thin filament fibres. Just like Hooke envisioned, the production is inspired by the silkworm. The liquid material is pressed through a spinneret to produce long, thin filament threads, just like silk fibroin is pressed through the silkworm's spinous glands. Yarn can be produced as monofilament yarn with just a single filament, or as multi filament yarn, composed of a bundle of, often very fine filaments, twisted together. The third type of yarn consists of staple fibres, spun to yarn. A staple fibre is filament fibre cut into appropriate length and then spun into yarn (Wiklund 1984, p. 91).

Regenerated cellulosic fibres are made from natural polymers, such as wood pulp and cotton linters. The different polymers are dissolved in chemicals and become a viscous liquid (hence the name *viscose*). The liquid is pressed through a spinneret, thus producing filament fibres. Earlier regenerated fibres were often called artificial silk (Fjaestad & Riksan-tikvarieämbetet 1999, p. 134). Charles Friedrich Schönbein discovered in 1846 that nitric

acid could nitrate cotton to gun cotton. This led to the first successful attempt to create a regenerated fibre by George Audemars of Lausanne, Switzerland in 1855. The fibre was made from nitrated inner bark of mulberry dissolved in alcohol and ether which was drawn into the air. In 1884 Joseph Swan presented *some samples of artificial silk* to the Society of Chemical Industry. The first factory to produce artificial silk was started in 1892 by Count Louis-Marie-Hilaire Bernigaud, Comte de Chardonnet (Woodings 2001, pp. 2-3). In modern days there are two main types of regenerated cellulose fibres; one that consists of pure cellulose where viscose, modal and cupro fibres can be found, the other one is cellulose acetate with acetate and triacetate fibres (Wiklund 1984, p. 91).

The polymers of natural fibres and regenerated fibres are natural and found in the origin of the fibre, the cellulose, or the protein, but the polymers of synthetic fibres need to be created. This is done by polymerisation where petroleum, carbon, lime, salt, water, air and/or other materials react with one another at high temperatures and under high pressure. The polymer, which is always a thermoplastic when used as a textile fibre, will then be melted or dissolved in a solvent, and transformed to fibres by melt spinning, dry spinning, or wet spinning (Wiklund 1984, p. 97).

Synthetic fibres are ordered depending on how they are built chemically. Common synthetic fibres are polyamides, polyesters, acrylics, olefins such as polyethylene and polypropylene fibres (Tinkham 2001, p. 3). There are some common characteristics of most synthetic fibres. They all have a low ability to regain moisture. This is the reason they often become static which makes them attract dirt. Synthetic fibres are very strong and often have a high elasticity which makes them wrinkle resistant. They are very malleable as they are thermoplastic. Folds can be created that will stay even though the textile has been washed in water (Wiklund 1984, p. 99).

## **2.3 Polyester fibres**

### **2.3.1 Polyethylene Terephthalate (PET)**

Polyester was discovered around 1847 by the Swedish chemist Berzelius. Industrial production started 1941 and unsaturated polyester, which is used in drink containers, food packaging or electrical insulators were developed in 1942 (Shashoua 2008, p. 20).

All polyester polymers contain ester linkages in the polymer chain. An ester is a salt formed via a reaction between an acid and an alcohol. Polyesters mean many organic salts (Gohl & Vilensky 1980, p. 112). There are different types of polyesters, but the one most used to create textile fibres is polyethylene terephthalate (PET). The first polyester fibre was developed in 1941 and is known as Terylene. PET is a saturated polyester. The saturated polyesters are strong, hard, has a high crystallinity and are thermoplastic. A thermoplastic softens when heated and hardens when cooled. PET is created by an esterification reaction be-

tween ethylene glycol and terephthalic acid where water is condensed (Shashoua 2008, p. 55).

Polyesters have branched chain structures, resulting from side reactions during synthesis. As they are branched, they pack less effectively and have a higher molecular weight than linear polymers. (Shashoua 2008, p. 93).

PET is a semi-crystalline polymer composed of both amorphous and crystalline regions (Jog 1995. p. 531). High tenacity polyester can have a crystallinity of 80-90% (Timár-Balázs & Eastop 1998, p. 11).

Most polyesters are thermoset but polyethylene terephthalate is thermoplastic. The reason that PET is thermoplastic is that it is not crosslinked in the manufacturing process. The glass transition temperature,  $T_g$ , lies between 73-80°C. Melting temperature,  $T_m$ , lies between 245-265° C. It retains its mechanical properties up to 175° C. Elongation at break is 30-300% (Shashoua 2008, pp. 244-245). There is a universal recycling code for PET with the number 1 (Figure 1). The code is an explanation of what the object is made of (Wikipedia 2023).

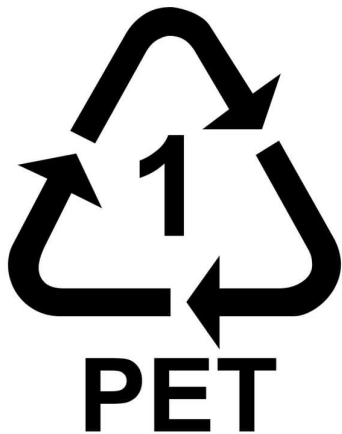


Fig. 1. The universal recycling code for for PET. Image from <https://ismwaste.co.uk/images/help/plastic1-pet.jpg>.

### 2.3.2 Characteristic Properties of Polyester Fibres

Polyester has a density of 1,39 g/cm<sup>2</sup>. The diameter of a polyester fibre ranges between 12µm to 25µm. Polyester has no specific microscopic appearance, apart from being very regular and featureless. The magnified image can be likened to that of a glass rod (Gohl & Vilensky 1980, pp. 112-113).

Because of the crystalline nature and uniform fibre structure of polyester, the tensile

strength is higher than for natural fibres. Another consequence is that the elongation at break point is smaller. The high crystallinity makes the fibre stiff and hydrophobic. As the polyester polymer has great alignment, the moisture absorption of polyester fibres is lower than that of natural fibres. Because of this low moisture regain, polyester often develops static electricity. It absorbs 0,4-0,8% moisture at 65% RH and 20°C, which is a very small amount (Timár-Balázs & Eastop 1998, pp. 57, 60-61).

Because of the crystallinity of polyester, the fibres are strong to very strong. When polyester is wet, the tenacity remains unchanged because of the hydrophobic and crystalline polymer system which resists entry of large amounts of water molecules. Polyester can also be characterised by high resistance to stretch, toughness and stiffness, crease resistance and shape retention. Because of its hydrophobic character, it absorbs fats, greases and oily soiling. This oleophilic nature (affinity for oil rather than water) of polyester creates considerable laundering problems. The only way to effectively remove greasy soiling is with the use of organic solvents (Gohl & Vilensky 1980 p.p. 115-116, Timár-Balázs & Eastop 1998, p. 61).

To identify polyester fibres, a burn test can be performed. It is difficult to ignite polyester. The fibre burns briefly with a shiny, yellow orange, sputtery, sooty flame and has a sweet smell. The burned material forms a hard, shiny, black bead (Reddy-Best & Ordoñez 2022, p. 24).

### 2.3.3 Degradation of Polyester Fibres

The degradation of PET is mainly caused by UV light, oxygen, moisture and temperature (Asadi, Uhlemann, Stranghoener & Ulbricht 2021, p. 4). The proportions of amorphous versus crystalline regions are crucial in deterioration. It is mainly in the amorphous regions of the fibre that the polymer chain is accessible to deteriorating agents. As polyester has an estimated crystallinity of 65-80% it has a good resistance to chemical degradation (Timár-Balázs & Eastop 1998, p. 11).

PET is impervious to water but has low permeability to oxygen. The ester group (-COO-) imparts susceptibility to hydrolysis and chain scission (Shashoua 2008, pp. 244-245). UV light can cause photolysis of PET fibres, especially if they are un-pigmented. Atmospheric oxygen (O<sub>2</sub>) can cause chain scission and accelerate crosslinking. PET is not sensitive to hydrolytic degradation at ambient temperatures, but above T<sub>g</sub> (67-80°C) the ester bonds can be hydrolytically cleaved causing an increase of crystallinity. Acid and alkaline conditions, and the intensity of them, increase the rate of hydrolysis. Degradation of PET due to temperature starts in the manufacturing process where the melting temperature is 250-260°C. Thermal degradation leads to accelerated chain scission (Asadi et al. 2021, pp. 4-7).

PET can degrade both visibly and invisibly. Visible degradation is shown through discolouration or cracks. Yellowing is caused by photolytic degradation and haze formation is induced by hydrolytic degradation. When light and moisture are coupled, the effects are more serious. The invisible degradation will be shown if the textile is put under additional stress, and ultimately will become visible through stress-induced cracks (Asadi et al. 2021, p. 7).

Polyester can transport charge via its surface, as the oxygen atoms in the ester linkages have mobile electrons. The polar groups can attract moisture or statistically charged contaminants, ex dust, on the surface, which have the potential to accelerate hydrolytic breakdown (Shashoua 2008, p. 102).

The polyester polymer fibre is resistant to acidic hydrolysis but alkaline conditions may hydrolyse the polymers at their ester groups. The alkaline hydrolysis is restricted to the surface of the fibre due to its crystalline nature (Gohl & Vilensky 1980, p. 117). According to Asadi et al.(2021, p.7), the rate of hydrolysis is dependant on the intensity of acidic or alkaline conditions, and is increased in both conditions.

## **2.4 Dirt and staining**

Many different types of dirt can be found on textiles and in textile fibres. They can cause chemical and physical damage to the textile if not removed (Tinkham 2001, p. 23). Staining agents are adsorbed on the fibre and diffuse into the interior of the fibres. In “Chemical Principles of Textile Conservation” by Ágnes Tímár-Balázsy and Dinah Eastop (1998, pp. 157-159) 4 different ways to classify soiling are described: according to source, harmfulness to textiles, the form of the dirt and methods of removal. The sources of dirt can be functional use or handling, the site of excavation/recovery and airborne pollutants. Examples of dirt that can be harmful to textiles are solid dirt which can cause tension and mechanical damage and deterioration products of the fibre themselves which can cause discolouration of textile, an increase of light sensitivity and acid hydrolysis. There are many other types of dirt that also can be harmful to textiles. Dirt can have the form of particles, of molecular soiling or of soiling that forms a large mass on textile. What method to use for removal of dirt is dependent on whether the dirt is loosely bound to the surface (surface soiling) or if it remains on the textile after surface cleaning (bonded soiling).

Dirt can adhere to textiles through mechanical entrapment or through different types of chemical bonds. The more contact areas where dirt and fabric can create bonds, the stronger the adhesion is between the soil and the textile (Tinkham 2001, p. 24). Dirt is attached to textiles by secondary bonds that provide the adhesion between the dirt and the fibre molecules. To be able to separate dirt from textiles, the adhesion between the dirt and the fibre molecules must be diminished. The cohesion between the dirt it self and

the primary and secondary bonds between particles of dirt that cannot be separated into molecules, must also be diminished (Timár-Balázs & Eastop 1998, p. 159).

There are different factors that determine the adhesion of the dirt to the textile where polarity is the most important factor to consider. Other factors are the ambient conditions for both dirt and textile, the morphology of the fibre as well as of the dirt, the consistency and age of the dirt and the structure of the yarn and fabric.

Non-polar dirt, such as carbon black, waxes with a paraffin base, and dust, are bound to textiles by Van der Waals forces. Sometimes this type of dirt may be removed by surface cleaning. It might also be removed when wet cleaning, even though the dirt does not dissolve in water. It can be done as an electric double layer will be formed at both the interface of the water/particle and water/fibre when a textile is immersed in water. The double layer creates a repulsion force that promotes the removal of non-polar dirt, even though it is not water soluble. Ionic and polar soils as well as mixed polar and non-polar soiling are adhered to textiles by Van der Waals forces, dipole secondary bonds and hydrogen secondary bonds (Timár-Balázs & Eastop 1998, pp. 160-161).

Many stains are either acidic or alkaline. More than half of all stains can be solved in water; warm water is more effective solvent than cold water. The best water to use for stain removal is distilled water. Glycerine can be used to lubricate and dissolve old, hard stains. Isopropanol can be used instead of ethanol, as it dissolves some stains better. Soap dissolves many stains. An example is Marseille soap, also known as Spanish silk soap. It is important to have absorbing materials at hand when removing stains. The materials can be cotton fabric, blotting paper, filter paper, unglued cellulosic paper or cellulose pulp. A stain should, if possible, be dissolved, not be rubbed off, otherwise the fibres can be affected (Hylander 1956, pp. 34, 56, 71).

## **2.5 Cleaning Methods**

Cleaning is one of the most common operations in the conservation of cultural heritage. It has the benefits of both restoring the aesthetic appearance of an artefact, as well as having an active role in the preservation of it. Dirt and soiling do not only alter the appearance of an object, but they also induces or takes part in surface degradation processes, processes that might irreversibly alter the artefact (Baglioni, Poggi, Chelazzi & Baglioni 2021, p. 2).

To remove dirt from textiles, the adhesion forces between textile and dirt must be diminished. The forces can be diminished by mechanical action, such as using a brush or a vacuum cleaner or a surfactant, by swelling the dirt and then removing it mechanically and by dissolving the dirt.



Surface cleaning is also sometimes called dry cleaning, which can be misleading, as commercial cleaning with chemicals is called dry cleaning as well. When working as a conservator we are talking about dry cleaning as opposed to wet cleaning (Timár-Balázs & Eastop 1998, p. 175). Surface cleaning removes loosely bound particles, such as loose dirt, grit and loose fibres and dust, from the surface or from within the textile.

There are different dry methods to clean a surface. A brush can be used to loosen dirt. The dirt can then be picked up by a vacuum or a soft cloth. Suction can be used straight on an object, depending on the strength of the textile, or as a combination of a vacuum with a brush attachment. To prevent loose materials from the object being sucked into the vacuum, a net can be placed on top of the textile or fastened around the nozzle of the vacuum. It is also possible to work the other way around, by using some type of blower to blow away the dirt from, for example, around beadwork or embroidery (Landi 1992, pp. 79-80). When vacuuming a textile the sucking forces must be stronger than the adhering forces of the dirt to the textile. A way to achieve this is to create a free flow of air by creating an air gap in between the textile and the surface it is placed on (Benjaminson 2012, p. 129).

When wet cleaning, the interaction between water, surfactant, textile, dirt and agitation is what removes the soiling (Tinkham 2001, p. 25). When wet cleaning historical textiles the fragility of the textile must always be considered. If there are areas that are more fragile than the rest of the textile, extra precaution needs to be taken, for example, those areas can be sewn into a net. It is not suitable to wash historical textiles in a washer, the washing should be done by hand for more control and less friction.

When washing a textile there is an alternation between soaking and rinsing. This can be done in different ways; either by using a flow of water across a flat, sloping surface for rinsing or by using some kind of tank with a depth of water (Landi 1992, p. 80). If you have access to a wash table that can be raised in one end, both methods can easily be used.

The agitation needed to remove dirt can be done by vibrating the water with the hand for very sensitive textiles, by creating a stronger flow of water through the textile by using a sponge or a paint roller, or by sucking the water through the textile by using a sucking/vacuum table or a wet vacuum cleaner.

## **2.6 Spot Cleaning**

Some stains can be removed with surface cleaning, but that is often not enough to remove a stain. A stain might need wet methods, chemical methods or the use of solvents to be removed or reduced. Stain removal often includes mechanical action in combination with organic solvents, acid and alkaline solutions, bleaching agents and enzymes. Stain re-

removal in textile conservation needs to be done with care and caution as the materials often are sensitive and degraded. Stain removal is often performed on padded absorbent materials or on suction tables. It is often employed as a pre-treatment followed by wet or solvent cleaning of the whole object (Timár-Balázszy & Eastop 1998, p. 237). Sometimes it is not desirable or even possible to wet clean an object. The condition might be too poor for wet cleaning as the fragile artefact might disintegrate by the weight of the water absorbed by the fibres, different parts might be sewn together and to wet clean the object, the parts would need to be taken apart, or there might be risks of dye bleeding and staining of the object (Sachdeva, Suri, & Bhagat 2020, p. 1293).

It is important to investigate the stain, fibres and dyes and their sensibility to different cleaning agents before starting the treatment. Stained fibres may be in a very deteriorated condition. The edges of a hard or stiff stain may have suffered from mechanical degradation. Especially fatty and oily stains can benefit from a lubricating pre-treatment. This can be done with glycerine/glycerol, pure mineral oils and detergents with long hydrophobic chains (lubricating properties). It is often important to thoroughly rinse the area after cleaning a stain, as there otherwise might be dissolved materials from the stain and the stain removal agent left in the fabric (Timár-Balázszy & Eastop 1998, p. 237).

Stains can sometimes be removed by placing the textile on an absorbing material and swab the stain, using cotton swabs dipped in a cleaning agent (Timár-Balázszy, Csányi & Máréfy 1993, p. 332).

It is hard to remove stains without leaving either a cleaner patch or creating a ring mark. Ring marks are created as soluble dirt is drawn towards the edges of the wet area by capillary action, as the solvent dries faster around the perimeter of the wet area.

A suction table can be used to minimise the risk of creating ring marks. When using a suction table, the procedure is to place absorbent paper or towelling with a top layer of washed cotton fabric on the perforated area of the table/box and cover that with the stained textile. The cleaning agent used is slowly fed into and through the stain with the vacuum on so that the flow of air helps limit the spread of the liquid and thus prevent ring marks. The loosened stain is carried into the absorbent paper, which must be changed frequently. Warm air blown around the edges can further help prevent the formation of ring marks (Landi 1992, p. 93).

Poultices and gel can be used for local introduction of a cleaning agent. Poultices often use diffusion or capillary action. It first moistens the substrate through diffusion, and then, when starting to dry, it pulls out the moisture, including dissolved dirt, from the substrate using capillary action (Schmitt & Foskett 2016, p. 157).

Water soluble stains can be reduced or removed by the mist from an ultrasonic humidifier or a preservation pencil, where the mist can be heated to a desired temperature. The mist can also be used to soften the fibres in areas of old ring marks. A mixture of deionized water and IMS (denatured alcohol) can be a useful solvent to try first (Landi 1992, p. 93).

## 2.7 Cleaning agents

A wet cleaning can be performed with only water. Different pH can give different results, but as the pH also affects the fibres it is important to be aware of what fibre is being treated, and not to make the water very acidic or alkaline. Cellulose is sensitive to acids but not so much to alkalis. Wool resists acid hydrolysis, but has a higher sensitivity to alkaline conditions (Timár-Balázs & Eastop 1998, pp. 28-29, 53). Recommendations are given that the pH of a washing solution never should exceed pH 8,5, even for cellulosic fibres. The solution for cleaning protein fibres should be kept neutral or slightly acidic (Landi 1992, p.69).

Detergents can be added to the water to help with the cleaning of textiles. Other additives that can be used for cleaning historic textiles are sequestering agents, soil carriers and enzymes. When using enzymes you usually denature them at the end of treatment to make them inactive. This can be done by changing the pH or by adding sequestering agents (Timár-Balázs & Eastop 1998, p. 235).

Water is a very important part of cleaning textiles, as it serves both as a solvent and as a medium for the cleaning process. Water is the most polar liquid, and it dissolves many types of dirt. It can dissolve inorganic soiling, e.g. ionic salts, as well as polar organic soils, such as sugars and some polysaccharides and proteins (Wilks et al. 1992, p. 75).

Water has a neutral pH of 7. As it is such an excellent solvent it often contains different types of ions when found in nature. Some ions, especially cations, can cause further deterioration in textiles. They can reduce the cleaning power of a washing solution for dirt containing that type of ion. Ions can be catalysts for chemical reactions that can cause further deterioration of the textile. They can discolour the textile and bind organic dirt and polar organic compounds by linking the polar groups of the dirt to those of the textile. Anions can change the pH of the water, causing an acid or alkaline pH (Timár-Balázs & Eastop 1998, p. 187). To avoid ions in the water, it can be deionized or distilled. In literature about cleaning historical textiles, it is often suggested to use deionised or distilled water at the end of the cleaning process, to make sure there are no active substances left that could cause further deterioration (Landi 1992, p. 81).

As mentioned, water is the most polar solvent and with “like dissolves like” it dissolves many types of polar soils. It also dissolves a lot of the yellow and acidic degradation prod-

ucts that can be found in natural fibres. Water can improve the flexibility and softness of a textile by acting as a plasticiser. When plasticised it is easier to reorientate yarns and fibres to recover the original texture and dimensions. Water relaxes the strains in fibres, yarns and fabrics which removes creases and wrinkles. Water is not expensive and is readily available. Finally, and of great importance, water is not a health and safety hazard. Water in combination with surfactants and other additives can work as excellent cleaners (Timár-Balázs & Eastop 1998, p. 194).

The surface tension of the water, that is the tension between the phases solid, liquid and gas, can make the wetting of textiles slow. To fasten the wetting process, surfactants can be used to reduce the surface tension. A surfactant is a surface active agent whose molecules have both hydrophilic and hydrophobic parts, thus lowering the surface tension of solutions. A detergent is an aqueous solution of one or more surfactants. Soaps are surfactants that are metallic salts of fatty acids (Conservation-wiki 2017).

The structure of a surfactant is a hydrophilic (water loving) or oleophobic (oil hating) head/part and a hydrophobic (water hating) or oleophilic (having an affinity for oils) non-polar tail. Surfactants are divided in four groups depending on their different charges (figure 2). Anionic surfactants are negatively charged, non-ionic surfactants are not charged, cationic surfactants are positively charged and amphoteric surfactants are both positively and negatively charged. The charge of the amphoteric surfactants depends on the pH. A high pH makes the surfactant more negatively charged and a low pH, more positively charged. The surfactants most commonly used in textile conservation are anionic and non-ionic (Tinkham 2001, p. 6).

The water molecules and the hydrophobic tails are not attracted to one another, which makes the water try to squeeze the hydrophobic part of the surfactant out from the water. A surfactant layer is created on the surface, with the non-polar tails in the air, and the hy-

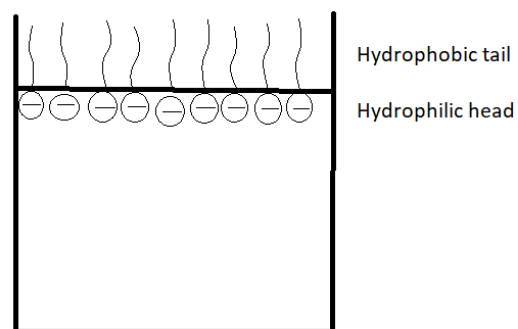
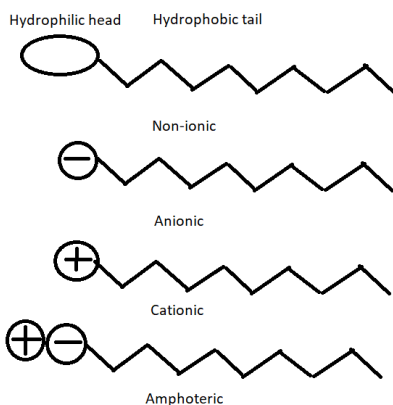


Fig. 2. The four most common types of surfactants.

Fig. 3. The hydrophilic heads absorbed by water and the hydrophobic tails pushed out from water.

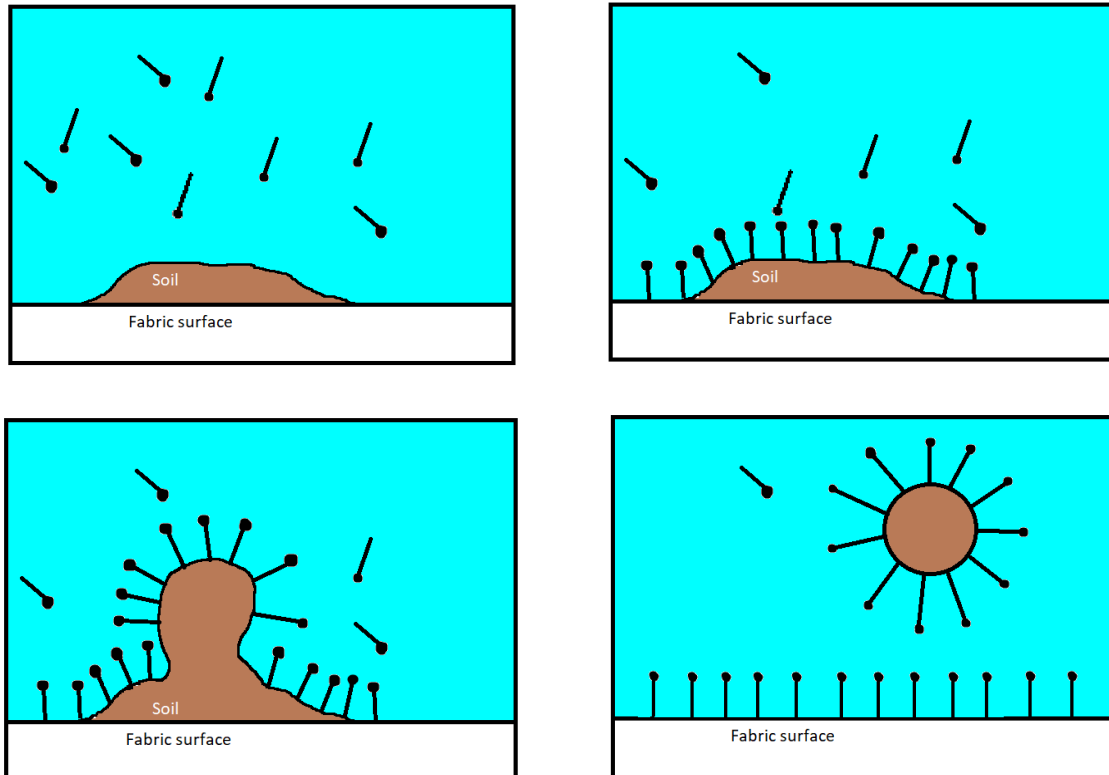


Fig. 4. How micelles encapsule, roll up and solubilise soil.

drophilic heads/parts dissolved by the water molecules (figure 3). The surfactant layer makes the water drops collapse and thus increases the surface area. When more surfactant molecules are pushed out, the water will spread and wet the surface of the textile (Timár-Balázszy & Eastop 1998, p. 195).

The concentration of surfactants at the surface reaches a critical point where the surface tension remains stable. The remaining surfactants start forming micelles, clusters of surfactant molecules, within the water. The concentration where clusters start forming is called the critical micelle concentration, CMC (Wolbers 2003, p. 30).

The micelles are the reason that surfactants can help release dirt from a surface. They can encapsulate and solubilise substances that do not dissolve in water (Timár-Balázszy & Eastop 1998, pp. 203-204) (Figure 4).

Different surfactants have different critical micelle concentration (CMC). When comparing the CMC of non-ionic and anionic surfactants, the CMC of non-ionic surfactants is lower, meaning that a lower concentration of non-ionic surfactants is needed to reduce surface tension than with anionic surfactants. The cleaning power of surfactants is more effective when the surfactant forms micelles, the concentration needs therefore to be higher than the CMC. When cleaning historic textiles it is common to use a concentration of 2-6 times the CMC value. A higher concentration than that makes it more difficult to remove the sur-

factant, and more action creates higher stress in the textile (Marko 2020, p. 83). The critical micelle concentration of non-ionic surfactants is low, 0,05-0,5 g/l. The CMC of an anionic surfactant is higher, 0,5-3 g/l (Timár-Balázszy & Eastop 1998, p. 202).

### 2.7.1 Anionic surfactants

Anionic surfactants ionise in water. The hydrophilic head will ionise to a positively charged cation; a metal ion, and the hydrophobic tail will become a negatively charged anion. The ionisation is hindered in acidic conditions. In alkaline conditions the surfactant can be fully ionised. The surfactant activity will increase with increasing pH. When considering using anionic surfactants, it is important to be aware that aged fibres and acid dyes can be sensitive to alkaline conditions (Timár-Balázszy & Eastop 1998, p. 97).

Soaps are anionic surfactants. Soaps should not be used in hard water, as they have the ability to form lime-soap, a calcium or magnesium salt of soap. Lime soap is insoluble in water and gives the textile a grey tone. Soap can also create an alkaline pH in the washing solution by releasing metal ions, for example  $\text{Na}^+$  which forms NaOH with water. The cleaning efficiency of soap is not very good in cold water.

Anionic surfactants can also be made from alkyl sulphates which contain 11-18 carbon atoms. Alkyl sulphates have similar detergency properties as soap but is not sensitive to the hardness of water. The salts they form are water soluble, hence they do not form lime-soap (Timár-Balázszy & Eastop 1998, p. 196). Temperature and water-hardness has an impact on alkyl sulphates. Low temperature and harder water means that the molecular weight of the surfactant needs to decrease to keep a good detergency. The C12-C14 range is therefore more desirable than the C16-C18 range (Tinkham 2001, p. 8).

Sodium Lauryl Sulphate/ Sodium Dodecyl Sulphate (SLS/SDS), a fatty alcohol sulphate/ primary alkyl sulphate is an anionic surfactant frequently used in conservation . It can be found in many detergents, for example in Orvus WA paste and Neutral hand wash liquid (Tinkham 2001, p. 8; CCS Healthcare AB 2012).

Anionic surfactants often have a good prevention of soil redeposition and are very effective in removing negatively charged polar soil (Tinkham 2001, p. 36).

Polyester fibres are hydrophobic and stay hydrophobic in an anionic washing liquid. The hydrophobic surface prevents liquid oily soil from rolling up into micelles, resulting in hardly any effect on oily soil on polyester (Ballard 2009).

### 2.7.2 Non-ionic surfactants

Non-ionic surfactants do not ionise in water, they stay neutral. They are made of a hydrophobic part of an alkyl chain, the tail, and a hydrophilic part of ethylene oxide units, the head. This can be used to manipulate the surfactant to be tailor made to what is needed. For example a lauryl alcohol with three ethylene oxide units is not water soluble, but one with nine ethylene oxide units is. A longer tail of the non-ionic surfactant gives a lower surface tension and reduced critical micelle concentration. Fewer ethylene oxide units also creates a lower surface tension and reduced critical micelle concentration (Timár-Balázs & Eastop 1998, pp. 198-199).

Non-ionic surfactants are better wetting agents than anionics and they dissolve well in cold water. They also have an effective cleaning power at low concentrations. Tests on anionic and non-ionic surfactants performed by Boring and Ewer in 1991 showed that the anionic surfactants removed more soil from wool than the non-ionics, but on cotton they removed the same amount of soil. The anionics showed better anti-redeposition results than the non-ionic surfactants (Tinkham 2001, p. 36). Non-ionic surfactants emulsifies oils well and remove organic soils more effectively than an-ionic surfactants

The temperature of the cleaning solution affects the surfactants, as they all have their own ideal temperature for solubility. At some temperatures the surfactants can not dissolve fully and the water becomes cloudy. This is named the cloud point. Non-ionic surfactants often have a rather low cloud point and dissolve when the temperature is lower than the cloud point. The cloud point depends heavily on the polyoxyethylene chain length (Holmberg 2002, p. 109). Anionic surfactants have a higher cloudy point, called Kraft point, and dissolves when the temperature is higher than the Kraft point. Sequestering agents can be used in a washing solution to help surfactant dissolve at lower temperatures. The reason for why non-ionic surfactants have a lower cloud point is that they have a large hydrate layer around the micelle, because of the long polar parts of the surfactant molecules, that bind the water molecules (Timár-Balázs & Eastop 1998, p. 202).

### 2.7.3 Sequestering/Chelating Agents

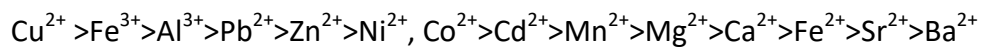
Sequestering and chelating agents form bonds with metal ions and keep the ions in a complex. Sequestering means *locking away* and the word chelating originates from the greek word for *claw*. They both have the same function, but operates in different ways (Caryle, Townsend, & Hackney 1990, p. 44).

The complex formed with metal ions can be used to soften hard water. Sequestering agents can also be used as cleaning agents, they can enhance the cleaning effect of surfactants and act as soil carriers. As they bind metal ions present in dirt, the quantity of dirt to

be removed with the help of surfactants will decrease. Sequestering agents can also be a part of the removal of fatty dirt by breaking it up, bringing it into colloidal solution and dispersing the dirt. The combination of sequestering agents and surfactants often enhances the effectiveness of wet cleaning (Timár-Balázsy & Eastop 1998, pp. 190-191, 207, 225).

Examples of chelating agents used in textile conservation are EDTA and citric acid and its salts, such as di- and triammonium citrate. The pH of diammonium citrate is 6, and, as the more ammonium ions, the higher the pH, triammonium citrate has a pH of 7 (Caryle, Townsend, & Hackney, 1990 p. 44). The use of very strong chelating agents, such as EDTA, pH 8, might affect metal complexes of dyes with resulting colour changes (Boutrup 1983).

Sequestering agents prefer different metal ions and a sequestering agent has a specific preference order of different metals. Citric acid prefers  $\text{Cu}^{2+}$  but if there are none, or when they are all sequestered, it binds with other metal ions in the following preference order:



Citrates are often preferred for cleaning of historical textiles, as they have good sequestering properties at low temperatures, they have a pH close to neutral and they bond metal ions within a wide pH range. In a concentration of 1-2% they are effective in removing lime soap (Timár-Balázsy & Eastop 1998, p. 224). Ammonium citrate can be used to remove foxing, which is a problematic stain sometimes found on textiles (Dwan 2015, p.10).

#### 2.7.4 Solvents

Solvents other than water can also be used for cleaning textiles. Cleaning textiles with organic solvents is what is known commercially as dry cleaning. Organic solvents can dissolve or swell different types of soiling, such as tars, bituminous materials and waxes as well as oily, greasy dirt. Other reasons for using solvent cleaning is when water cleaning can harm the textile or other materials attached to the textile. Solvent cleaning can be done by immersing the textile in solvent or used locally on stains and soiling.

A problem with using solvents on textiles is that they might cause desiccation of the fibre. Desiccation is caused by extraction of water bound physically within the fibre. For example alcohols (ethanol) and ketones (acetone) bring bound water with them when evaporating. If the organic solvent contains some water, the water regain of the fibre may be improved (Timár-Balázsy & Eastop 1998, pp. 59-62).



### 2.7.5 Enzymes

Enzymes are proteins that are highly specialised. They act as organic catalysts and can increase the rate of a chemical reaction considerably. The suffix '-ase' is used to signify that we are dealing with an enzyme (Punekar 2018, pp. 33, 43 )

Enzymes are often used to break down dirt from large molecules to smaller fragments. They are often called digesters due to their role in metabolism.

Enzymes are specific to a substrate, that is, it breaks down a specific substrate. It also often gets its name from that substrate. Examples of this is cellulase that digests cellulose, protease digest proteins (except collagen). Other enzymes common in textile conservation are diastase for starch, trypsin for proteins, lipase for fats and olease for oils (Timár-Balázs & Eastop 1998, pp. 233-234).

## **2.8 Analytical methods**

### 2.8.1 Colour Measurements

It can be complex to describe a colour, as there are so many variables involved, such as the coloration, the surface gloss or mattness, and the contrast between neighbouring colours. Physical measurements can reveal the wavelength characteristics and aid in the identification of colourants. There are many analytical tools that can be used for colourant identification. One non-invasive technique is spectrophotometry. Spectrophotometry measures the amount of light reflected or transmitted by a material at individual wavelengths of the spectrum (Johnston-Feller 2001, pp. 1-2).

A spectrophotometer has three essential parts: 1. a light source, 2. a monochromator that selects individual wavelengths and 3. a photo detector. Other essential parts are a reference standard, arrangements for illuminating the sample, and possibility to select or collect light that falls on the detector. A spectrophotometer measures the ratio between the light reflected or transmitted by a sample and the light reflected or transmitted by a reference standard. The ratio is often printed in numerical form (Johnston-Feller 2001, p. 6).

The most used method of classifying and measuring colour is based on colour spaces defined by the International Commission on Illumination (CIE). The CIE colour spaces are based on a set of three colour matching functions for the eye. The illuminant that is most commonly used in colorimetry today is daylight 65 (D65) standard source. The CIE lab colour space is a widely accepted method of expressing colour. L in lab gives a measurement of lightness, a expresses the position of a colour on the red-green axis, and b the position of colour on the yellow-blue axis (Saunders & Project Muse 2020, pp. 45, 46, 48)

Colour differences can be measured instrumentally and quantitatively so that the difference in colour can be expressed in standard CIE units. The currently used CIE unit for meas-

uring colour differences, is termed CIE 2000 and abbreviated as  $\Delta E_{00}$  (Saunders & Project Muse 2020, p. 58).

### 2.8.2 Infrared Spectroscopy

Infrared (IR) spectroscopy is a molecular spectroscopy that uses vibrational transmission to determine and identify molecular structures (Stuart 2007, p. 110). The mid infrared region, used for Fourier Transform Infrared (FTIR) spectroscopy, is found between 4000 - 400  $\text{cm}^{-1}$  (Larkin 2011, p. 14).

Vibrational spectroscopy can be used as a simple identification test or as an in depth full spectrum. It can be used for qualitative and quantitative analysis. Raman and mid-IR spectroscopy are complementary techniques. Raman spectroscopy gives the best information with symmetric vibrations of non-polar groups, while IR spectroscopy gives the best information with the asymmetric vibrations of polar groups. Vibrational energy levels are unique to each molecule. The spectra provided by a particular molecule can be described as its fingerprint. An IR spectra provides information on molecular structure, dynamics, and environment (Larkin 2011, p.1-2).

Almost all mid-IR instrumentation are Fourier transform infrared (FTIR) spectrometers. They are based on an interferometer that separates light into individual components. To get an acceptable quality FTIR spectra, it is important to prepare the sample in a correct way. The sample needs to be uniformly thick and homogeneously mixed, avoiding holes or voids. If the sampling is prepared in an incorrect way, it might create a false IR Spectra. False IR spectra are characterised by a broadening of the most intense bands and a strengthening of the weak bands (Larkin 2011, pp. 30-34).

Attenuated total reflection (ATR) is a FTIR sampling technique that uses the spectral information obtained from reflection properties. ATR is a contact sampling method that uses a crystal with excellent IR transmitting properties. It is quick, non-destructive and requires no sampling preparation. It is used to measure the IR spectra of surfaces as well as materials too thick or strongly absorbing to be analysed by other, more traditional transmission methods (Larkin 2011, p. 30).

FTIR spectroscopy is often used to study polymers. It is easy to find reference spectra collections and spectral databases for polymers (Stuart 2007, pp. 122-123).

### 2.8.3 Tensile Testing

Tensile testing applies tension to a sample and determines its breaking load and the elongation at break (Wilson 2012, p. 51).

A tensile testing machine elongates materials at a constant rate. The material is clasped between two grips, which slowly pulls apart. The applied load on the sample is measured

as well as the elongation of the sample. The imposed stress affects the degree to which a material strain. Stress is defined as the load (F) per unit area (A). Tensile stress shows how resistant the material is to stretching forces. The amount of deformation per unit length of the material gives us the strain (Stuart 2007, pp. 358-359).

- Stress ( $\text{Nm}^{-2}$ ) = Force (N)/Cross-section area ( $\text{m}^2$ )
- Strain = Elongation (mm)/Initial length (mm)
- Extension (%) = Strain x 100%
- Strain = Stress/Young's modulus (Wilson 2012, p. 52)

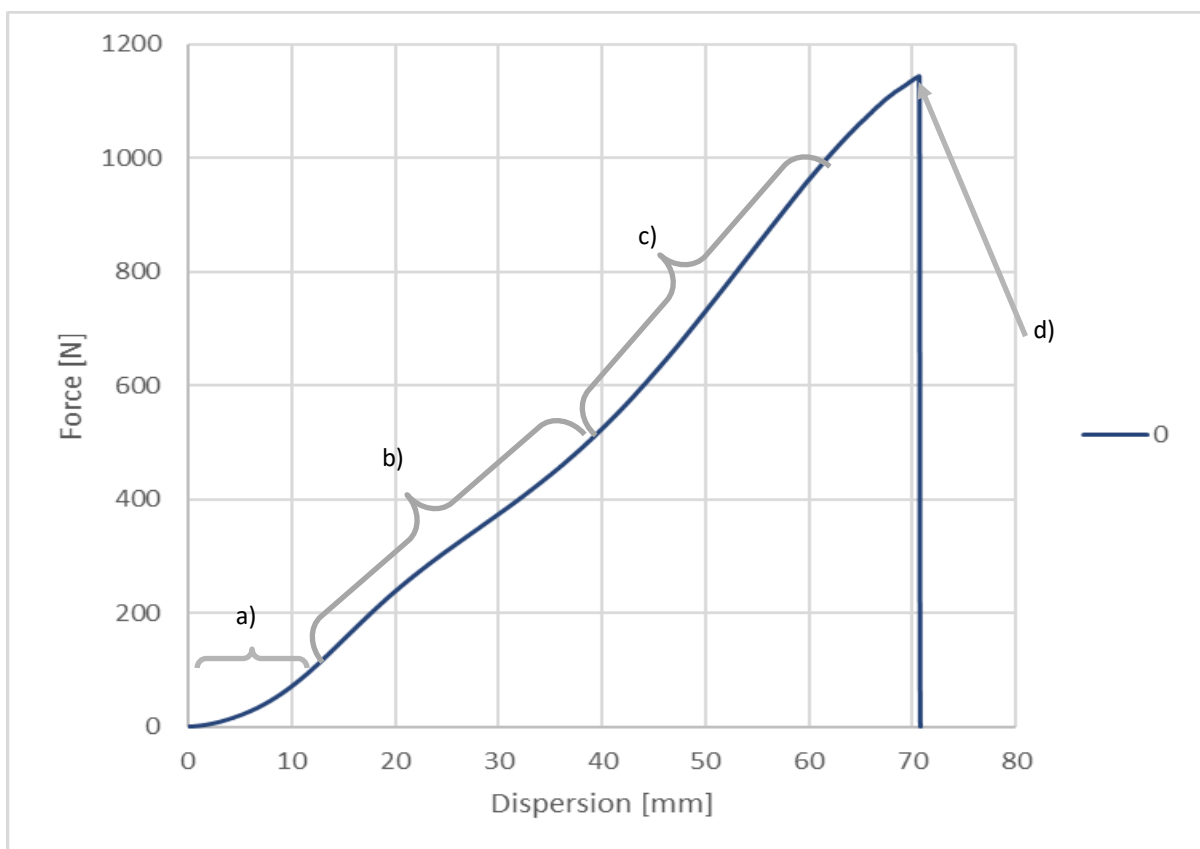


Fig. 5. a) The specimen slacks. b) The yarns of the warp are stretched straight and tight. c) The force is being taken by the fibres instead of the yarn. d) The breakpoint.

Figure 5 shows the different parts of a tensile strength curve. When stress and strain are proportional, creating a linear graph, the stretch is still reversible and the deformation is called elastic deformation. It creates a linear graph in a plot of load-elongation or stress-strain. The gradient of such a plot gives information of the stiffness of the material. The steeper the curve is, the more stiff the material is. When the stress and strain no longer is proportional, the polymer chains move past each other and plastic deformations occur.

The fibre does not regain its original form if the load is removed. The gradient of the curve in the plot bends. Eventually the polymer chains will break and the textile will tear. The breaking load (N), extension at break (%), strength and the extensibility can be settled at the breaking point (Hindborg 2019, p.42; Stuart 2007, p. 359; Wilson 2012, p. 52).

Polyester (PET) is a semicrystalline plastic where the behaviour under tensile forces can be described like this:

1. A reversible elongation of the amorphous ties.
2. Crystalline regions slide past one another.
3. Crystalline regions separate into crystalline segments. The segments are tied together by amorphous structures. In this stage the irreversible plastic deformation starts.
4. The crystalline segments and amorphous ties are further stretched and the cross-section starts to decrease, called necking. The necked areas will continue to decrease until break of fibre occurs (Asadi et al. 2021, pp. 7-8).

### 3. Interviews

During the autumn of 2022, interviews were conducted with 12 textile conservators regarding the cleaning of historical textiles. The interviewees chosen should be trained as textile conservators. There should be a variation on when and where they were trained. There should be conservators that worked in different parts of Sweden and worked in museums, in the private sector as well as in the Swedish church.

16 textile conservators throughout Sweden were asked via e-mail to participate and interviews were conducted with 12 of them. Many of the interviewees got their conservation education at the University of Gothenburg in Sweden and some gained their education in England, Switzerland and Austria. They were educated from the 1980s to the 2020s. 6 of them worked in museums, 5 in the private sector and one in the Swedish church.

9 of the interviews were conducted by telephone and three in person. The interview took approximately one hour.

The interviews were conducted using a qualitative technique with planned, open-ended questions. The questions were:

1. What kind of textiles are you cleaning? What materials are they made from?
2. What equipment do you use for surface cleaning? What techniques do you use?
3. What equipment do you have available for wet cleaning?
4. What techniques do you use when wet cleaning?
5. What types of stains do you see? What type of cleaning agents do you use for stains? What methods do you use to treat stains?
6. What kind of water do you use?
7. Which detergents do you use when wet cleaning? For what type of fibre? For what type of dirt?
8. How do you handle the textile during cleaning?
9. How do you investigate if there are any surfactant's left in the textile after rinsing?
10. How do you dry the textile?
11. How do you flatten the textile?

12. Do you bleach textiles? What method or bleaching agent do you use?
13. Do you write a cleaning appendix and put it in your report?
14. Do you measure pH? When do you measure pH? How do you measure pH?

### **3.1 Results from Interviews**

The interviews were conducted both for a course about cleaning of historical textiles, as well as in preparation for writing this thesis. Question nr 5, 6 and 10 were used in the thesis, and the answers were, along with information from the literature review, used to decide on what types of stains, types of cleaning agents and types of cleaning methods to use in the thesis (Appendix 1).

The interviewees mainly used tap water, but also distilled and deionized water. The drying methods used can be seen in appendix 1.

When looking at the stains found by the interviewees (Appendix 1), a clear difference in choice of cleaning agents could be seen between fatty/greasy stains and other stains. There was also a difference in choice of cleaning agent depending on what type of fibre that was being cleaned and what pH was the most suitable for that fibre.

The interviews showed that the detergents often used by textile conservators in Sweden were no longer in production. During a literature research in the autumn of 2022 I found that the anionic detergent Orvus WA paste and the non-ionic detergent Dehypon LS54 were researched and recommended by conservators in other countries (Sato & Quye 2019; Canadian Conservation Institute (CCI) 2008).

Many of the interviewees wanted to use or used gall soap, but wanted to know if it is a safe option to use in textile conservation. Some of the interviewees thought that there might be enzymes in the gall soap, but did not know which ones.

Most of the interviewees did not use chelating agents when cleaning textiles, but one, trained in England, used it a lot, for many purposes.

The interview, as well as literature research, showed that glycerol is being used as a pre-treatment to soften a stain pre-cleaning (Tímár-Balázs & Eastop 1998, p. 237; Holm 1946, p. 23).

The interviewees all used cotton swabs and blotting paper as methods for stain removal. The ones who had access to a suction table found it very useful during stain removal.

## 4. Selected Materials

The literature review and the answers from the interviews resulted in deciding to choose three different types of staining agents, one acidic, one alkaline and one fatty. The acidic and alkaline staining agents were chosen as high or low pH can affect textile fibres differently. The fatty stain was chosen as polyester has an affinity for fats, which often make those type of stains hard to remove. The results also led to decisions around what cleaning agents to use. Three detergents were chosen, one chelating/sequestering agent and one solvent. It was also decided to pre-treat some samples with glycerol to assist the stain reduction.

### 4.1 Used Staining Agents

#### 4.1.1 Coffee

The chemical composition of coffee is not fully known. Various chemical compounds that can be found in coffee are approximately: caffeine 2-5%, acids 7-17%, trigonelline 1-2%, protein 1-6%, sugar 1-5%, polymeric carbohydrates 30-50% and coloured ingredients 20-35%. Coffee stains are mainly caused by hydrophilic coloured components that are molecularly or colloiddally soluble in water. Coffee stains are caused by acidic substances. The pH of instant coffee from Folgers Crystals is 5. Coffee contains phenolic acids, nicotinic acid and aliphatic acid but they are all colourless and can therefore not stain fibres. They can, on the other hand, affect staining by forming acidic coloured species in coffee. Caramelised monosaccharides and sucrose can react with the phenolic acid chlorogenic acid and form a brown substance resembling humic acid which has an intensifying effect in dyeing (Kissa 1995, p. 79; Vashurina, Pogorelova & Kalinnikov 2003).

#### 4.1.2 Ashes

Soot is a dirt often found on historical textiles. It often has its origin in air pollutants, log burners and candles. It is a mixture of carbon in particle form, organic tars, resins and other, inorganic materials. Particulate carbon makes up less than 50% of the weight of soot (Hackett 1998). It is not possible to buy synthetic soot to stain samples and it is not easy to create it, therefore wood ashes were chosen to represent soot as they are closely connected. Wood ash typically contains Carbon, Calcium, Potassium, Magnesium, Phosphorus and Sodium (Wikipedia 2023). Wood ash is alkaline, which adds an interesting aspect to the study with acidic coffee and alkaline ashes.

#### 4.1.3 Sebum

Sebum is the oily substance found on human skin. It is often found on historical clothes, for example in the neck line of an ecclesiastic stole. The sebum used in this study is synthetic. Sebum has a melting range between 43 and 66°C (Scientific Services S/D inc. 2021).

## 4.2 Tested Cleaning Agents

When washing historical textiles it is important to use a suitable detergent. It should not contain perfume, colourants, whiteners or enzymes which might remain in the fibres after rinsing and may harm the fibres. Another important part of the detergent is that it should have a neutral pH and should clean well at lower temperatures.

#### 4.2.1 Orvus WA Paste

Canadian Conservation Institute (CCI) recommends Orvus WA paste as a suitable anionic detergent to use for cellulosic historical textiles (CCI 2008). It contains Sodium Lauryl Sulfate, Lauryl Alcohol and Sodium Sulphate (P&G 2023). The pH is 7,8 (P&G Professional 2015) and it is soluble in cold or hot water (Conservation Support Systems 2023). Orvus WA Paste is easy to get hold of and can be bought from many different distributors.

Orvus WA Paste has a measured CMC of approximately 3,0 g/l at 35°C (Tinkham 2001 p. 49). The stains were cleaned with a concentration of 7,5 g/l which is 2,5 x CMC. Surfactants start forming micelles at CMC and above, and micelles help with the extraction of soil (Wolbers 2003, p. 30).

Tinkham (2001) investigated the difference between using agitation, that is movement of the cleaning media, during stain reduction and not using agitation. Her results showed that Orvus WA Paste reduced the stains further with agitation than without agitation.

#### 4.2.2 Dehypon LS54

Before year 2000, non-ionic Synperonic N was used to clean historical textiles in the UK. In 2000 it was banned in EU due to environmental issues regarding its biodegradability (Sato & Quye 2019). Thorough research was conducted by Fields et al. and Sato & Quye to find a suitable replacement for the non-ionic Synperonic N. Dehypon LS54 was deemed to be a suitable replacement. Dehypon LS54 has a cloud point at 30° which makes it fitting for washing historical textiles. The optional concentration of Dehypon LS54 to use for washing



historical textiles is 3 g/l (Fields, Wingham, Hartog, & Daniels 2004; Sato & Quye 2019). Dehypon LS54 has a pH value of 6-7,5 (BASF 2018).

#### 4.2.3 Gall Soap

Many people in Sweden use gall soap for stain removal. The interviews showed that textile conservators are curious on how gall soap affects the textiles and if it is safe to use when cleaning historical textiles. According to the distributor of gall soap in Sweden, it contains ox gall/bile and chlorophyll and are effective against oily, fatty stains as well as stains from grass, berries, fruit and vegetables. They suggest to rub some soap on the stain and create a foam which should be left on the stain for a little while. The next step is to rinse it with water or to do a wet cleaning with the textile (Galltvål 2023). Gall consists of bile acids. Bile acids have both hydrophobic and hydrophilic properties, and form micelles. The micelles can emulsify lipids and facilitate fat absorption (Sayin 2016). On some web pages on Gall soap, it is said to contain natural enzymes (Star Aroma 2023) but according to Galltvål Sweden (2023), the soap does not contain any enzymes. Gall soap has a pH value of 10-10,5 (Sodasan 2019).

#### 4.2.4 Triammonium Citrate

Triammonium citrate is a chelating agent used in textile conservation of historical textiles. One of the interviewees used triammonium citrate in cleaning of historical textiles, but none of the others mentioned using it. Literature shows that it is used more frequently in other countries (Haldane 1999; Adler & Eaton 1996).

Triammonium citrate has a pH value of 7 (Caryle, Townsend & Hackney 1990).

#### 4.2.5 Ethanol

Landi (1992) describes that the solvents to try after water based solvents or regents are the alcohols. Tímár-Balázs & Eastop (1998) suggests using ethanol and water on body decomposition product stains. It was also suggested by the interviewees to treat fatty stains with ethanol.

## 5. Materials and Methods of Experiments and Analyses

To answer the research questions in chapter 1.3, experiments were conducted. The experiment consists of two cycles of artificial ageing of polyester fabric, three types of stains, five types of cleaning agents and two methods to clean the stains. Each version of the experiments was repeated three times.

The expectations were to find suitable cleaning methods and cleaning agents to use for stain removal/reduction on polyester fabric.

The treatment was evaluated through different analytical methods in the aim of answering the research question. The treated samples were compared to untreated reference samples as well as stained samples, not cleaned. To understand the colour differences, colour measurements were conducted using a spectrophotometer. To get an insight into if the artificial ageing had affected the polyester fibres, infrared spectroscopy was used. To gain an understanding of if the accelerated ageing, the staining and cleaning agents affected the polyester, tensile testing was conducted.

### 5.1 Experimental design

An experimental design was set up to plan the experiments. Table 1 shows the factors of importance to execute the experiments.

One factor of significance for conservation of historical textiles is the age of the textile. Therefore versions with unaged, aged once and a polyester fabric aged twice were set up.

Different types of stains are adhering with dissimilar strength to a specific type of textile fibre. Therefore three types of dirt were chosen to stain the polyester fabric.

The cleaning agents chosen had different strengths and weaknesses and the variety of produce helped answer which types are suitable to use for cleaning polyester fabric with the types of stains chosen in this study.

Another factor of importance was what cleaning method to use when removing stains from polyester fabric. Two approaches of cleaning methods were chosen: using cotton swabs to work mechanically on the stain and using a suction table and letting the cleaning agent drip through the stain.

The final important factor was to use multiple replicas. For this pilot study, three replicas were deemed appropriate, but for a full scale study at least five replicas should be used.

**Table 1. Factors, versions and naming of samples**

Factors/Variables	Version 1	Version 2	Version 3	Version 4	Version 5
<b>Condition of polyester</b>	0. Unaged	1. Aged 570 h/ stain not aged	2. Aged 570 + 384 h/stain aged 384 h		
<b>Type of stain</b>	A. Coffee	B. Ashes	C. Sebum		
<b>Type of cleaning agent</b>	a. Orvus WA Paste	b. Dehypon LS54	c. Gall soap	d. Triammonium Citrate	e. Ethanol
<b>Type of cleaning method</b>	I. Cotton Swabs	II. Suction Table			
<b>Replicas</b>	× First replica	○ Second replica	□ Third replica		

Standardised set-ups of the experiments were organised, to make certain the experiments were performed consistently (Table 2). All experiments were performed on textile samples made of polyester fabric with three replicas per sample. Coffee is a liquid and spreads rapidly in a woven fabric. Tests were done to find an appropriate amount of coffee to create a visible stain with an acceptable amount of spreading. The stains made by ash and sebum were easier to control, and followed the ISO standard 105-E07:2010. The time required to

**Table 2. Standardised set-up of experiment**

Type of fibre	Polyester
Number of replicas	3/sample
Amount of coffee in stain	0,04 ml
Diameter of ashes stain	20 mm
Diameter of sebum stain	20 mm
Time for pre-treatment with glycerol	15 min
Number of cotton swabs /stain	7
Number of times sponge was pressed down for rinsing the stain	10x3
Amount of cleaning agent dripped through the stain	2 ml x 4
Time the stain was covered with blotting paper in between dripping	30 sec
Amount of deionised water dripped through to rinse the textile	40 ml

pre-treat stains with glycerol was tested and 15 minutes were deemed appropriate. The amount of cleaning and rinsing to be used were also tested out to find suitable quantities.

As a final step of the experimental design, the outcome of the experiments was evaluated. All samples were analysed using colour measurements and tensile testing. The unstained samples were also analysed using infrared spectroscopy.

## 5.2 Test Fabric

The textile used for the experiments was a contemporary, white 100% polyester micro satin bought at Gårda Textil, Gothenburg, Sweden, January 2023. It had a density of  $140 \text{ g/m}^2$ . Prior to sampling, the textile was washed in a washing machine at  $40^\circ\text{C}$  without detergent, and hang-dried at ambient room temperature.

## 5.3 Sampling

177 textile samples were prepared according to ISO standard 13934-1, Textiles - Tensile properties of fabrics, part 1: Determination of maximum force and elongation at maximum force using the strip method. They were cut in warp direction with a width of 50 mm + fringe of 5 mm per side and a gauge length of 200 mm (Figure 6).



Fig. 6. Samples prepared with gauge length 200 mm and width 50 mm + fringe. The samples are placed in the climate chamber, ready for ageing.

The samples were given names according to a system (Table 1) where the ageing was marked with 0, 1 or 2, the staining agent was marked with A,B or C. The cleaning agents with a, b, c, d or e. The cleaning method was marked with Roman numbers I or II, and finally the three samples of each type was marked with a cross, a circle or a square. 1. A. a. I. x. is an example of a sample that is aged once (1), stained with coffee (A) and cleaned with Orvus WA Paste (a), the method used is cotton swabs (I) and it is replica number one in the batch (x).

## 5.4 Staining agents

Three different types of staining agents were used.

1. Coffee. Instant coffee was prepared by dissolving 12,5 g/l Ica Basic instant coffee in hot water (Kissa 1995). The coffee was cooled to room temperature before use. pH of the coffee was 4,5-5, and pH of the coffee stains after cleaning 6,4-7,6.
2. Ashes. Wood ash from spruce was sifted through a fine sieve to remove larger particles. The ashes were mixed with deionized water, 1 g of ashes to 4 ml water. pH of the ash and water mixture was 11, and pH of the ash stains after cleaning 7-8,6.
3. Synthetic sebum. Item # 2218027 from Testfabrics, Inc. (January 2023).

## 5.5 Staining

Three different types of stains were prepared according to ISO standard 105-E07:2010, Textiles - Tests for colour fastness - Part E07: Colour fastness to spotting: Water. The amount of staining agent needed for each type of stain, as well as what methods to use for staining, were examined prior to staining the samples.

1. Coffee. A graded pipette was used to stain the fabric with 0,04 ml coffee. The coffee drops were worked into the fabric with the rounded end of a glass rod until it had a  $\varnothing$  of 20 mm. The stains spread in the fabric in weft length (figure 7).
2. Ashes. The ash and water mixture was stirred and added to the textile with a paintbrush. The mixture was worked into the fabric with a paintbrush until it had a  $\varnothing$  of 20 mm. It was left to dry and ashes that were loosely bound to the surface were removed using a vacuum. The ash and water mixture had an average weight of 0,057 g before vacuuming (figure 8).

- Synthetic sebum. The rounded end of a glass rod was dipped in sebum and sebum was worked into the fabric until it had a  $\varnothing$  of 20 mm. The sebum used had an average weight of 0,014 g (figure 8).

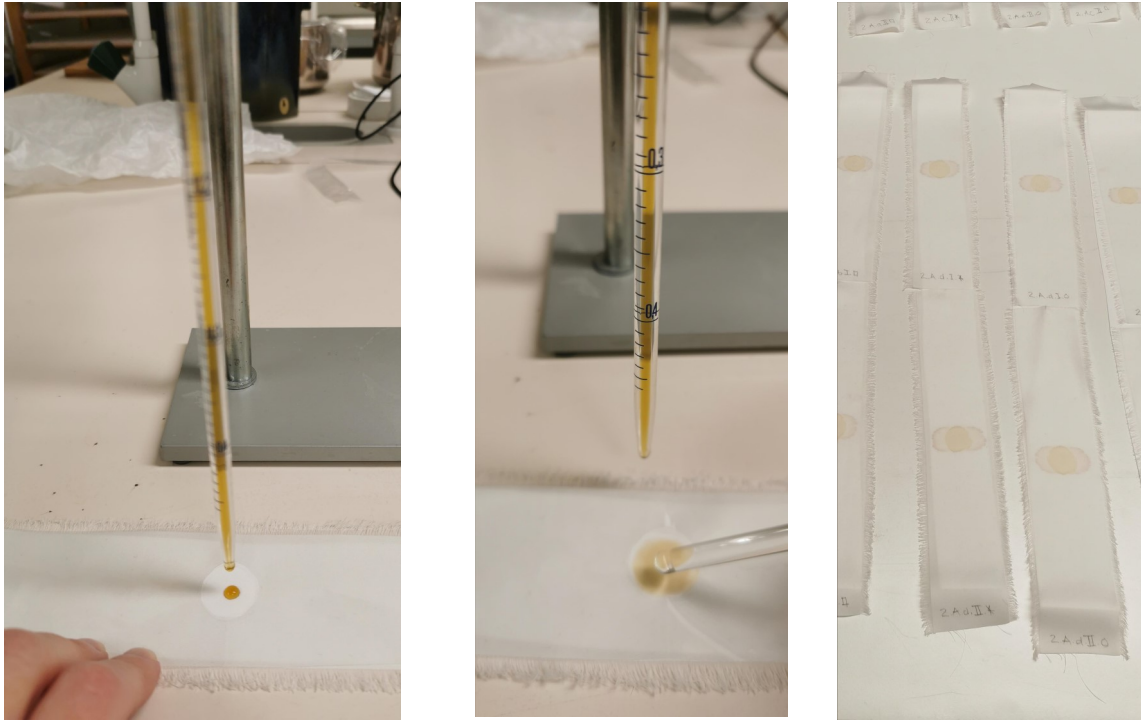


Fig. 7. Staining polyester fabric using coffee. The coffee spread in the weft of the fabric.

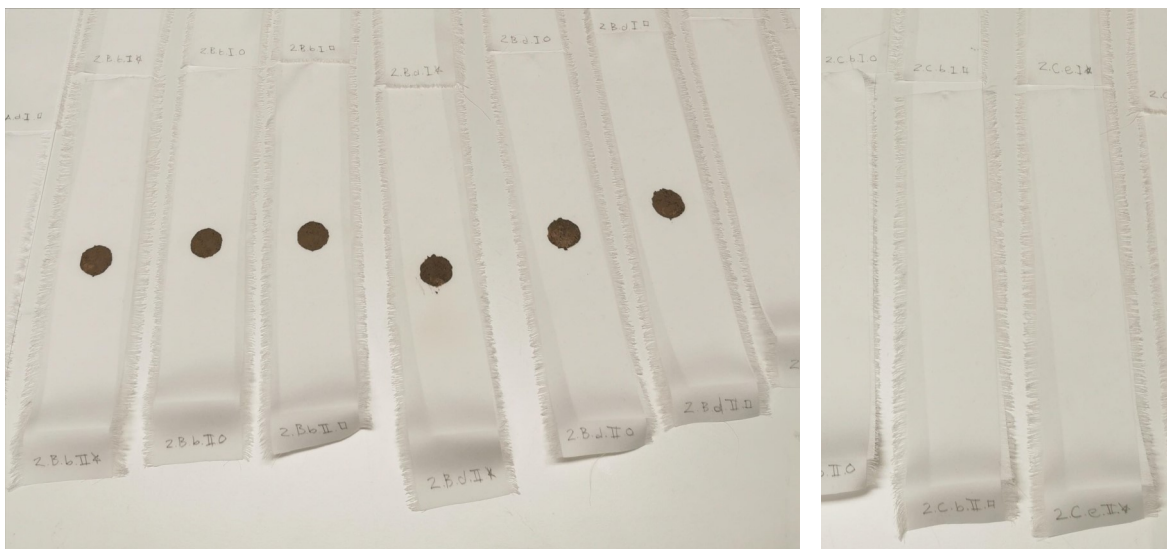


Fig. 8. Stains from ashes and sebum.

## 5.6 Accelerated Ageing

A Clima Temperatur Systeme CTS climate chamber from Climate Test Systems AB was used to age the textile samples (Figure 9). The samples were aged at 79° C, 0% relative humidity (RH) for 90 h and then at 95° C and 60% RH for 480 h. A second ageing was performed with half of the samples at 95° C and 60% RH for 384 h (Figure 10). The samples were hung in the climate chamber by a thread sewn through the top middle of the sample, and tied to the rack in the chamber. A MSR 17/22 data logger was placed in the chamber to record temperature and relative humidity during the ageing. The temperature and relative humidity in the climate chamber stayed quite consistent during the ageing. The temperature lowered from 65°C to 62°C at around the 1177 measurement of the second ageing. It remained at that temperature during the rest of the ageing (Appendix 2).



Fig. 9. The CTS climate chamber with all samples ready for ageing.

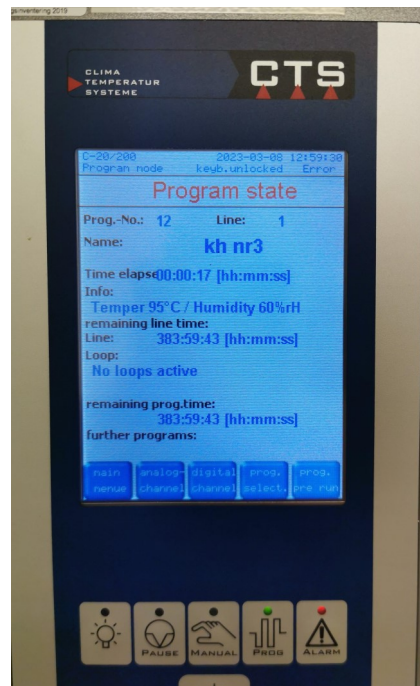


Fig. 10. The settings used in the second ageing.

## 5.7 Cleaning agents

The cleaning agents tested in this study were:

- Anionic surfactant Orvus WA Paste by Protector and Gamble. The concentration used was 7.5 g/l (Tinkham 2001 p. 49).
- Non-ionic surfactant Dehypon LS 54 by Basf. The concentration used was 3 g/l (Sato & Quye 2019).

- c. Gall soap Galltvål by Galltvål Sverige AB. The concentration used was 7.5 g/l. Instead of rubbing the soap on the textile as suggested by the distributor, a solution was prepared. As soaps are anionic surfactants, the solution was prepared in the same manner as the solution for Orvus WA Paste, with 7,5 g soap per litre of water.
- d. Chelating agent Triammonium Citrate by Alfa Aesar. The concentration used was 3 g/l. In this study, triammonium citrate was used on coffee and ash stains.
- e. Organic solvent Ethanol, 97%. In this study, ethanol was used to treat stains from sebum.

## 5.8 Stain Removal

Two types of stain removal methods were used, one by placing the textile on top of absorbing material and swabbing the stains, using cotton swabs dipped in the cleaning agent and the other by dripping the cleaning agent through the fabric, using a suction table. The treatment was carried out at ambient temperature.

Coffee stains and sebum stains were pre-treated with glycerol for 15 minutes. Glycerol is used to lubricate a stain (Tímár-Balázsy & Eastop 1998, p. 237) and might also help remove the coloured tannins of coffee (Holm 1946, p. 23). Trials were done to treat ash stains with glycerol, but no effect was observed, therefore no pre-treatment of ash stains were done.

In the cotton swab method the sample was placed on blotting paper, after which it was swabbed with cotton swabs. Seven cotton swabs were used for each stain. Each swab was



Fig. 11. Cleaning stained samples using cotton swabs and blotting paper.



dipped once in a cleaning agent and rolled over the stain, starting at the edges, and going towards the middle. After treatment with one swab the blotting paper was moved so that the stain would have a clean surface underneath. After finishing treatment the stain was rinsed using a natural sponge and deionised water. The sponge was pressed down 10 times before it was rinsed again in deionised water. The rinsing was repeated three times. The sample was pressed between two pieces of blotting paper to remove excess water and then left to dry on a netted frame (Figure 11).

The suction table, used as the second method, had a circular shape with a diameter of approximately 35 cm, and was custom-built by a metal craftsman. The top of the table includes a metal platter with holes covered by polyester wadding. On top of the wadding another metal platter with smaller holes was placed. The table was connected to a wet vacuum cleaner when handling water based cleaning agents, and to the ventilation system when handling organic solvents. The metal top was covered with a highly absorbent paper towel and plain woven cotton fabric, folded in four layers, to avoid the holes on the metal platter making marks on the samples and to absorb dissolved dirt. To adjust the amount of suction, a polyester sheet (Melinex®), with a hole cut out for treatment of the stains, was placed on top. The stained fabric was placed on the suction table, and 2ml cleaning agent was dripped through the stain using a pipette. The stain was covered with blotting paper and Melinex® for 30 seconds to assist the removal of loosened dirt particles. These two steps were repeated 4 times. The sample was placed on a dry and clean part of the cotton fabric and the stain was rinsed on the suction table, using 40 ml deionized water dripped through the cleaned area and a little bit outside the area to reduce the risk of ring marks. The textile was moved to a dry area, and dried on the suction table. During the drying period the textile was first covered with blotting paper and Melinex® for 10 seconds and then without covering until dry (approximately 30 sec.) (Figure 12).



Fig. 12. Cleaning stained samples on a suction table. Photo by Johanna Nilsson.

## 5.9 Photography

All samples were photographed using a Panasonic Lumix DC-TZ90 digital camera . They were photographed in a darkroom with two spotlights and a dark background. The camera was set on manual with aperture f 8 and shutter speed at 3.2 or 4. Both sides of the sample were photographed after staining and after cleaning. The samples aged twice were also photographed after ageing. The front was photographed after tensile testing.

## 5.10 Colour Measurement

Equipment:	Konica Minolta CM-26d Spectrophotometer.
Illumination/viewing system:	Diffused illumination, 8° viewing angle
Wavelength range:	360 to 740 nm
Measurement diameter:	3 mm
Daylight illuminant:	D65

The CIELAB colour-difference equation where used in this study.

The spectrophotometer was used to measure colour of the stains and the textile and to calculate the colour differences  $\Delta L$ ,  $\Delta a$ , and  $\Delta b$  as well as the total colour difference,  $\Delta E$ .

A general guide to the  $\Delta E$  perception ranges is:

- 0-1,0 Not perceptible by the naked eye
- 1-2 Perceptible through close observation
- 2-10 Perceptible at a glance
- 11-49 Colours are more similar than the opposite
- 100 Colours are exactly the opposite (Schuessler 2023)

L shows lightness where 0 = Black and 100 = visible white, a shows the green-red field where - is green and + is red, and b shows the blue-yellow field where - is blue and + is yellow (Mokrzycki & Tatol 2011, p.15).

The colour of the textile was measured before ageing and staining and the resulting colour was chosen to be the reference that the other measurements were compared to.

The colour of the textile was measured after the two ageing periods, which also were used as reference samples.

The colour of the staining was measured after staining, after the second ageing and after cleaning. Each measurement was repeated three times with a small moving of the spectrophotometer in between each repetition.

All samples were measured at the front and at the back of the textile/stain.

### **5.11 Infrared Spectroscopy**

Equipment:        Alpha FTIR spectrometer, Bruker  
                          Platinum-ATR

Infrared spectroscopy was conducted to search for changes in the molecules of the fibres after ageing the textile. This was done by comparing the spectrum of the chosen samples and searching for differences.

Measurements were taken on un-aged polyester, to use as a reference, on polyester aged once and on polyester aged twice.

The textile was folded once before placed in the spectrometer to create a double layer of fabric. This was done to reduce noise in the spectrum, which might occur if the material is too thin or has gaps between the fibres.

### **5.12 Tensile Testing**

Equipment:                SHIMADZU Autograph AGS-X  
                                  Trapezium Lite X software

Characteristics:        10 kN load cell  
                                  Flat grips with rubber pads

Gauge length:         200 mm

Speed:                    100 mm/7 min extension

Environmental conditions: 20°C, 65% RH

Acclimatisation:        20°C, 65% RH for 24 h

Tensile testing was conducted to investigate the mechanical degradation of fibres in the samples due to ageing, staining and cleaning (Figure 13).

Tensile testing was done at the Swedish National Heritage Board. The tensile testing instrument report can be found in appendix 3.

All samples were placed in a room with climate control, set to 20°C, 65% RH for 24 h prior to testing.

59 sets of samples, with three replicas/samples, plus 6 extra of the un-aged samples, were tested. The samples were clamped centrally and straight in the grips without using pre-tension (slack mounting) (Figure 14). A pre-tension of 0,5 N was then applied and the gauge length corrected accordingly.



Fig. 13. Tensile testing of a sample stained with ashes.

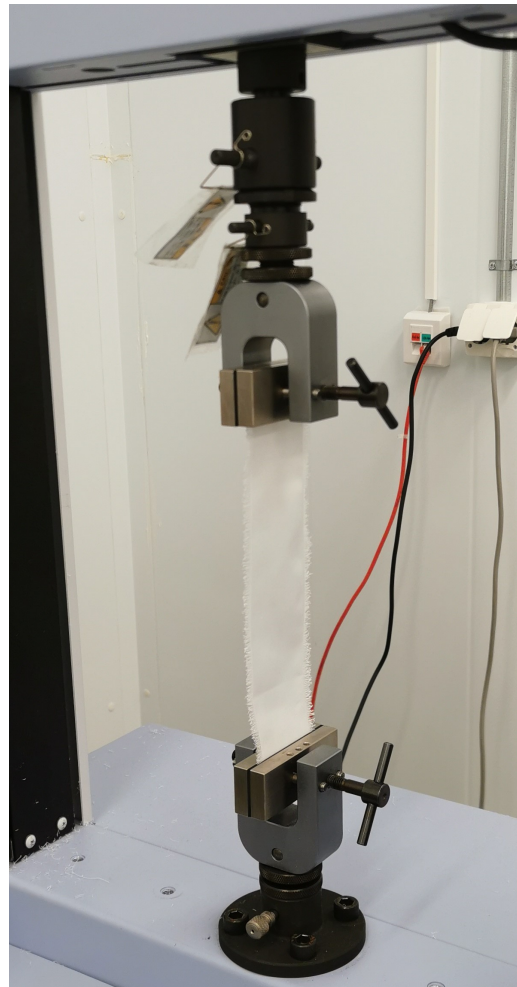


Fig. 14. Tensile testing with slack mounting.

## 6. Results

### 6.1 Stain Removal

#### 6.1.1 Cleaning of Un-aged Stains using Cotton Swabs

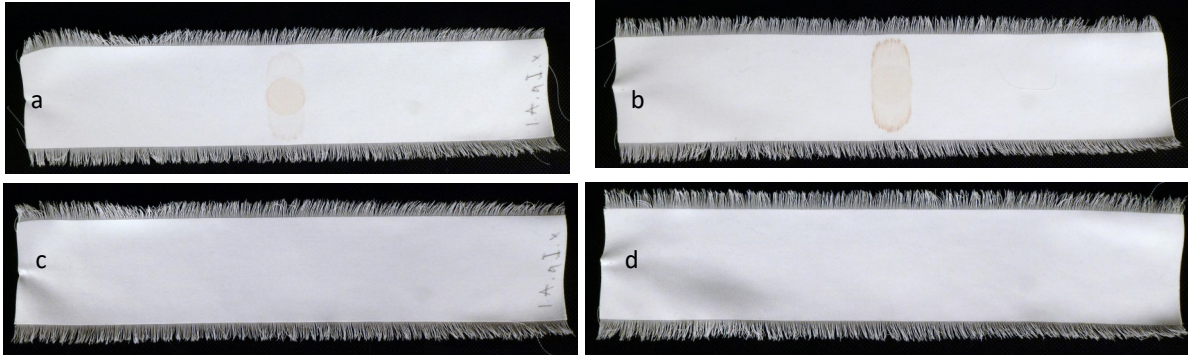


Fig. 15. Sample with un-aged coffee stains treated with Orvus WA Paste and cotton swabs. a) Front before treatment. b) Back before treatment. c) Front after treatment. d) Back after treatment.

The un-aged coffee stains were easy to remove using all four cleaning agents (ethanol was not used on the coffee stains). There were no ring marks left behind that could be seen with the naked eye (Figure 15).

Neither Orvus WA Paste nor Dehypon LS54 removed the un-aged ash stains completely, but they were reduced (Figure 16). Gall soap removed the ash stains so that they could not be seen with the naked eye (Figure 17). Triammonium citrate removed part of the ash stains in a similar manner as Orvus WA Paste and Dehypon LS54.

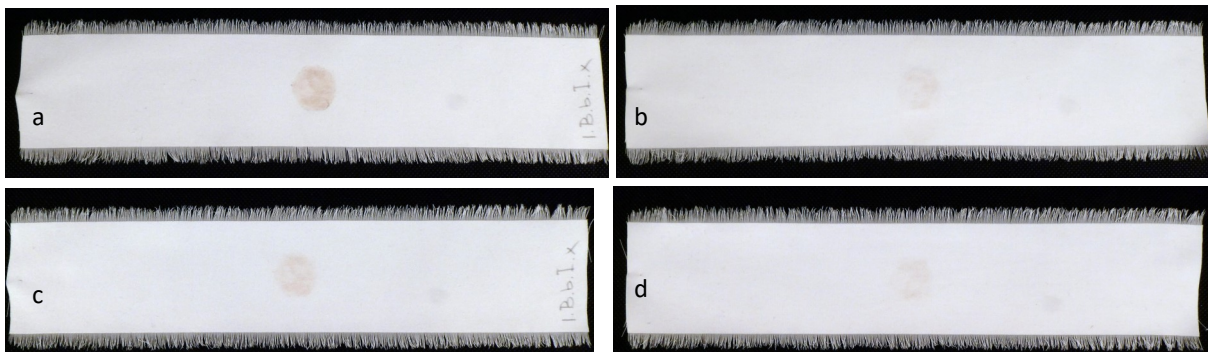


Fig. 16. Sample with un-aged ash stains treated with Dehypon LS54 and cotton swabs. a) Front before treatment. b) Back before treatment. c) Front after treatment. d) Back after treatment.

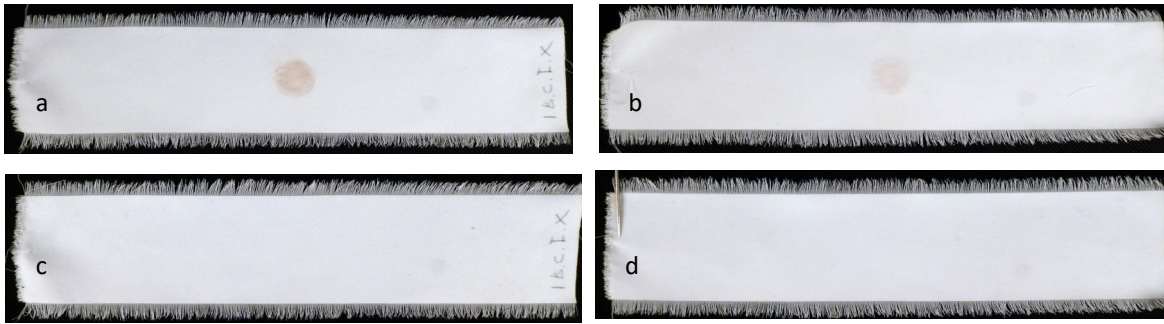


Fig. 17. Sample with un-aged ash stains treated with Gall soap and cotton swabs. a) Front before treatment. b) Back before treatment. c) Front after treatment. d) Back after treatment.

The greasy stains from sebum were somewhat reduced by Orvus WA Paste and Dehypon LS54. Gall soap reduced the stains further, to the point that they were hard to detect with the naked eye (Figure 18). Ethanol changed the appearance of the stains and they turned white and less translucent. It also created a ring mark around the stain (Figure 19).

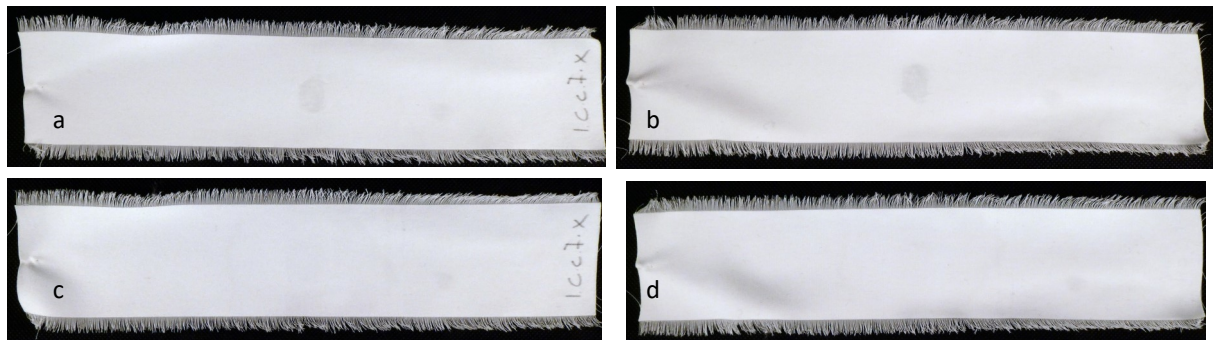


Fig. 18. Sample with un-aged sebum stains treated with Gall soap and cotton swabs. a) Front before treatment. b) Back before treatment. c) Front after treatment. d) Back after treatment.

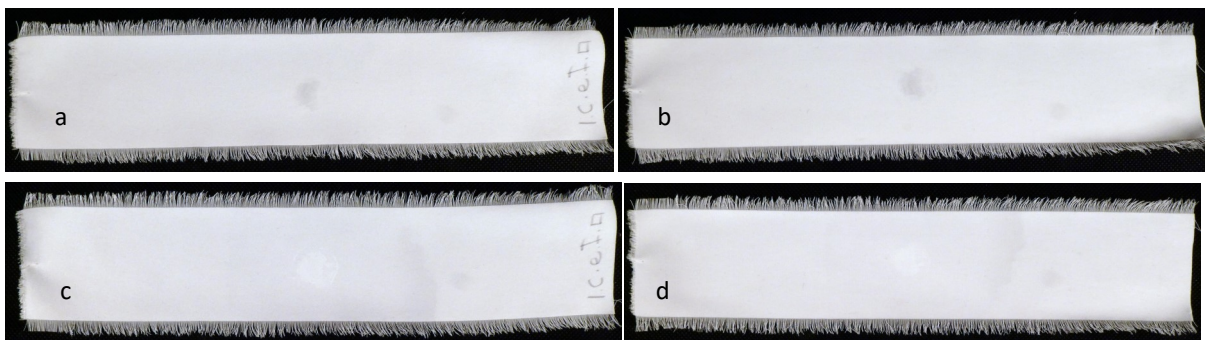


Fig. 19. Sample with un-aged sebum stains treated with Ethanol and cotton swabs. a) Front before treatment. b) Back before treatment. c) Front after treatment. d) Back after treatment.

### 6.1.2 Cleaning of Un-aged Stains on a Suction Table

The coffee stains were quite easy to remove, especially using the three detergents Orvus WA Paste, Dehypon LS54 (Figure 20) and Gall soap. Triammonium citrate did not remove the whole stains, and they were still barely visible, especially on the backside (Figure 21). When examining the four pieces of blotting paper used for removing loosened dirt from each stain, it was clear to the naked eye that the two first used pieces had absorbed material from the coffee stain through capillary forces (Figure 22).

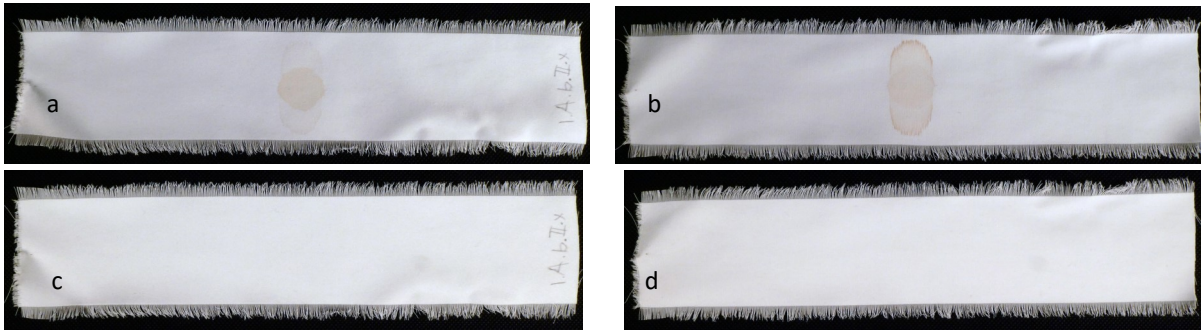


Fig. 20. Sample with un-aged coffee stains treated with Dehypon LS54, using a suction table. a) Front before treatment. b) Back before treatment. c) Front after treatment. d) Back after treatment.

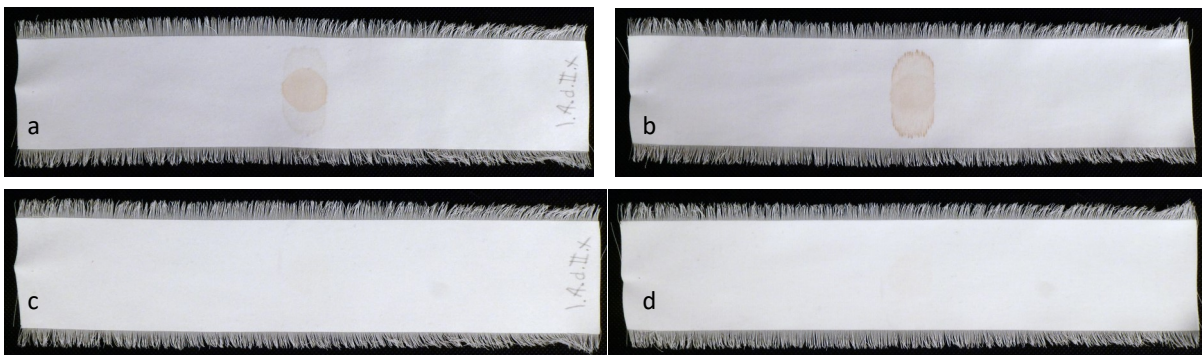


Fig. 21. Sample with coffee stains treated with triammonium citrate. a) Front before treatment. b) Back before treatment. c) Front after treatment. d) Back after treatment.

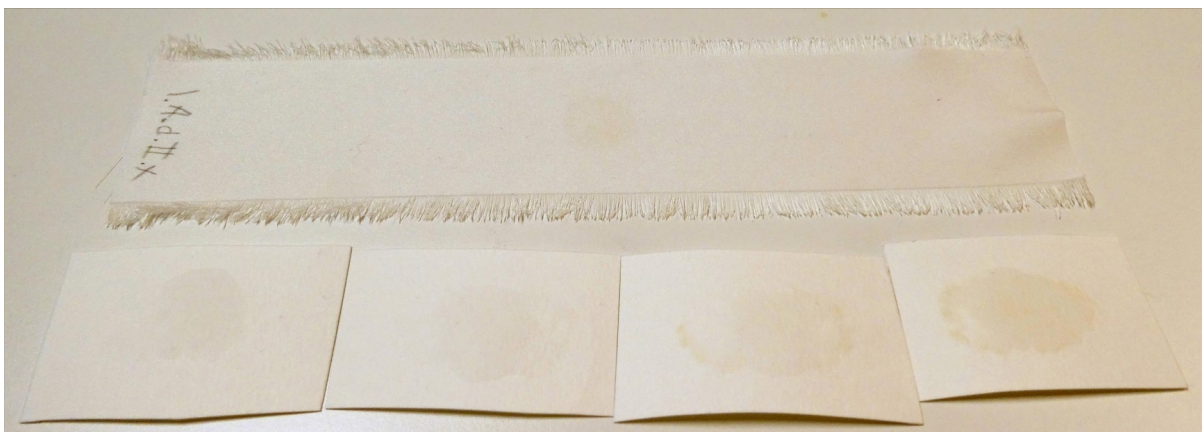


Fig. 22. Blotting paper used for removing loosened dirt during treatment on suction table.

When cleaning the stains made from ashes and deionised water, the blotting papers did not show any residues to the naked eye. The stains cleaned with Orvus WA Paste and Gall soap appeared somewhat lighter to the naked eye, and the ones cleaned using triammonium citrate, even a bit more light (Figure 23). Dehypon LS54 hardly had an effect visible to the naked eye. None of the cleaning agents appeared to be very effective on the ash stains using the method of dripping cleaning agents through the stain.

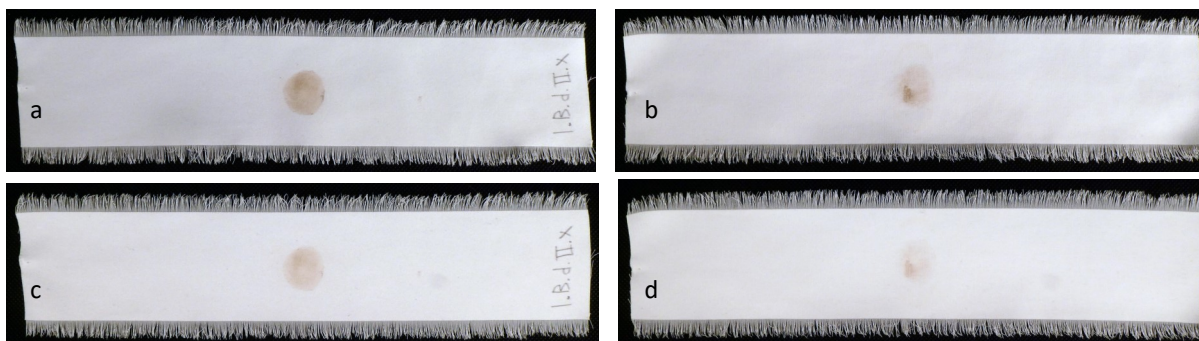


Fig. 23. Sample with un-aged ash stains treated with triammonium citrate, using a suction table. a) Front before treatment. b) Back before treatment. c) Front after treatment. d) Back after treatment.

The greasy stains from sebum were barely reduced by Orvus WA Paste and Dehypon LS54. Gall soap reduced the stains further, but they were still quite visible. When dripping ethanol through the synthetic sebum, the stains changed appearance and became whiter and not as translucent as before cleaning. They also appeared to stay more on top of the fabric than inside it (Figure 24).

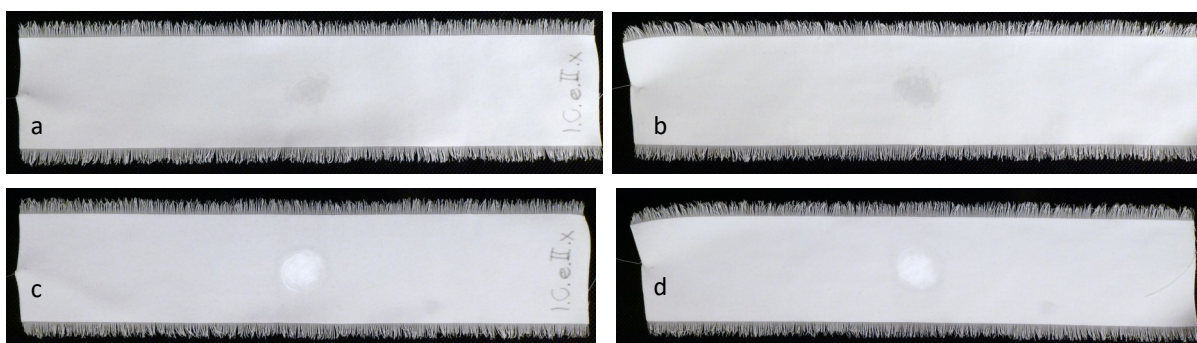


Fig. 24. Sample with un-aged sebum stain treated with Ethanol, using a suction table. a) Front before treatment. b) Back before treatment. c) Front after treatment. d) Back after treatment.

### 6.1.3 Cleaning of Aged Stains using Cotton Swabs

The coffee stains darkened considerably during the ageing (Figures 25 and 26). The coffee stains were darker on the back vs the front. This was due to the staining process with the fabric placed on a non-absorbing cover paper. This made excess coffee stay between the textile and the cover paper and create a higher concentration of coffee at the back.



Orvus WA Paste (Figure 26) and Dehypon LS54 reduced the stains somewhat. The coffee stains were reduced further using Gall soap and triammonium citrate. None of the cleaning agents reduced the stains completely.

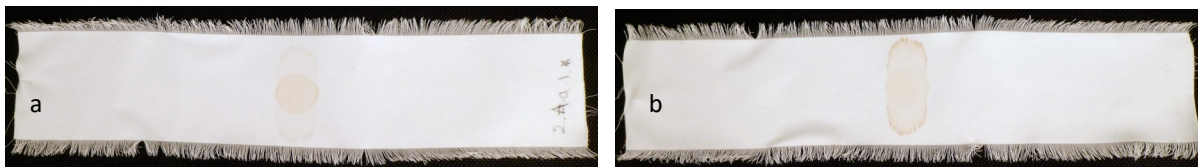


Fig. 25. Coffee stain before ageing. a) Front. b) Back.

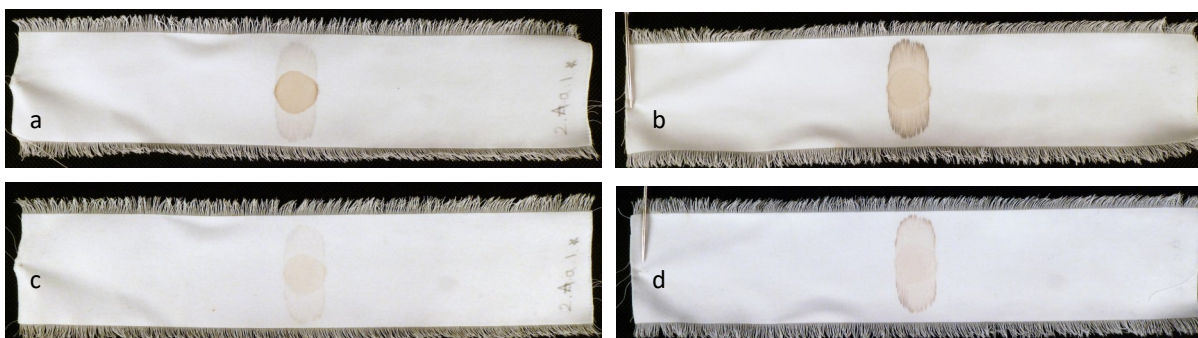


Fig. 26. Coffee stain after ageing (a, b) and after treatment using cotton swabs with Orvus WA Paste (c, d).

The ash stains appeared somewhat lighter after ageing (Figures 27 and 28). Orvus WA Paste, Dehypon LS54 and triammonium citrate reduced the darkness of the stains, but they were still visible to the naked eye. Gall soap reduced the stains so that they could not be seen by the naked eye (Figure 28).

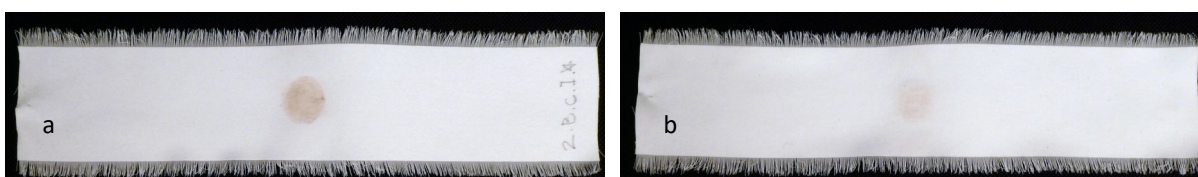


Fig. 27. Ash stain before ageing. a) Front. b) Back.

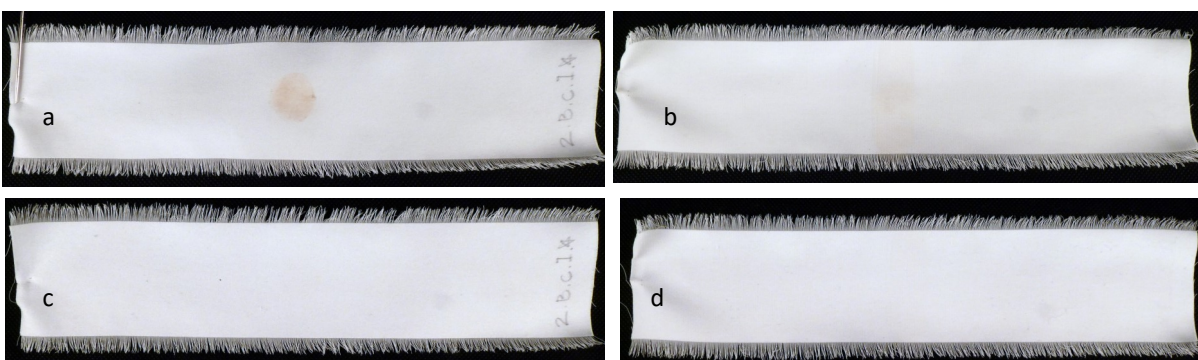


Fig. 28. Ash stain after ageing (a, b) and after treatment with Gall soap (c, d).

The sebum stains changed appearance completely during artificial ageing. They spread in the weft and turned yellow/brown (Figures 29 and 30). Orvus WA Paste reduced the darkness of the stains to some extent. The difference was hardly noticeable to the naked eye when using Dehypon LS54 as the cleaning agent (Figure 31). Gall soap made a clear difference, and lightened the sebum stains considerably (Figure 30). Ethanol made the stains spread in the textile and made it look greasy (Figure 32).

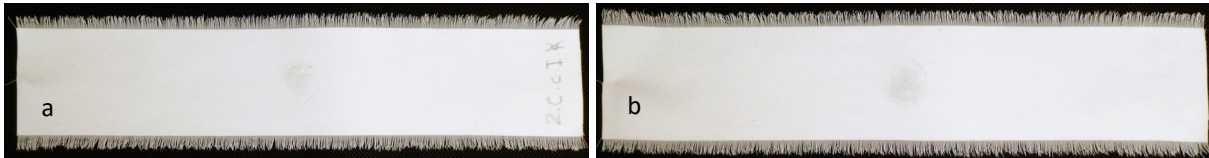


Fig. 29. Sebum stain before ageing. a) Front. b) Back.

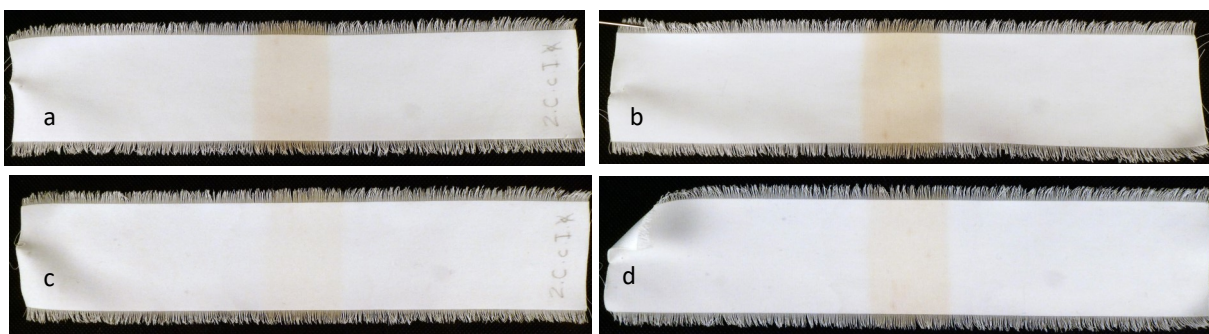


Fig. 30. Sebum stain after ageing (a, b) and after treatment with Gall soap (c, d).

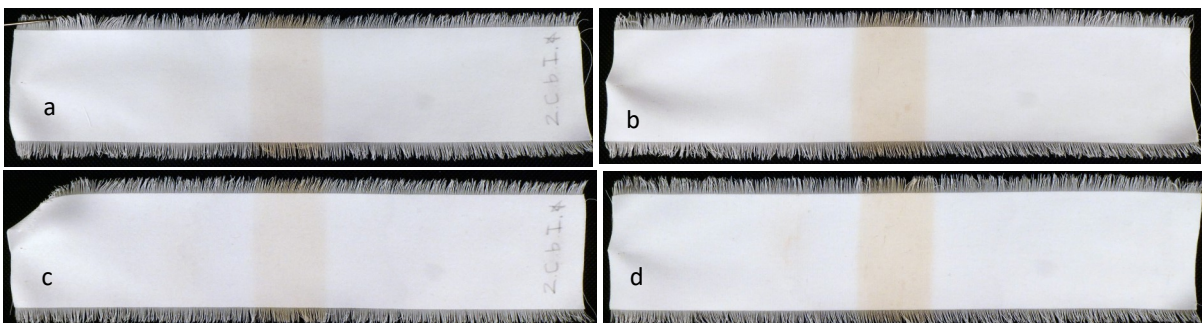


Fig. 31. Aged sample with sebum stain treated with Dehypon LS54 and cotton swabs. a) Front before treatment. b) Back before treatment. c) Front after treatment. d) Back after treatment.

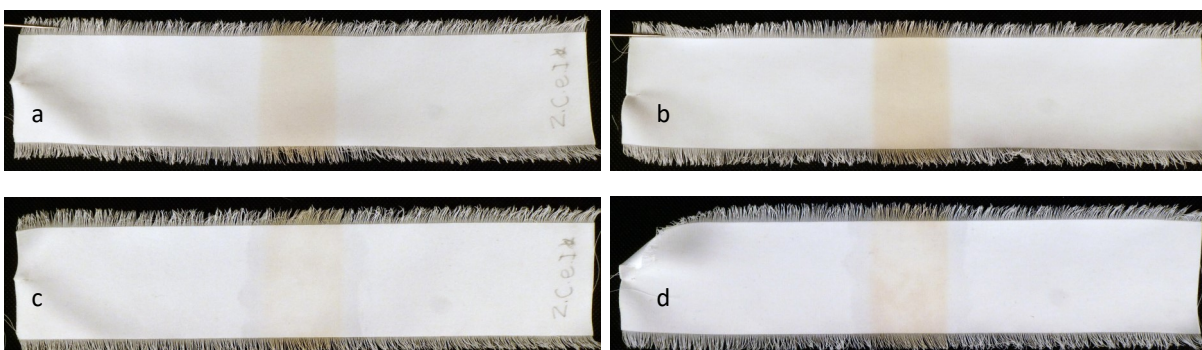


Fig. 32. Aged sample with sebum stain, treated with cotton swabs and Ethanol. a) Front before treatment. b) Back before treatment. c) Front after treatment. d) Back after treatment.

#### 6.1.4 Cleaning of Aged Stains on a Suction Table

The coffee stains darkened considerably during the ageing process. Orvus WA Paste reduced the darkness of the stains to some extent. It was more obvious on the front compared to the back. Dehypon LS54 barely reduced the stains. Ring marks were formed around the area cleaned using Dehypon LS54 (Figure 33). Gall soap reduced the stains considerably but they were still visible to the naked eye. Part of a ring mark was created at the side of one stain cleaned with Gall soap (Figure 34). Triammonium citrate reduced the stains more than Orvus WA Paste but less than Gall soap.

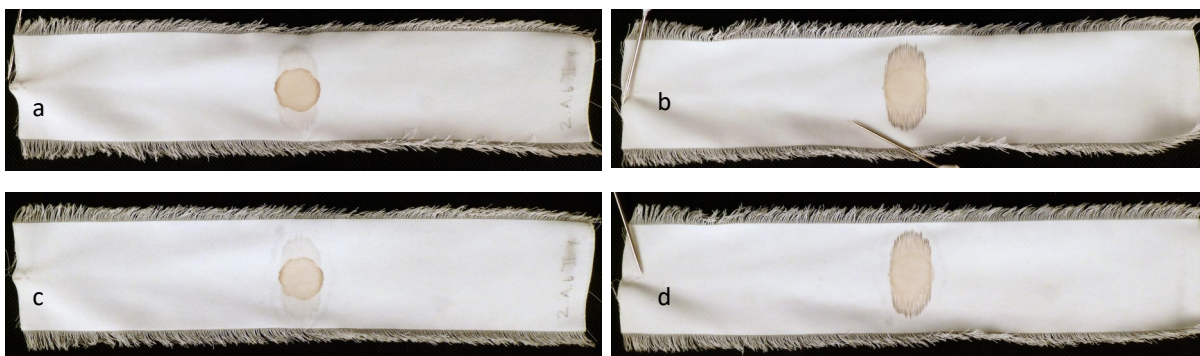


Fig. 33. Sample with coffee stain treated with Dehypon LS54. a) Front before treatment. b) Back before treatment c) Front after treatment. d) Back after treatment.

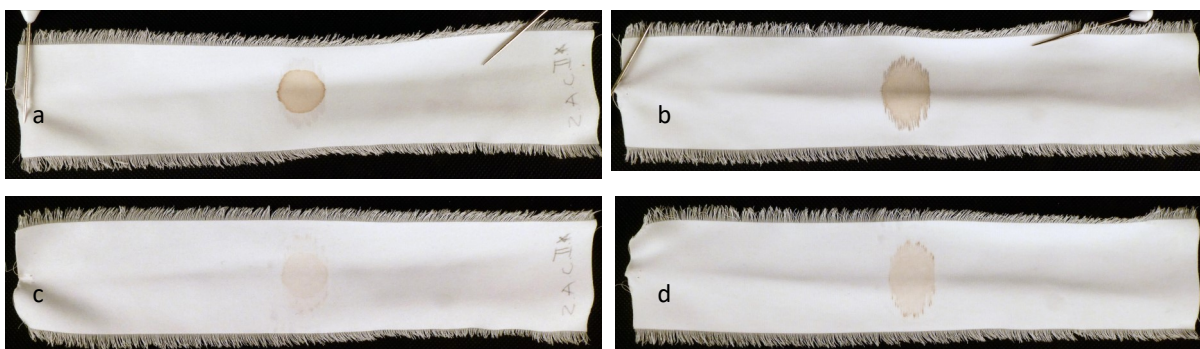


Fig. 34. Sample with coffee stain treated with Gall soap. a) Front before treatment. b) Back before treatment c) Front after treatment. d) Back after treatment.

The ash stains appeared somewhat lighter after ageing. The stains cleaned with Orvus WA Paste appeared somewhat reduced to the naked eye and the ones treated with Dehypon LS54 and triammonium citrate (Figure 35) appeared a bit more reduced. The stains treated with Gall soap appeared more reduced than the others.

The sebum stains changed appearance completely during artificial ageing. They spread in the weft and turned darker/yellowish. Trials were done in an effort to understand whether the heat caused the yellowing of the stain or if it was caused by all factors of ageing. A heat spatula was used on un-aged sebum. The spatula was set at 95°C, the same temperature as in the climate chamber. The stain did not turn yellow.

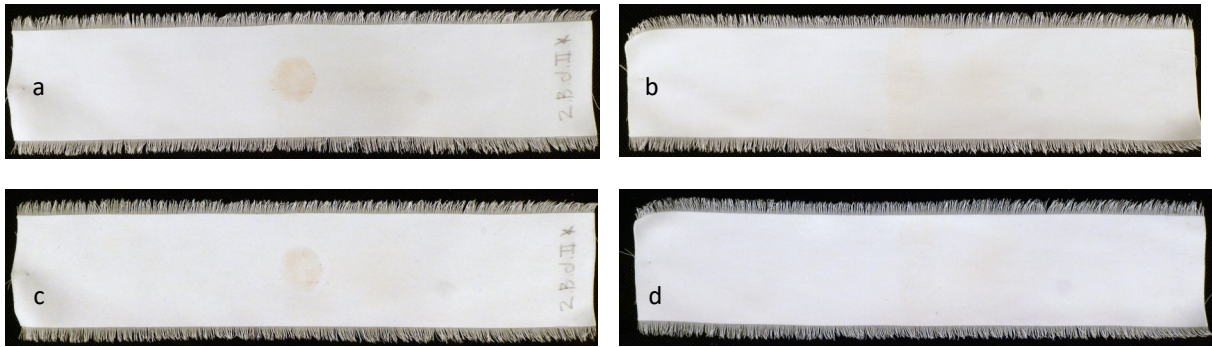


Fig. 35. Sample with ash stain treated with triammonium citrate. a) Front before treatment. b) Back before treatment c) Front after treatment. d) Back after treatment.

The aged sebum stains treated with Orvus WA Paste and with Dehypon LS54 appeared to have been slightly reduced. On two of the samples treated with Orvus WA Paste and all samples treated with Dehypon LS54 (Figure 36), ring marks were created along one or both sides of the stain. The samples treated with Gall soap appeared slightly more reduced than the others. Ring marks could be seen on two of them as well. Ethanol created very different stains than before treatment. Parts of the stains had a whitish appearance and other parts seemed more transparent. The ethanol spread in the fabric and created new outer borders (Figure 37).

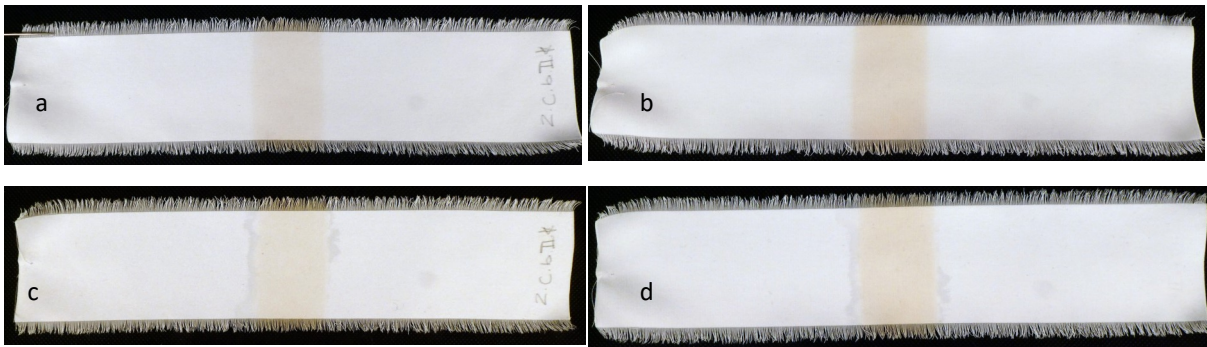


Fig. 36. Sample with sebum stain treated with Dehypon LS54 on a suction table. a) Front before treatment. b) Back before treatment c) Front after treatment. d) Back after treatment.

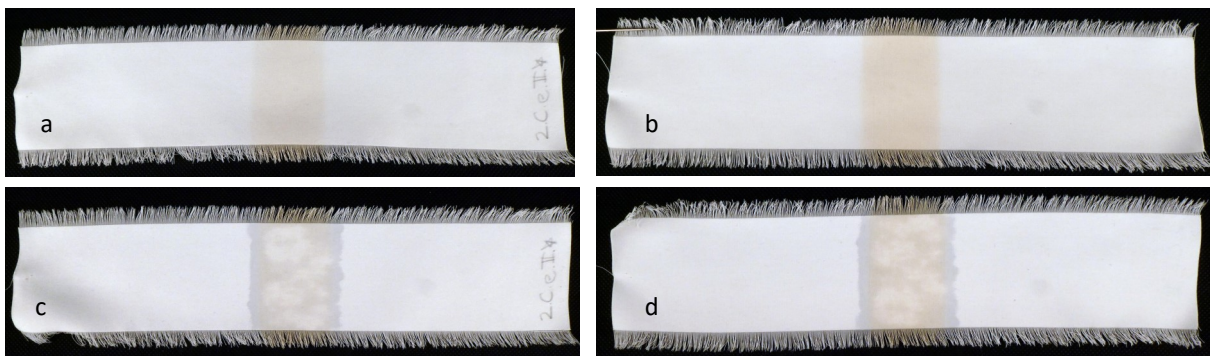


Fig. 37. Sample with sebum stain treated with Ethanol. a) Front before treatment. b) Back before treatment c) Front after treatment. d) Back after treatment.

## 6.2 Colour Measurement

The coffee stains and the ash stains were not uniform in colour, they had darker parts and lighter parts (Figures 25 and 27). This made the results of the colour measuring inconsistent. Hence three measurements were taken on each stain, with a small movement of the spectrophotometer in between each measurement.

The results from the colour measurements are shown in bar graphs. Each bar is showing an average  $\Delta E$  of 9 measurements taken on three replicas of the same type of stain, treated with the same cleaning agent and cleaning method. The bars show  $\Delta E$  before and after cleaning. In the naming of the samples, I show the cotton swab method and II the suction table method. For explanation of  $\Delta E$ ,  $\Delta L$ ,  $\Delta a$  and  $\Delta b$  see chapters 2.8.1 and 5.10. Bar graphs with  $\Delta E$ ,  $\Delta L$ ,  $\Delta a$  and  $\Delta b$  can be found in appendix 4.

### 6.2.1 Un-aged Coffee Stains

The colour measurement shows that the un-aged coffee stains were not unanimous in colour before cleaning (Figure 38). The total colour difference between the un-aged stains were 2  $\Delta E$ . When using cotton swabs to clean the stains, Orvus WA Paste and Dehypon LS54 had a colour difference of 10  $\Delta E$  between before and after cleaning. Gall soap had a difference of 11  $\Delta E$  and triammonium citrate 9  $\Delta E$ .

When using the method of dripping the cleaning agent through the stain on a suction table, the difference in  $\Delta E$  was generally a bit higher. Orvus WA Paste and Gall soap had a difference of 11  $\Delta E$ , Dehypon LS54 10,5  $\Delta E$  and triammonium citrate 10 $\Delta E$ .

Both  $\Delta L$  (lightness) and  $\Delta a$  (red-green colours) are very close to 0 in all cleaned samples, which tells us that they are very similar to the un-treated, un-aged reference sample. In this case  $\Delta b$ , with its yellow tone, is the part that mainly affects  $\Delta E$  (Appendix 4).

### 6.2.2 Aged Coffee Stains

The colour measurement shows that the aged coffee stains were more unanimous in colour before cleaning than the un-aged (Figure 38).  $\Delta E$  differs between the samples with 1 unit.

The changes in  $\Delta E$  when using cotton swabs to clean the stain were: Orvus WA Paste, 8  $\Delta E$ , Dehypon LS54 8  $\Delta E$ , Gall soap 9  $\Delta E$  and triammonium citrate 7,5 $\Delta E$ .

There were bigger variations in  $\Delta E$  when the stains were treated on a suction table and there were less colour differences than with cotton swabs. Gall soap had the highest differ-

ence of the samples cleaned on a suction table, with  $\Delta E$  8,5, almost the same as when using cotton swabs. Orvus WA Paste and triammonium citrate both had the same colour difference of 6,5  $\Delta E$ . Dehypon LS54 had the lowest with 4  $\Delta E$ .

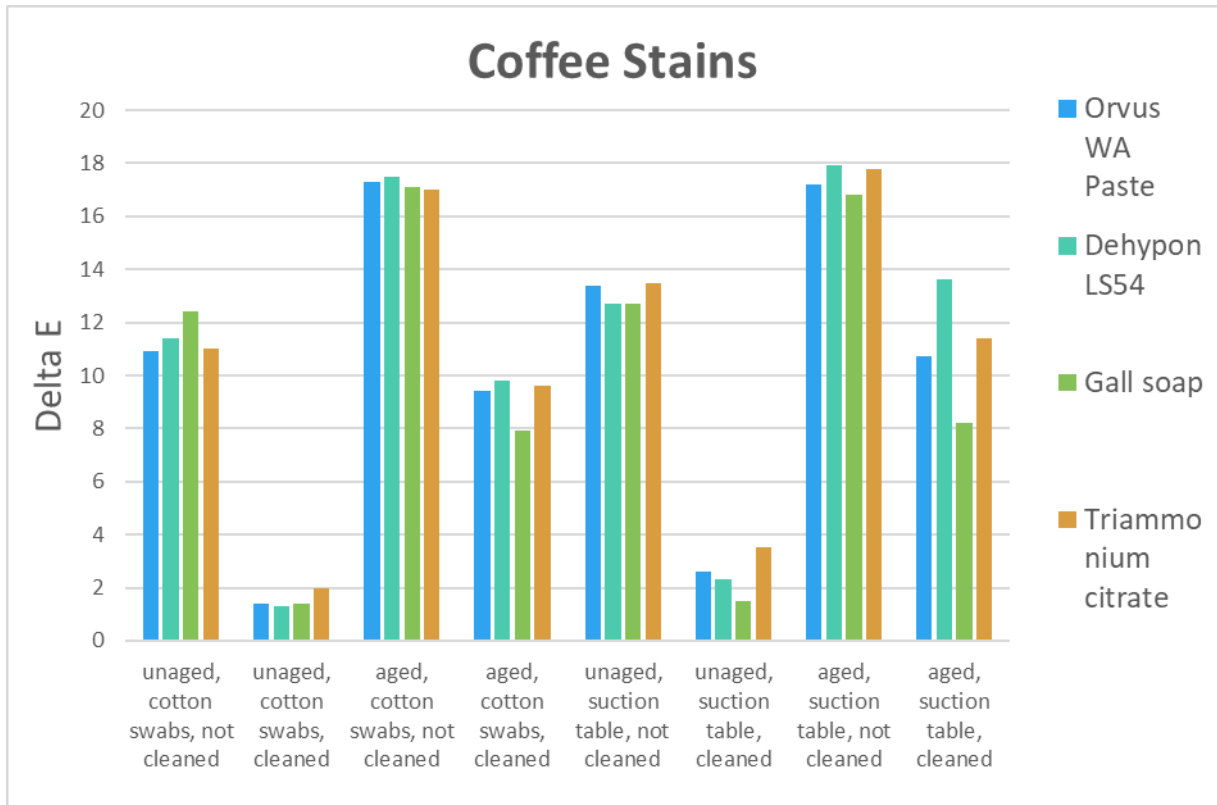


Fig. 38.  $\Delta E$  for coffee stains before and after cleaning.

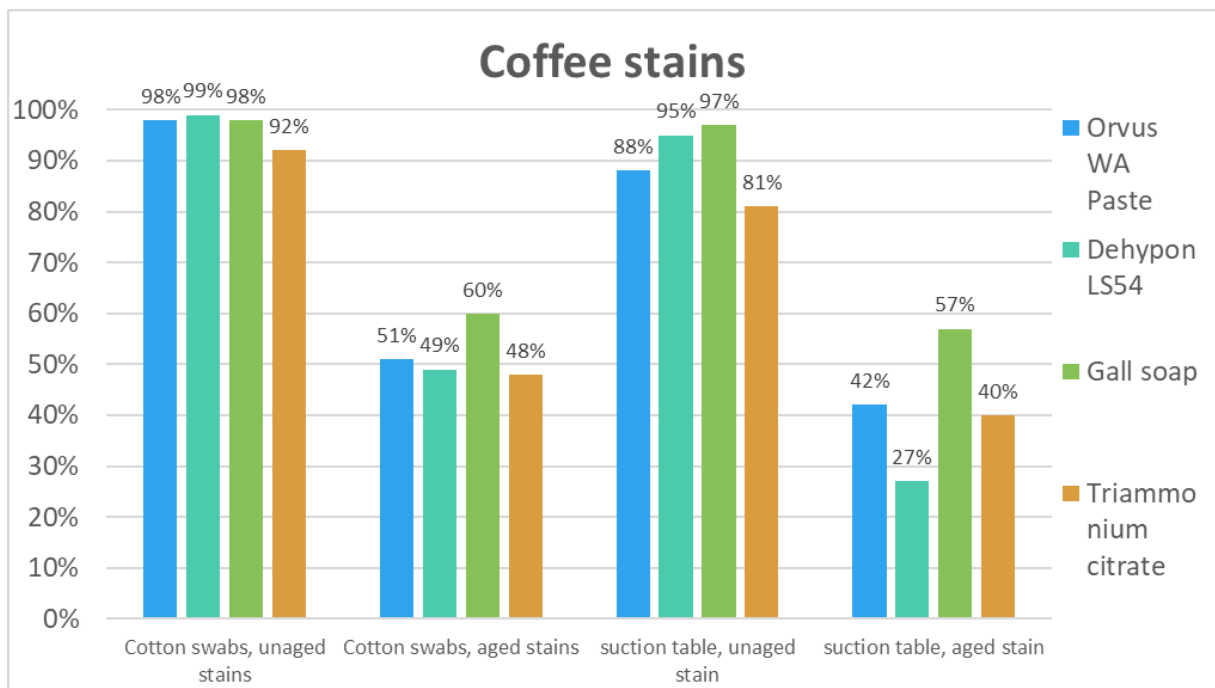


Fig. 39. The bars show how many percent the coffee stains were reduced during cleaning.

Figure 39 shows how many percent the average of the stains are reduced, compared to the reference sample aged the same time as the measured sample.

All treated stained areas were darker than the reference sample. Gall soap was closest in lightness to the reference sample with  $-2 \Delta L$  (Appendix 4).

### 6.2.3 Un-aged Ash Stains

The colour measurement showed that the un-aged ash stains were varied in colour before cleaning (Figure 40). The total colour difference between the un-aged, untreated stains was  $9 \Delta E$ .

When using cotton swabs, there were huge differences in how much  $\Delta E$  changed. Gall soap had the highest difference in  $\Delta E$ , with 14 units changed between before and after cleaning. Triammonium citrate had the second highest difference with  $8,5 \Delta E$ , and Orvus WA Paste was close with  $7,5$ . Dehypon LS54 had the lowest  $\Delta E$  difference with  $5 \Delta E$ .

The colour differences changed a lot when treating the ash stains on a suction table. Here Dehypon LS54 had the largest colour difference with  $7,5 \Delta E$ . Orvus WA Paste had  $5 \Delta E$  and triammonium citrate slightly less with  $4,5$ . Gall soap had the smallest colour difference with  $4 \Delta E$ .

The stains treated with cotton swabs and Gall soap were the ones closest to the reference sample in colour after cleaning.  $\Delta E$  was  $1,4$ , which is very close to the reference sample aged once ( $1,2 \Delta E$ ) (Figure 44, Table 3).

### 6.2.4 Aged Ash Stains

The colour measurement showed that the aged ash stains were varied in colour before cleaning (Figure 40). The total colour difference between the aged, untreated stains was  $3,5 \Delta E$ .

The stains treated with cotton swabs and Orvus WA Paste, Dehypon LS54 and triammonium citrate showed similar colour differences between untreated and treated stains. Orvus WA Paste had a difference of  $9 \Delta E$ , and the other two  $8,5 \Delta E$ . The differences when using Gall soap were bigger with a difference of  $13 \Delta E$ .

When using the suction table as a cleaning method, the differences were quite similar with all four cleaning agents. Gall soap had a difference of  $5 \Delta E$  and the other three  $4,5 \Delta E$ .

The stains treated with Gall soap and cotton swabs were the one closest in colour to the

reference sample. They had an average colour difference of 2,2  $\Delta E$  in comparison to 1,7  $\Delta E$  in the twice aged reference samples (Table 3).

Figure 41 shows how many percent the average of the stains are reduced, compared to the reference sample aged the same time as the measured sample.

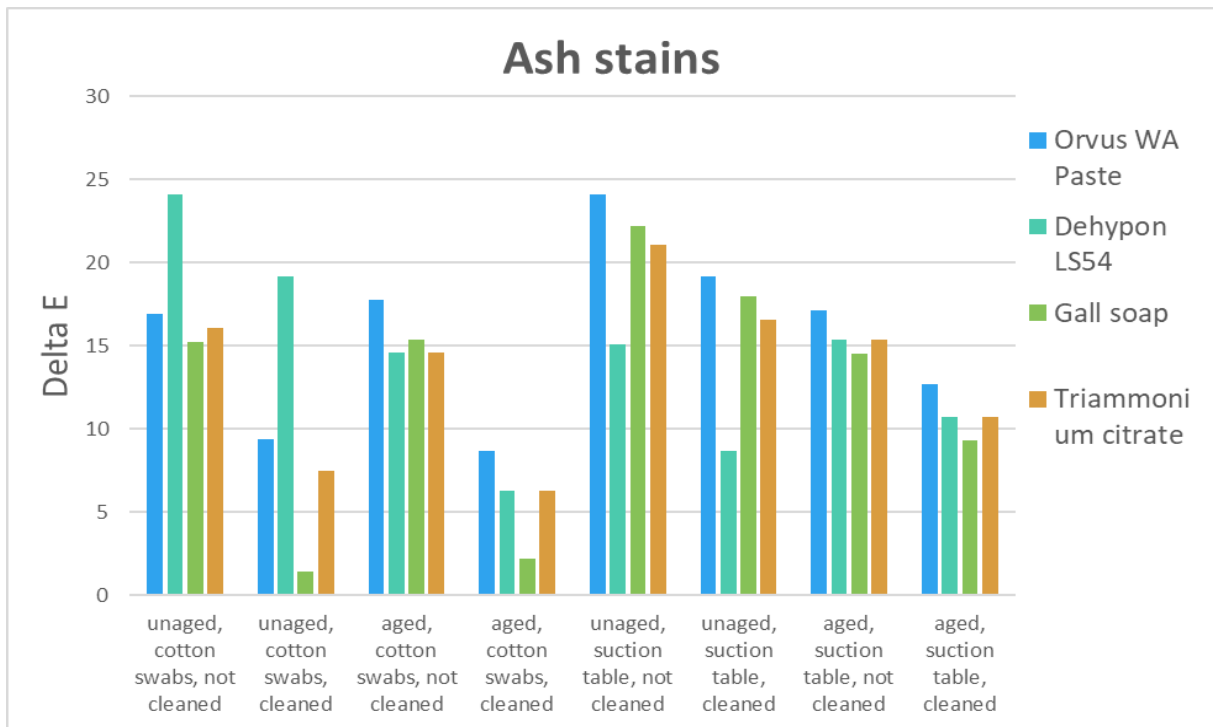


Fig. 40.  $\Delta E$  for ash stains before and after cleaning.

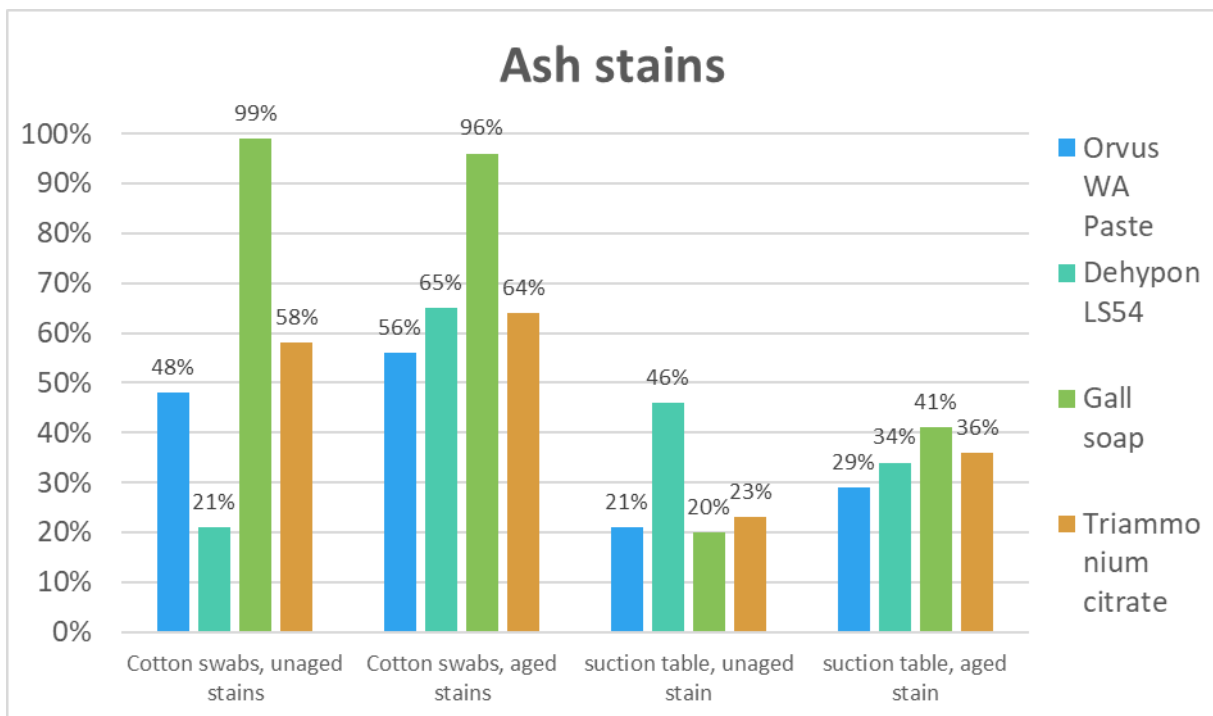


Fig. 41. The bars show how many percent the ash stains were reduced during cleaning.



### 6.2.5 Un-aged Sebum Stains

Un-aged sebum did not affect the colour of the textile very much. The colour differences in between the un-treated samples were 1,2  $\Delta E$  (Figure 42). The colour differences between stained and cleaned stains treated with Orvus WA Paste, Dehypon LS54 and Gall soap were very small, between 0,1 and 0,5  $\Delta E$ . The stains treated with Ethanol had a somewhat larger change of 1,2 and 1,3  $\Delta E$ .

Four of the stains were lighter than the reference sample before cleaning and 5 of them were darker after cleaning (Appendix 4). As the colour difference is very small, the differences are probably not noticeable for the naked eye.

### 6.2.6 Aged Sebum Stains

The measurements did not show a large colour difference in between the stained samples before cleaning. The differences were 2  $\Delta E$  (Figure 42).

The changes in  $\Delta E$  when using cotton swabs to clean the stain were: Orvus WA Paste 3  $\Delta E$ , Dehypon LS54 1,5  $\Delta E$ , Gall soap 5  $\Delta E$  and ethanol 4  $\Delta E$ .

When using the suction table the changes were mostly smaller. Orvus WA Paste had a colour difference of 2  $\Delta E$ , Dehypon LS54 had the same as when treated with cotton swabs, 1,5

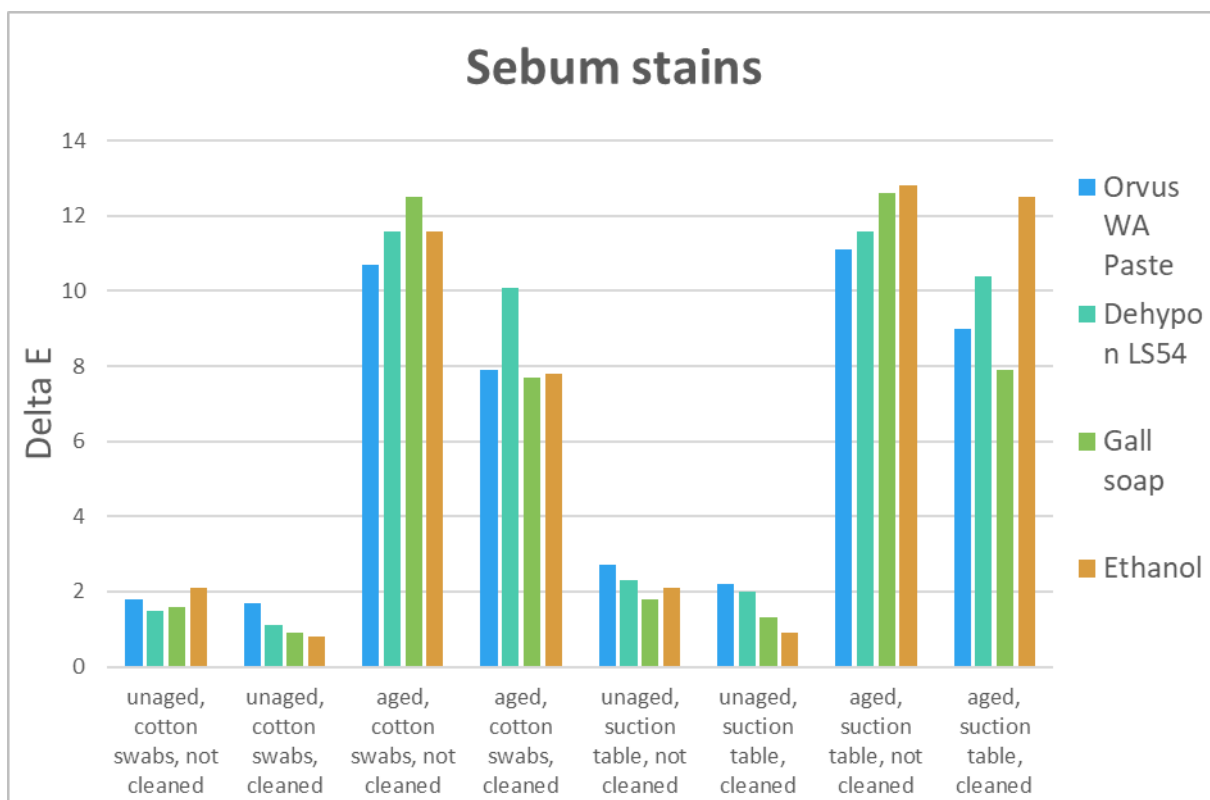


Fig. 42.  $\Delta E$  for sebum stains before and after cleaning.

$\Delta E$ , Gall soap 4,5  $\Delta E$  and ethanol a bit less difference with 0,5  $\Delta E$ .

Figure 43 shows how many percent the average of the stains are reduced, compared to the reference sample aged the same time as the measured sample. The unaged sebum stains were so similar in colour to the polyester fabric, that the colour measurements could not be used in understanding the efficiency of the different cleaning agents and cleaning methods.

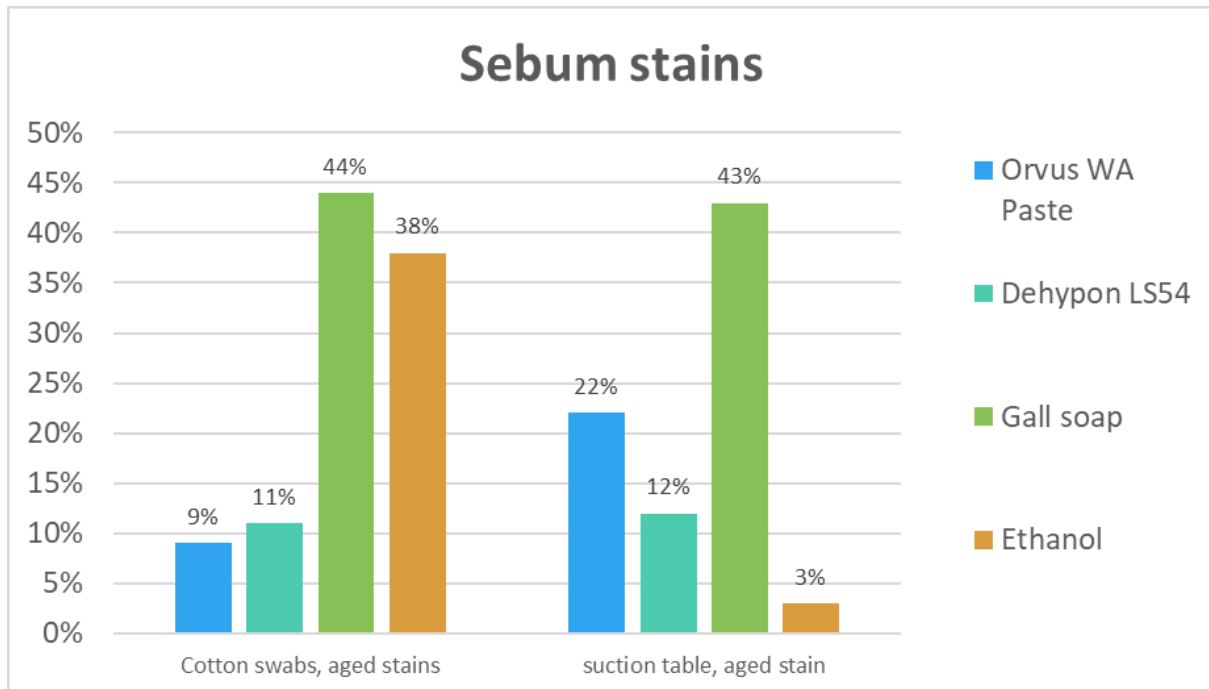


Fig. 43. The bars show how many percent the sebum stains were reduced during cleaning.

### 6.2.7 Aged Polyester

Figure 44 shows the total colour differences and lightness differences of the samples aged once and twice compared to an un-aged sample. The samples aged once had a very similar lightness as the un-aged sample and a total colour difference of 1-1,4  $\Delta E$ , not perceptible to the naked eye and perceptible through close observation. The samples aged twice were somewhat darker and the total colour difference was between 1-2  $\Delta E$ , perceptible to the naked eye through close observation.

### 6.2.8 $\Delta E$

Table 3 shows the average  $\Delta E$  of the un-aged, aged once and aged twice, reference samples. It also shows the average  $\Delta E$  of the stained samples, not cleaned.

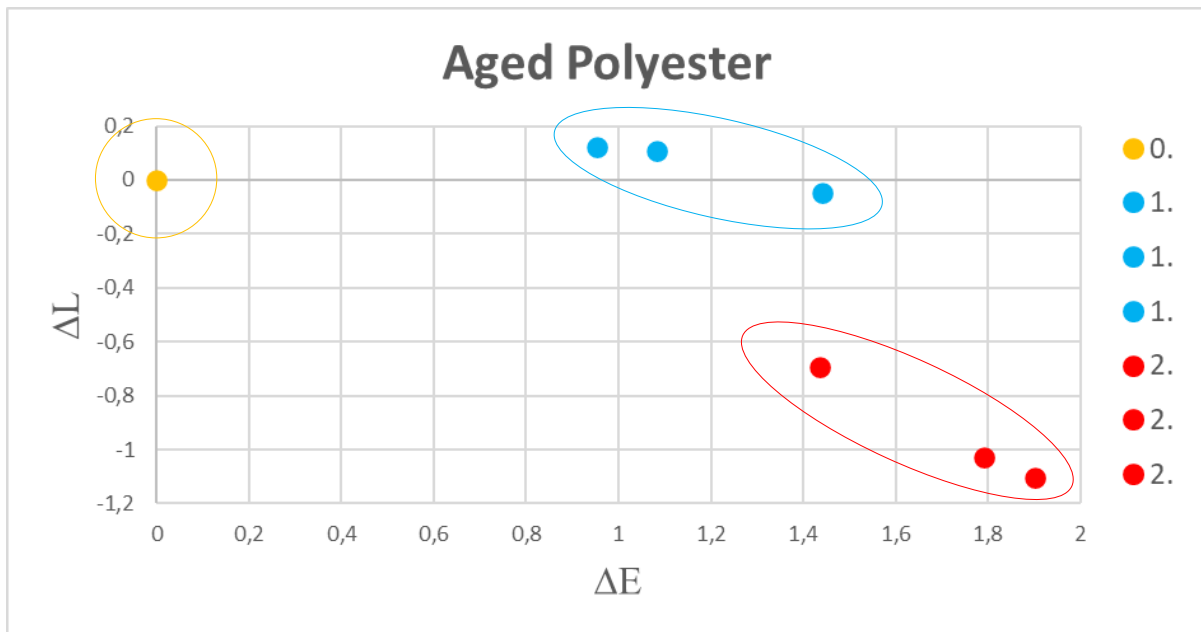


Fig. 44. Total colour difference and lightness in unaged sample (0), samples aged once (1) and twice (2).

Table 3. Average colour of samples not cleaned and of reference samples,  $\Delta E$

	Not aged	Aged once	Aged twice
Stained with coffee after first ageing	-	11,9	16,4
Stained with ashes after first ageing	-	17,1	14,9
Stained with sebum after first ageing	-	1,3	13,2
Reference samples, not stained	0,0	1,2	1,7

### 6.3 Infrared Spectroscopy

The spectra from the measurements are presented as an overlay spectrum (Figure 45). The black line shows the spectrum of the un-aged polyester. The blue line shows the spectrum of polyester aged once. The red line shows the spectrum of the polyester aged twice.

When comparing the three spectra, no distinctive differences can be noticed. There were no clear molecular changes shown in the two aged spectra (blue and red).

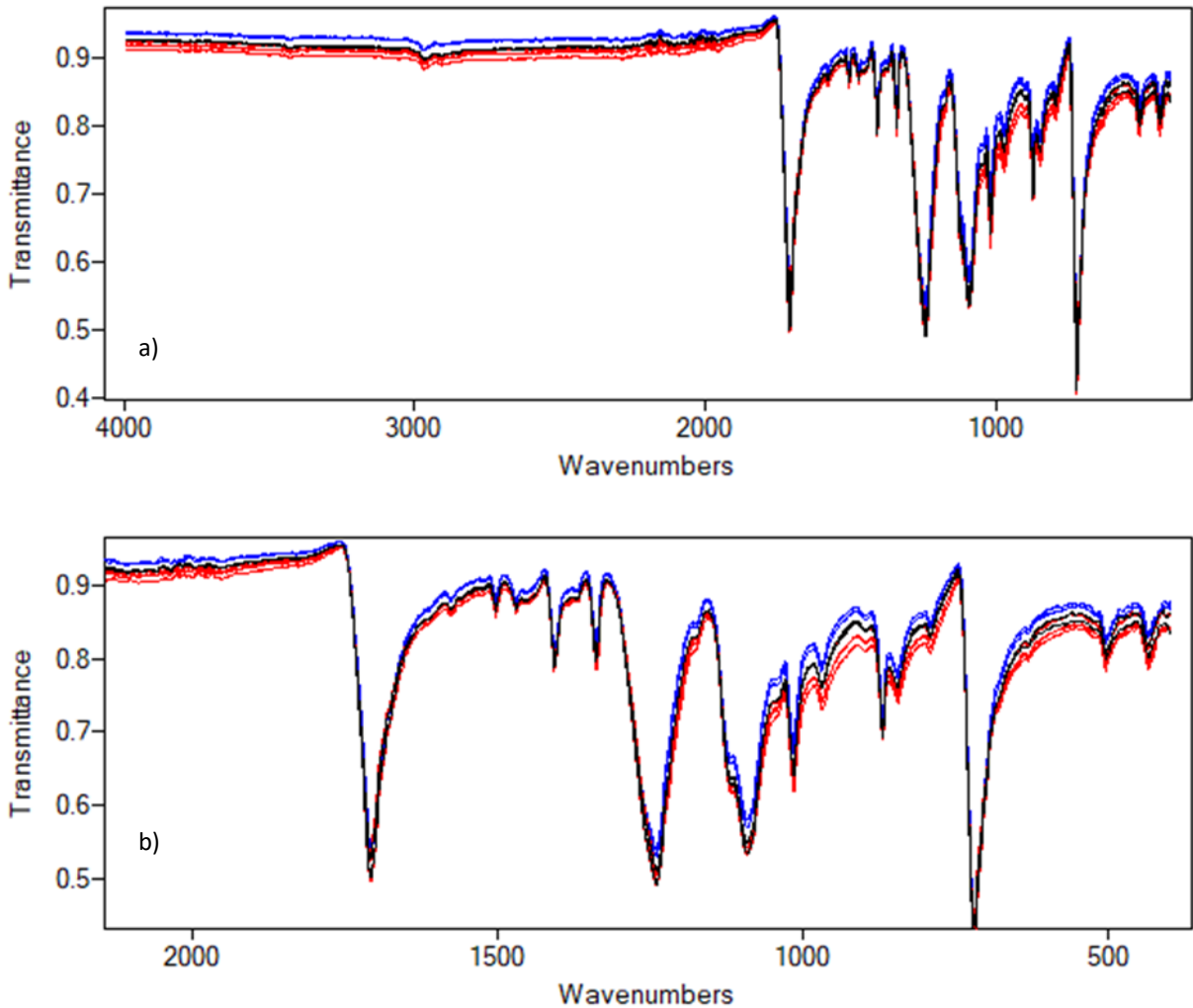


Fig. 45. a) FTIR overlay spectrum of polyester fabric b) with enhanced fingerprint area. Black = un-aged samples, blue = samples aged once, and red = samples aged twice.

## 6.4 Tensile Testing

The results from the tensile testing are shown in scatter plots displaying stress vs strain, where stress is the breaking force/mm<sup>2</sup> and strain is the elongation shown in %. Each plot shows samples aged once and twice and treated with cotton swabs (1 or 2 and I), stained but not cleaned and aged once and twice (1 or 2), and aged once and twice and treated on a suction table (1 or 2 and II). The blue/green dots show samples aged once and the red/orange/pink dots show samples aged twice. Each sample is replicated three times, displaying three dots in the scatter plot. The ISO standard 13934-1, dictates that each set should consist of at least five test specimens. As this study only has three test specimens, the tensile test result can not be seen as scientifically reliable. When performing tensile testing it is preferable to stain the whole sample instead of, as has been done in this study, stain a section of the sample, as it will give more reliable results. If these experiments were to be repeated, it is recommended to prepare different versions of samples for stain removal with colour measurements and for tensile testing.

### 6.4.1 Coffee

69 samples stained with coffee and cleaned with Orvus WA Paste, and not cleaned, tore at the top end of the sample. Two tore at the bottom and one below the stain (Figure 46).

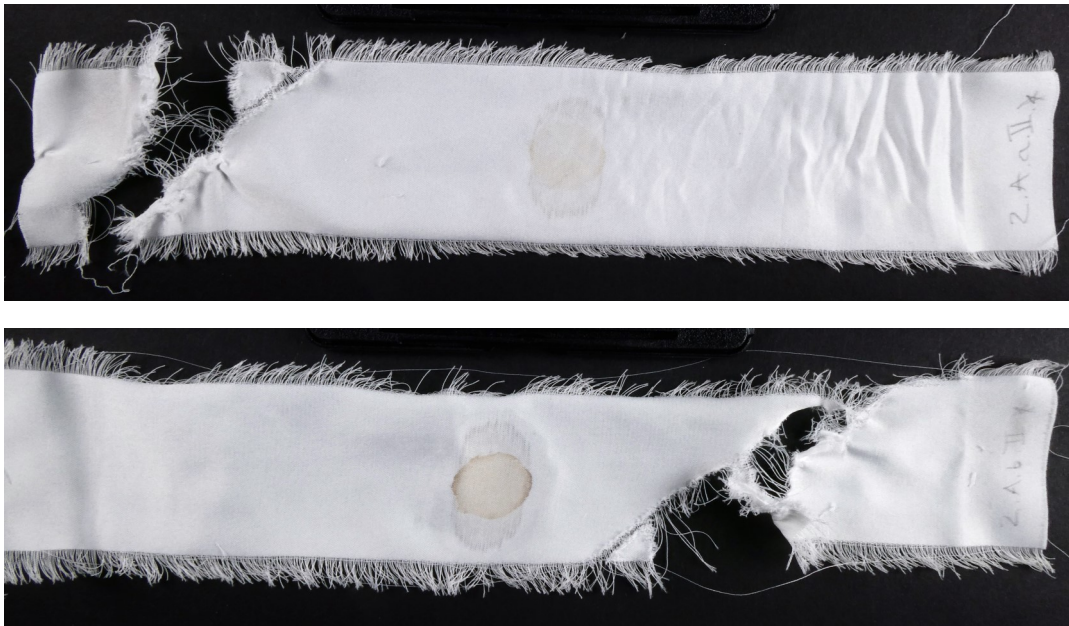


Fig. 46. Sample stained with coffee, one torn at the top and the other at the bottom of the sample.

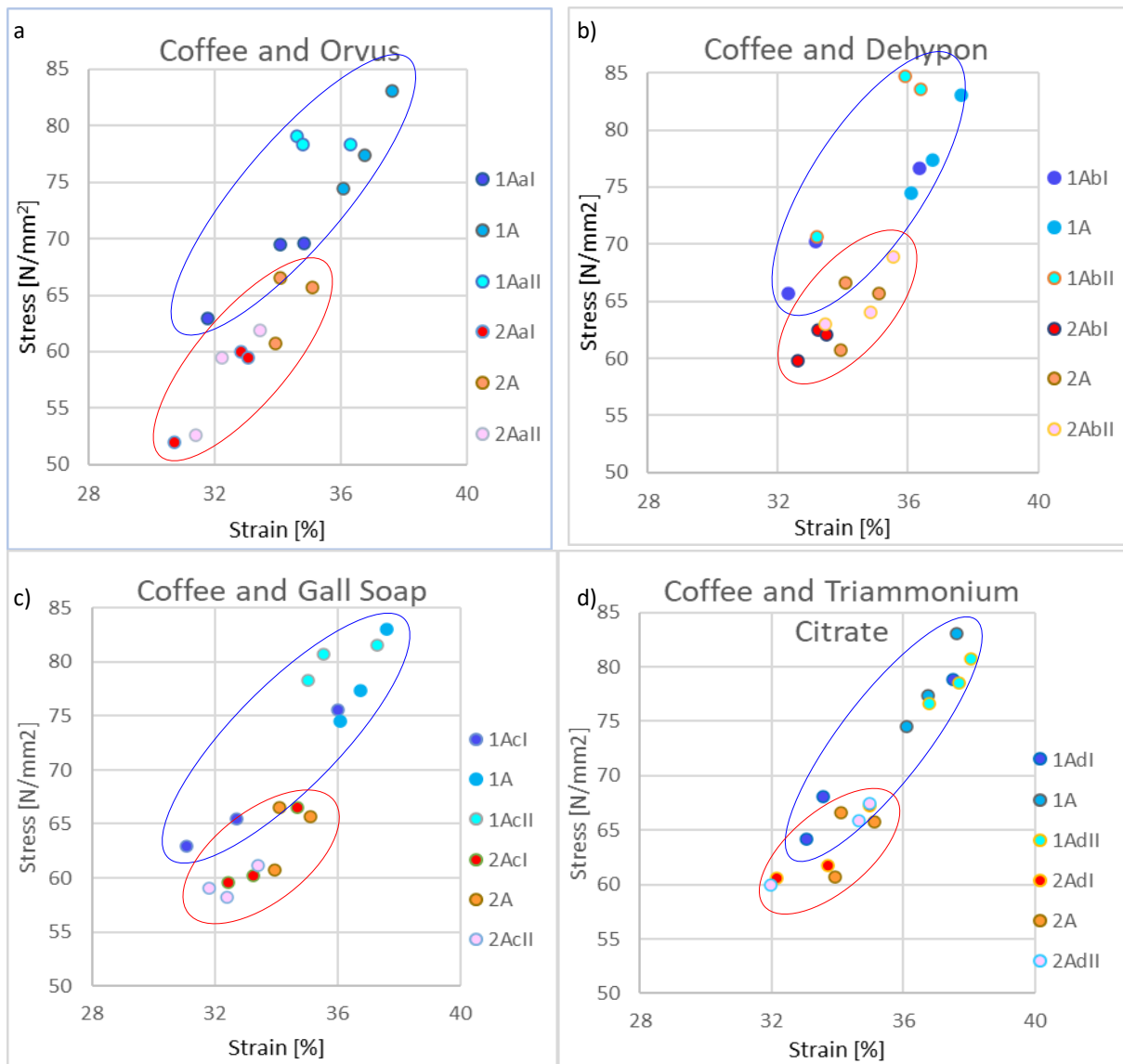


Fig. 47. Scatter plots showing results from tensile testing on coffee stains treated with a) OrvusWA Paste, b) Dehypon LS54, c) Gall soap, and d) Triammonium citrate. Aged once (1), aged twice (2), cotton swabs (I) and suction table (II).

Generally, both stress and strain were higher at breakage with the samples aged once (blue/green dots) compared to the samples aged twice (red/orange/pink dots). However, the breakage point of some of the samples with stains aged once and treated with cotton swabs were in the proximity of samples aged twice.

The breaking point of the coffee stained samples (Figure 47) aged once and treated on a suction table were mostly gathered close to the samples aged once and not cleaned. One sample treated with Dehypon LS54 differed, and had lower stress and shorter strain than the others (Figure 47 b).

The samples aged twice and treated with Orvus WA Paste were more spread out than the samples aged twice and treated with the other cleaning agents (Figure 47 a). Two of them

had a lower stress and shorter strain than all the other samples except from ashes aged twice and treated with Dehypon LS54 on the suction table (Figure 50 b).

#### 6.4.2 Ashes

The samples aged once, stained with ash and cleaned/not cleaned tore in similar fashion as the samples stained with coffee. Of the 36 samples 34 tore at the top of the sample, one at the bottom and one through the stain (Figure 48).

Many of the samples stained with ashes and then aged a second time had a different behaviour. 18 of the samples tore through the stain and 18 were torn at the top of the sample. All samples stained with ash and treated with triammonium citrate tore at the middle of the stain (Figure 49).



Fig. 48. Sample aged once and then stained with ash and cleaned with Dehypon. The tear from the tensile testing is situated at the top of the sample.



Fig. 49. One of the 18 samples torn straight through the stain. The sample is aged twice, and was stained with ash previous to and cleaned after the second ageing.

The scatter plot showed that the samples aged once generally had their breaking point at a higher stress and strain than the samples aged twice (Figure 50). The stains aged once and treated with cotton swabs broke the pattern, as some of them had a lower breaking point,

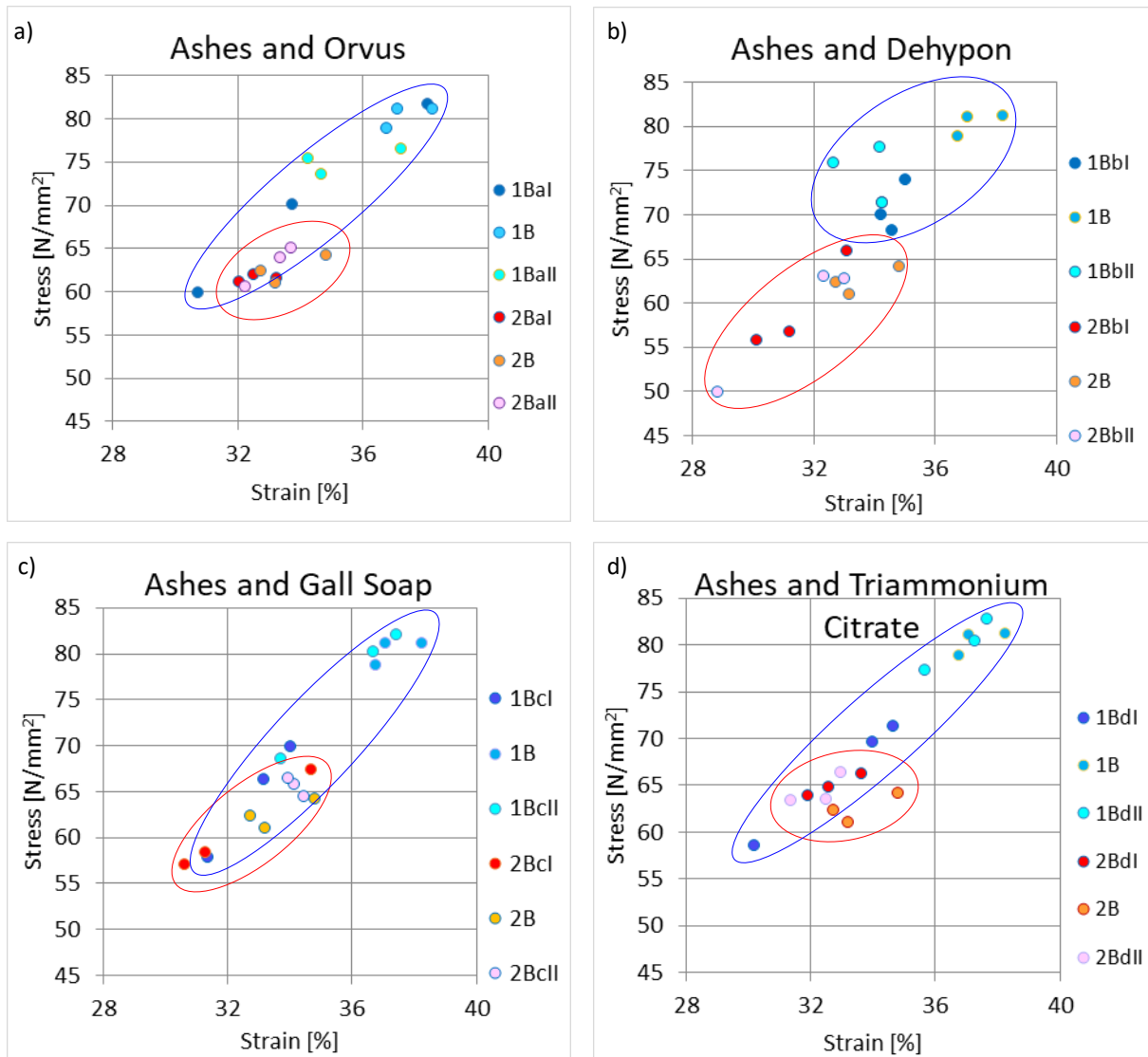


Fig. 50. Scatter plots showing results from tensile testing on aged polyester stained with ashes and cleaned with a) Orvus WA Paste, b) Dehypon LS54, c) Gall soap and d) Triammonium citrate. Aged once (1), aged twice (2), cotton swabs (I) and suction table (II).



Fig. 51. Average differences between samples aged once and treated with cotton swabs (blue) and suction table (red), compared to stained samples aged once (yellow) and aged twice (green).



situated closer to the samples aged twice. The average differences between the samples aged once and treated with cotton swabs and aged once and treated on a suction table, compared to stained samples aged once and twice can be seen in a line graph in figure 51.

The samples treated with cotton swabs had a dispersion more similar to the samples aged twice, whereas the samples treated on the suction table coheres with the samples aged once. One of the samples aged once and cleaned with Gall soap on a suction table is situated close to the cluster of samples aged twice (Figure 50 c).

The samples aged twice and cleaned with Dehypon LS54 and Gall soap are more spread out in the plots than the samples aged twice and cleaned with Orvus WA Paste and triammonium citrate (Figure 50 b, c). One of the samples cleaned with Dehypon LS54 has the lowest stress and strain of all samples tested for tensile strength (Figure 50 b).

#### 6.4.3 Sebum

Of the 72 samples treated with sebum, one tore at the middle of the stain, five tore at the bottom of the sample and 66 at the top of the sample (Figure 52).

The scatter plot showed that with sebum, more samples aged once, both treated with cotton swabs and on the suction table (Figure 53), had breakpoints closer to the reference samples aged twice than the one aged once (Figure 59). 4 out of 6 samples aged once and cleaned with Orvus WA Paste, had a shorter strain than the samples aged twice (Figure 53 a).



Fig. 52. The one sample that tore through the stain and one of the 66 samples that tore at the top of the sample.

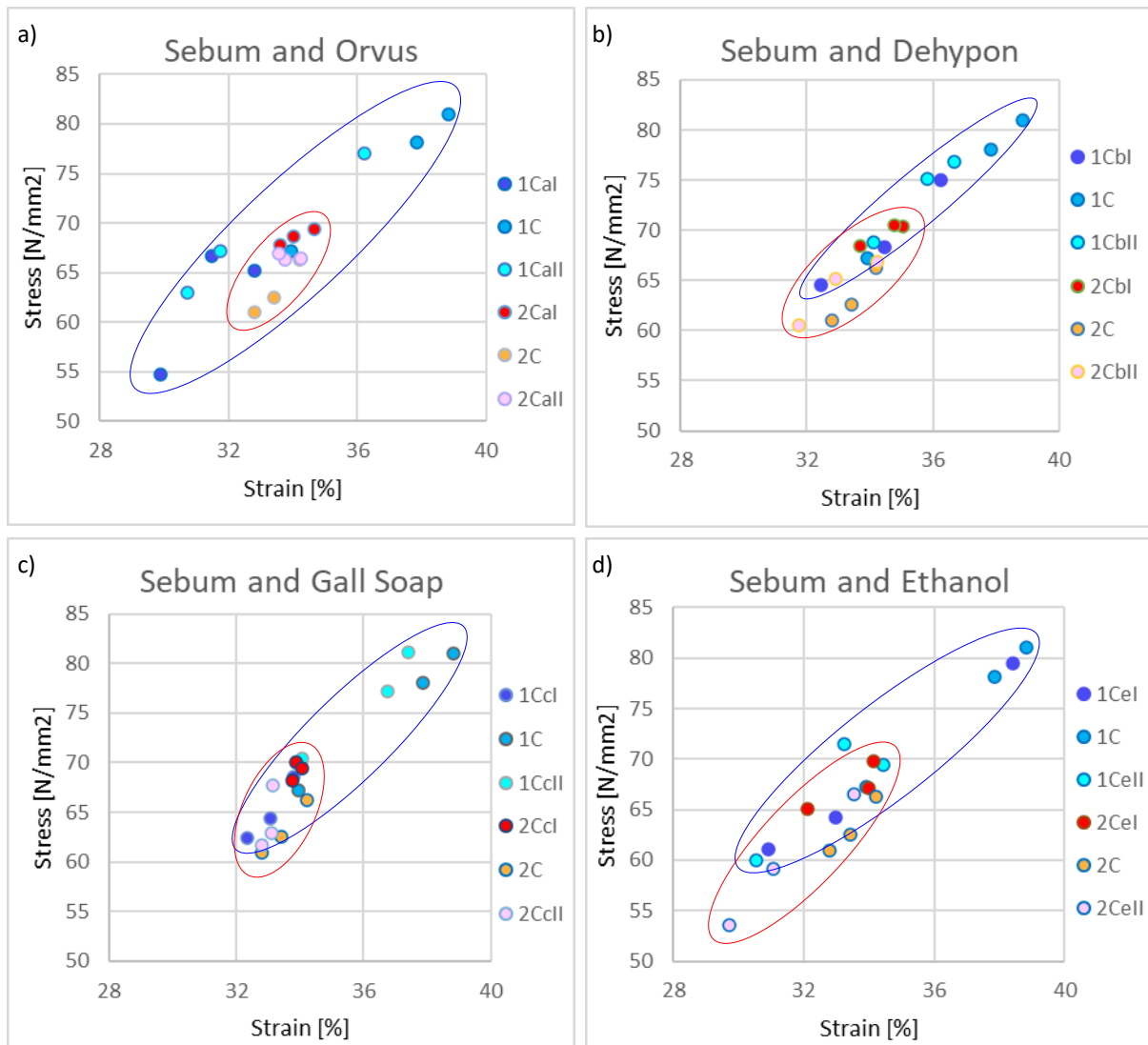


Fig. 53. Scatter plots showing results from tensile testing on aged polyester stained with sebum and cleaned with a) Orvus WA Paste, b) Dehypon LS54, c) Gall soap and d) Triammonium citrate. Aged once (1), aged twice (2), cotton swabs (I) and suction table (II).

The samples aged twice and cleaned with cotton swabs were all found within 65 - 71 N/mm<sup>2</sup> and 32 - 35 % strain, while the other samples were more varied in breakpoint position.

#### 6.4.4 Aged Samples

In the line graph in figure 54, differences can be seen between the un-aged reference samples and the reference samples aged once and aged twice. The un-aged samples had a variety of dispersion between 61-71 mm. They had a steeper curve than the aged samples. 2 of 3 samples aged once had a longer dispersion than the other samples.

The samples aged once, stained with coffee and treated with Orvus WA Paste, Dehypon

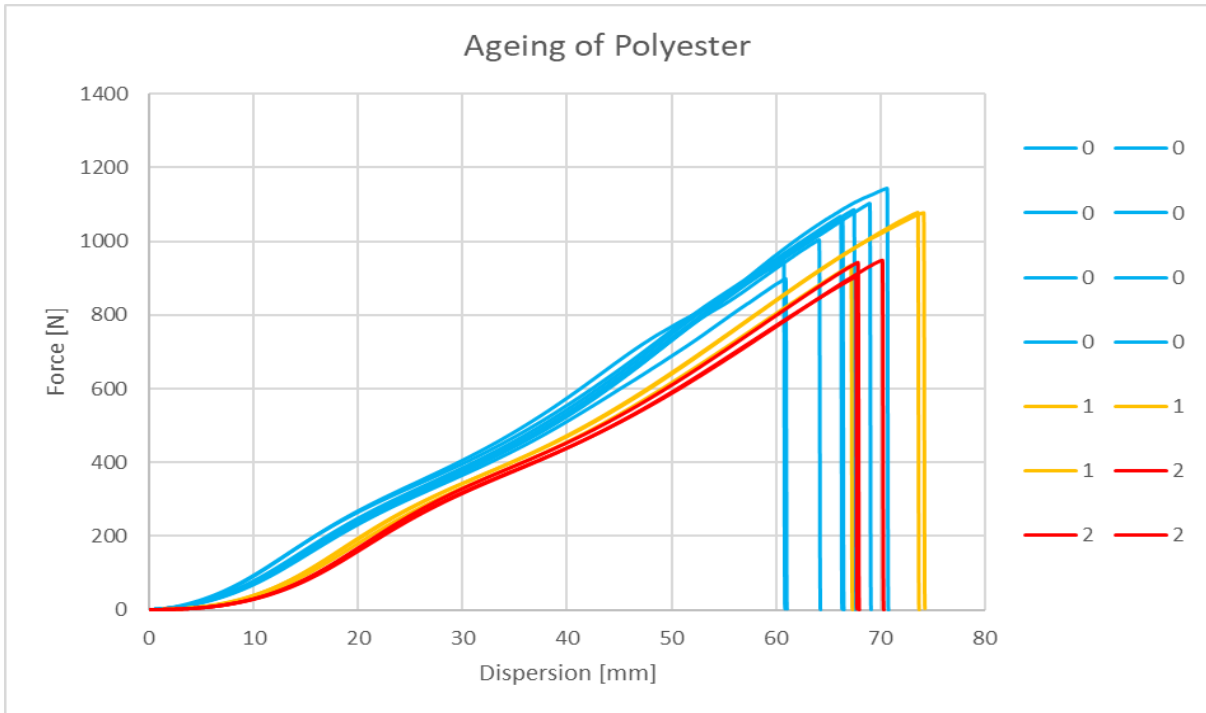


Fig. 54. Line graph showing the tensile strength measurements of un-aged polyester samples (0), samples aged once (1) and aged twice (2).

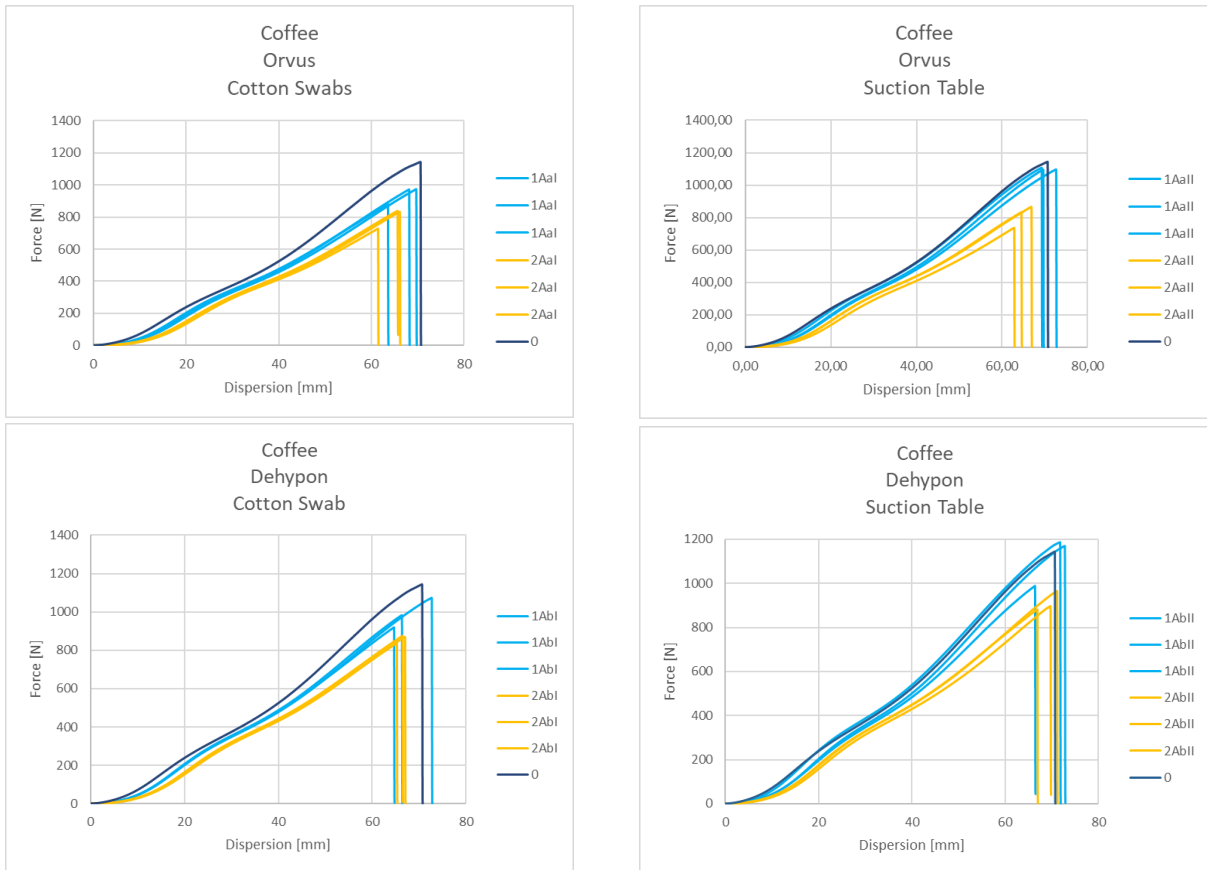


Fig. 55. Line graphs showing that samples aged once, stained with coffee, and treated on a suction table have a similar gradient on the curve as un-treated samples. Dark blue (0) is unaged polyester, light blue (1Aa/bII) is aged once and yellow (2Aa/bII) is aged twice.

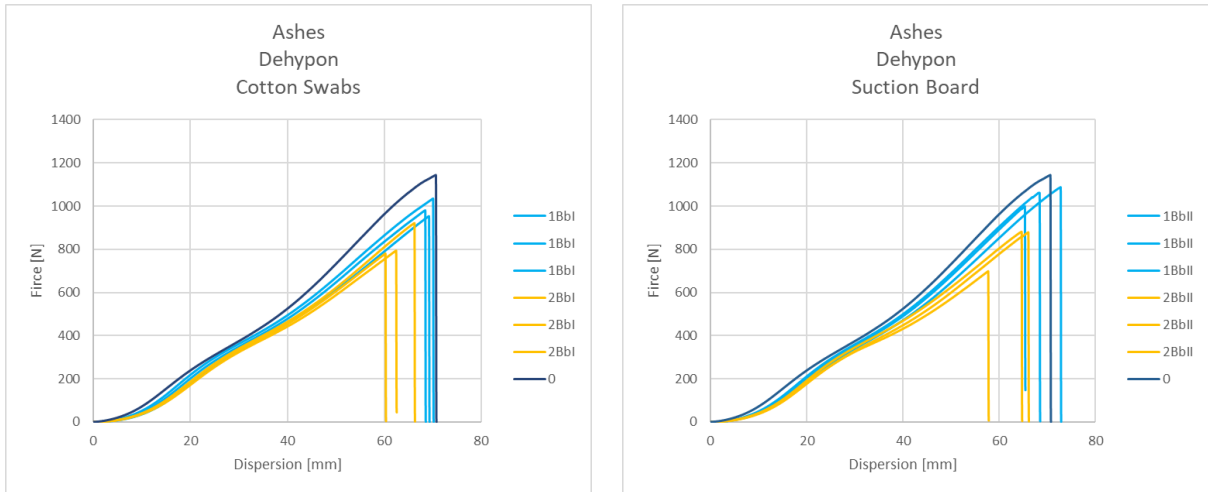


Fig. 56. Samples stained with ash and cleaned with Dehypon. Dark blue (0) is unaged polyester, light blue (1Bbl/II) is aged once and yellow (2Bbl/II) is aged twice.

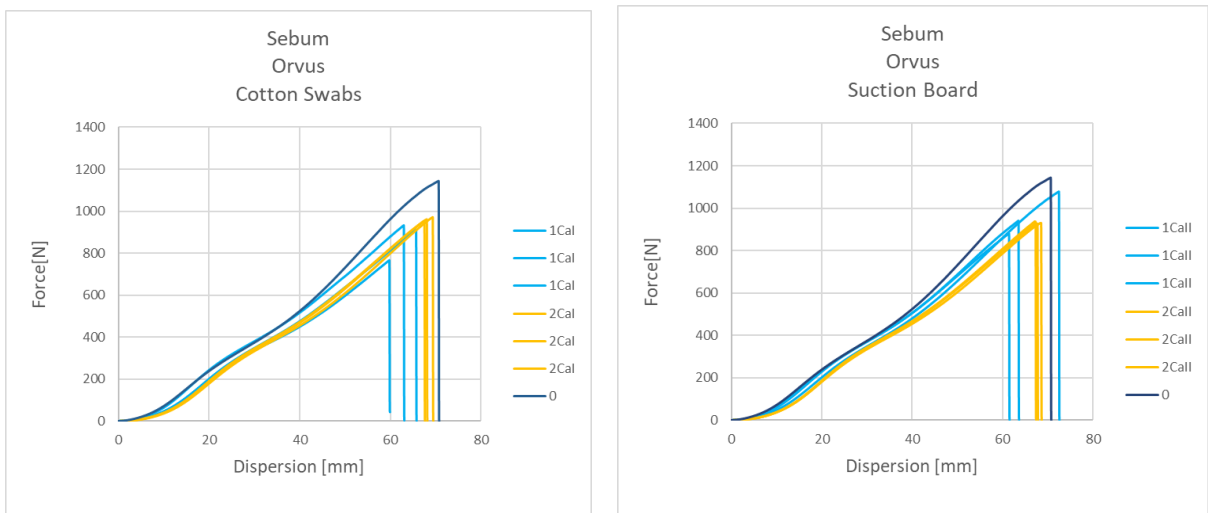


Fig. 57. Samples stained with sebum and cleaned with Orvus WA Paste. Dark blue (0) is unaged polyester, light blue (1Cal/II) is aged once and yellow (2Cal/II) is aged twice.

LS54 and Gall soap on a suction table had a very similar gradient of the curve as the unaged reference samples (Figure 55). Samples aged once, stained with ashes and treated on the suction table had a less similar gradient of the curve, and samples stained with sebum even less so (Figures 56 and 57). Samples treated with cotton swabs had a lower gradient and a more straight line than the samples treated on a suction table (Figures 55, 56 and 57).

Figure 58 shows a line graphs with samples that are aged once and twice and samples that are stained but not treated with a cleaning agent. It compares un-stained samples, samples that are stained but not aged and samples that are aged. All but one of the samples with un-aged stains takes a higher force and has a longer dispersion than the samples with aged stains.

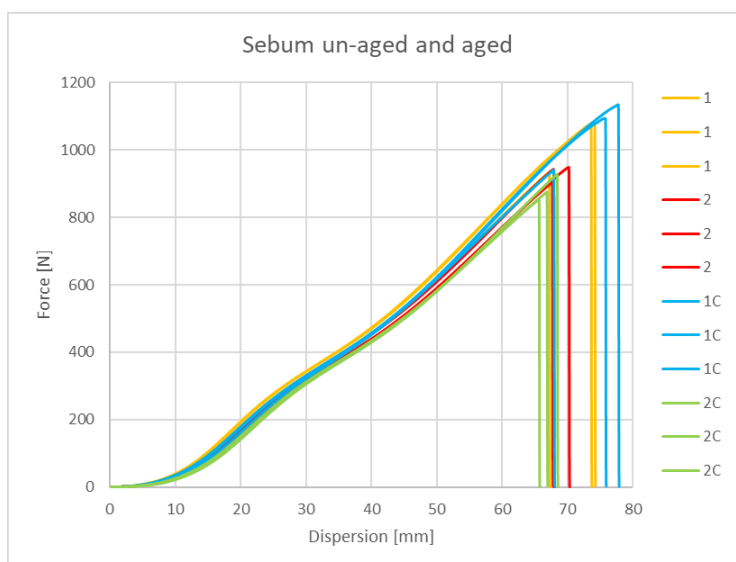
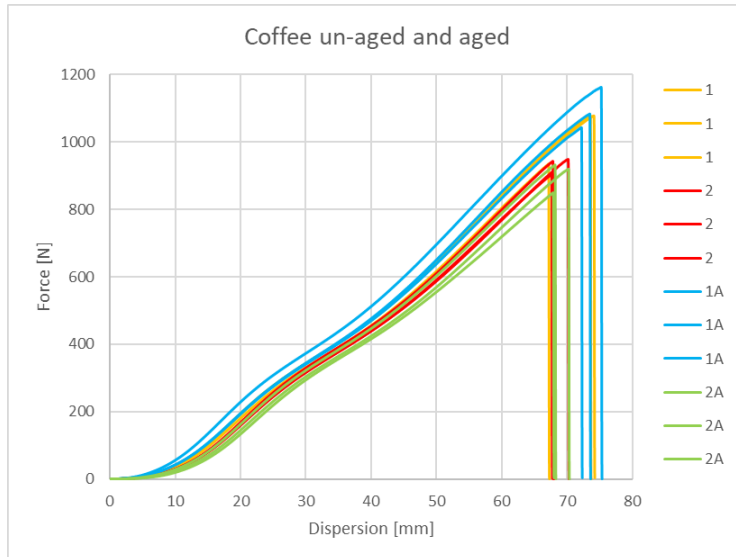


Fig. 58. Line graphs comparing unaged and aged stains. Yellow (1) aged once, red (2) aged twice, blue (1A/B/C) aged once and stained, green (2A/B/C) aged stain.

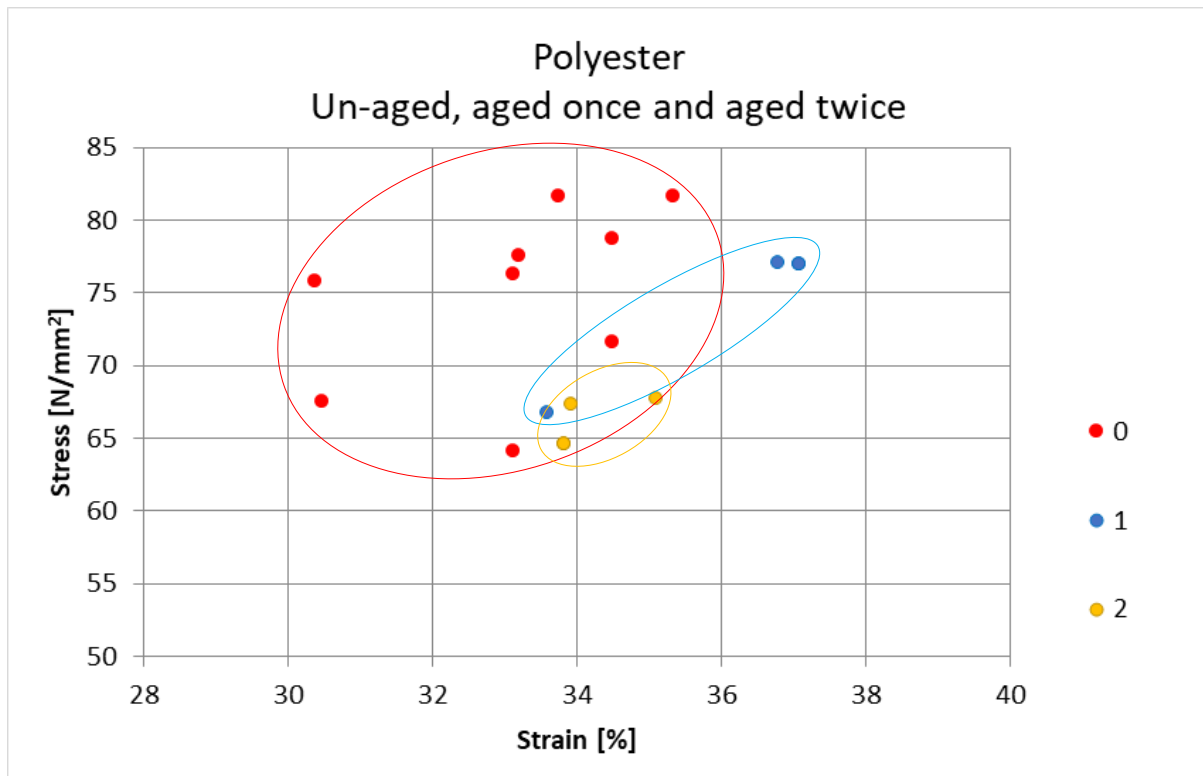


Fig. 59. Scatter plot showing the distribution of breakpoints in polyester that is unaged (0), aged once (1) and aged twice (2).

The scatter plot in figure 59 shows the distribution of breakpoints in polyester that is unaged, aged once and aged twice. The plot shows the 6 extra replicas mentioned in 5.12. The spread of the 9 un-aged replicas emphasises the importance of more than three samples. The scatter plot shows that only three of them would have given a totally different result.

## 6.5 Summary of Results

The coffee and sebum stains darkened during ageing while the ash stains faded. The sebum stains changed appearance during ageing. The un-aged coffee stains were easier to reduce than the aged ones. The aged ash stains were easier to reduce than the un-aged ones. The sebum stains treated with ethanol changed appearance during cleaning.

Gall soap has been very effective at removing the stains in this study. However, all cleaning agents have been effective on different types of stains (Figure 60).

While tensile testing, half of the aged ash samples broke across the stain. The rest of the samples broke in other areas of the sample, predominantly at the top end.

The tensile testing showed a trend, that samples aged once had a longer strain and higher

stress at breakpoint than samples aged twice. The main exceptions were some of the samples aged once and treated with cotton swabs, as some of them were located in the proximity of the samples aged twice. The stained samples and the cleaned samples had very similar breakpoints as the samples that were only aged.

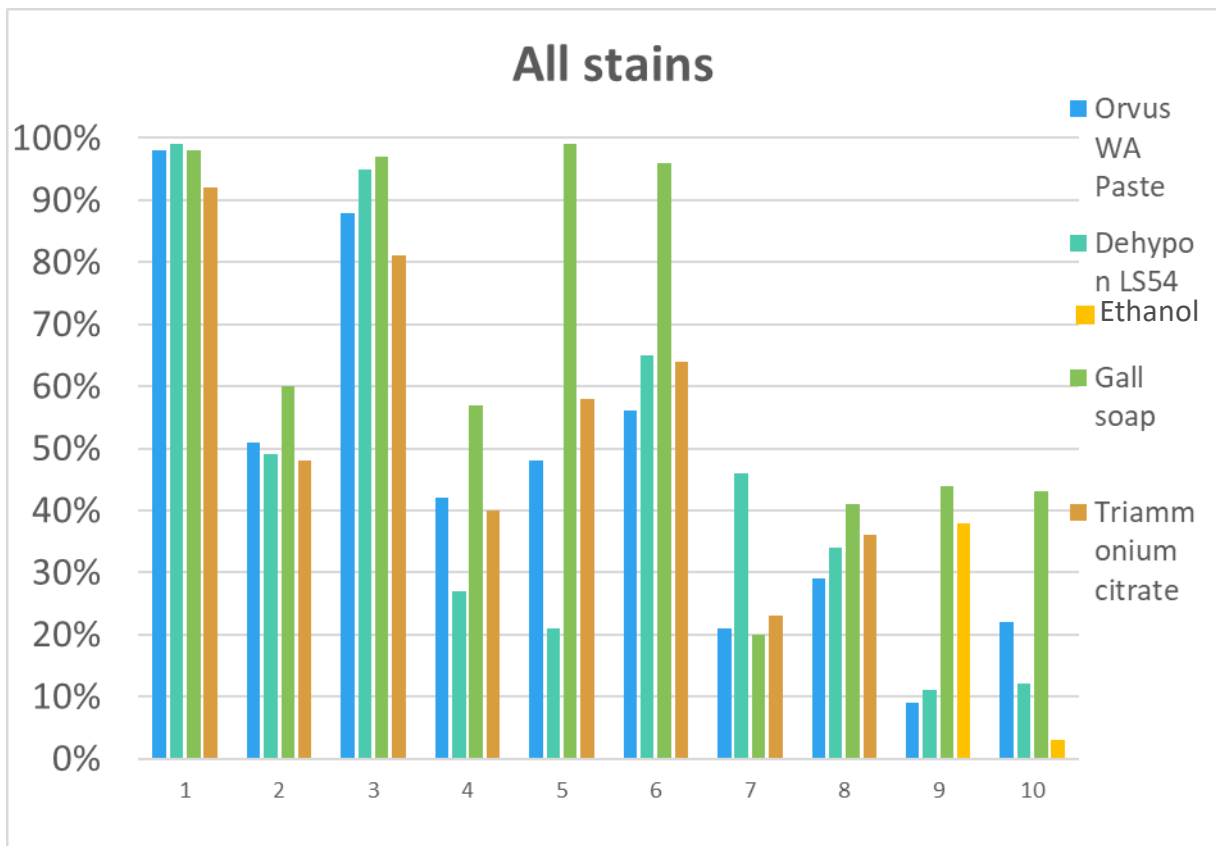


Fig. 60. Reduction of stains, shown in %. 1) un-aged coffee stains treated with cotton swabs, 2) un-aged coffee stains treated on suction table, 3) aged coffee stains treated with cotton swabs, 4) aged coffee stains treated on suction table, 5) un-aged ash stains treated with cotton swabs, 6) un-aged ash stains treated on suction table, 7) aged ash stains treated with cotton swabs, 8) aged ash stains treated on suction table, 9) aged sebum stains treated with cotton swabs, 10) aged sebum stains treated on suction table.

## 7. Discussion

### 7.1 What causes degradation of polyester fibres and in what manner do they degrade? Do different types of stains degrade polyester further?

The high crystallinity of polyester fibres helps polyester to be quite resilient to degradation. Timár-Balázs & Eastop described that the reason for the resilience is that the degradation mainly occurs in the amorphous regions. The literature review disclosed that the factors most susceptible to cause degradation of polyester fibres are UV-light, ozone, moisture and temperature and that a combination of degradation factors increase degradation of polyester fibres (Asadi et al. 2021, p. 7). As the samples in this study only were aged in high temperature and moisture, there was a risk that they would not degrade during ageing. Infrared spectroscopy did not show any signs of degradation, but tensile testing showed differences in the breakpoint and the gradient of the curve. The temperature and moisture did degrade the polyester fibres, but it is likely that ageing in a climate chamber with added UV-light, would have degraded the fibres further and given a different result.

The degradation of polyester fibres can be visible or invisible. When polyester fibres degrade visibly, the degradation can cause discolouration and visible cracks. Invisible degradation means that the degradation can not be seen by the human eye, but can be detected through, for example, tensile testing (Asadi et al. 2021, p. 7). The gradient of the tensile test curve expresses the stiffness of the fibre, the steeper curve, the more stiff the fibre. In this study, the gradient of the tensile strength curve was less steep in aged samples, and showed an inner degradation of the fibres. The ageing caused the fibres to lose some of its stiffness, which indicates that the crystallinity of the fibres decreased during ageing (Timár-Balázs & Eastop 1998). Samples aged once, stained with coffee and treated on the suction table with Orvus WA Paste, Dehypon LS54 and Gall soap had a very similar curve to the untreated reference samples, which indicates that they had regained the stiffness of the unaged samples (Figure 55). The stiffness of the polyester fibre is due to the high percentage of crystallinity of the fibres. Asadi et al. (2021) says that above  $T_g$  the hydrolytically cleaved ester bonds causes an increase of crystallinity. It appears as if an increase of the crystallinity has somehow occurred during the treatment of the textile.

The colour differences between the un-aged polyester, the polyester aged once and aged twice were not perceptible by a quick glance. However, when measuring the colour using the spectrophotometer the data showed differences, both between the un-aged samples and the samples aged once, and between the samples aged once and the ones aged twice (Table 3). The tensile strength results and the colour measurements showed that the aged samples had both invisible degradation within the fibres, and visible degradation with colour changes.

According to Asadi et al. (2021), both acidic and alkaline conditions can cause hydrolysis, and the hydrolysis is increased with lower and higher pH. Gohl and Vilensky (1980) de-



scribe that polyester fibres are resistant to acid hydrolysis, but not to alkaline hydrolysis. The tensile testing in this study showed that samples stained with an alkaline dirt, ashes, and left to age, were more prone to breakage in the stained area than other samples (Chapter 6.4.2). This indicates that the alkaline conditions over time degrade polyester fibres further than the other tested conditions. It may be seen as a confirmation of what Gohl and Vilensky described. On the other hand, it must be noted that the ashes had a pH of 11 which is at the top end of the weak alkalis, rather close to a strong alkali, whereas the coffee had a pH of 5-5,5 which is a quite weak acid, close to neutral. Hence the statement by Asadi et al., that the amount of hydrolysis in polyester fibres are affected by how strong the acid/alkali is, might be consistent with the results found in this study. To confirm this, tests with stronger acidity and weaker alkalinity should be performed.

## **7.2 What will be the effects if a stain is left to age?**

When comparing samples aged but not stained and aged and stained (Figure 58), the tensile strength data shows that samples aged once, as well as samples aged twice, have similar curves whether they are stained or not. There are two exceptions from the trend. One of the un-stained samples, aged once, and one of the samples aged once, stained with sebum. Both have similar curves as the samples aged twice. These results indicate that neither the aged, nor the un-aged stains impact the tensile strength of the samples.

However, the samples aged twice generally had lower tensile strength than the samples aged once. No differences between samples aged with different staining agents were seen in the measurements. When examining the samples after tensile testing though, it was seen that half of the aged, ash stained samples broke across the stained area, and hardly any of the other samples did. This indicates that the alkali ashes affected the degradation of polyester fabric during ageing. The rest of the samples broke mainly at the top of the sample, while some broke at the bottom. No results can be concluded from whether the break was at the top or the bottom.

All three types of stains showed different behaviour during ageing. The coffee stains darkened with an average darkening of 5  $\Delta E$ . The sebum stains darkened even more. They had an average darkening of 9,8  $\Delta E$ . The Ash stains, on the other hand, faded during ageing. The colour faded with an average of 3,8  $\Delta E$ . These results show that it is not certain that ageing causes darkening of stains, the ageing can also fade the stains, just as dyed textiles often fade over time.

The un-aged sebum stains were very similar in colour to the un-stained reference samples, the differences were more visible as a greasiness with a higher shine, rather than as colour changes. When aged, the shine was reduced and, as mentioned, the colour of the samples were quite different. To understand the stains and effects of ageing on fatty stains, it might

be important to take into account glossiness and translucency as well as measuring colour. A spectrophotometer can often be used to measure all three.

The shapes of the coffee stains and ash stains were the same before and after ageing, but the shape of the sebum stains changed dramatically. This was due to the melting temperatures of the sebum being lower than the temperature of the climate chamber. With the melting range of 43-66 °C, it is not very likely that sebum stains would spread in the fabric during real life ageing, if not situated in a very warm country. Temperatures in Sweden never reach those heights.

The ageing of the stains had consequences for the stain reduction. For coffee stains and sebum stains, the ageing caused the effect of the cleaning to decrease. It was harder to remove the stains after ageing. For the ash stains, a variation between decreasing and increasing the effect of cleaning could be seen. For most of the ash samples it was easier to reduce the stains after ageing. Most of them were closer in colour to the reference samples after ageing and cleaning than the cleaned un-aged stains were. Hence, all types of stains do not benefit visibly from being cleaned when they are fresh, even though many do. When considering treatment of ecclesiastic textiles that are used during service, and should enhance the experience of it, it is still often important to remove the stain as soon as possible, for the textile to give an appropriate appearance.

## **7.3 What cleaning agents are effective and suitable for cleaning polyester textiles?**

### **7.3.1 Coffee**

Kissas research (1995, p. 975) showed that coffee stains had a low affinity to polyester fibres. The same results can be seen in this study, as all four cleaning agents were effective on the un-aged coffee stains. Almost all colour was removed when cleaned with cotton swabs, and Gall soap removed the same amount on the suction table. Orvus WA Paste, Dehypon LS54 and triammonium citrate removed a little less on the suction table and the stains were still perceptible at close inspection. As mentioned earlier, the aged coffee stains were harder to reduce, and none of the cleaning agents reduced the aged stains as much as the un-aged stains. Hylander (1956) suggests neutralisation for acidic coffee stains and Timár-Balázs & Eastop (1998) describes that acidic, coloured stains may be mobilised and removed with alkalis. This is probably the reason that the anionic and slightly alkaline Orvus WA Paste and Gall soap were effective at removing the acidic coffee stains. However, the non-ionic, neutral Dehypon LS54 and neutral triammonium citrate also had a good cleaning effect on most coffee stains. As anionic surfactants, non-ionic surfactants, en-

zymes and chelating agents reduce dirt in different ways and have different strengths and weaknesses, it is often good to try a variety of cleaning agents if a stain does not reduce. As seen by the results in this study, different types of cleaning agents can be tried and prove effective on the same type of stain. The interviewees mentioned that trial and error often were needed in stain reduction.

The most effective cleaning agent for coffee stains, both un-aged and aged, in this study, was Gall soap. This might be due to the alkalinity of Gall soap, which neutralises the acid stains, as well as the overall efficiency of Gall soap as a stain reducer. Dehypon LS54 was the least effective on aged stains, especially on the suction table, but was very effective on un-aged coffee stains.

When the un-aged coffee stains were treated with Orvus WA Paste, Dehypon LS54 and Gall soap on the suction table, the tensile test curve changed and became very similar to the curve of the reference samples of un-aged, un-stained polyester fabric. As mentioned in chapter 6.1, this might indicate that the three cleaning agents, in combination with the acidic coffee stains, can regain the original stiffness of the polyester.

### 7.3.2 Ashes

The difference in how effective the four cleaning agents were on un-aged ash stains, was huge. One reason for this might be that the ash stains were inconsistent in colour and hue and seemed to adhere differently to the polyester fabric, even though the stains were created in the same way. When looking at colour differences before and after ageing, triammonium citrate is consistent in the amount of colour that has been reduced in both the un-aged stains, and in the aged stains (Table 5). Gall soap is consistent in the un-aged stains and Orvus WA Paste in the aged stains. The amount of ions in the ash stains might be a reason for Orvus WA Paste, Gall soap and triammonium citrate to be more consistently effective on the ash stains than Dehypon LS54, which neither ionise nor chelate the metal ions.

Gall soap was very effective in reducing the ash stains with cotton swabs, with close to 100% reduction. Orvus WA Paste, triammonium citrate and on the aged stain, Dehypon LS54, showed rather high amounts of colour reduction with cotton swabs as well. The stains treated on the suction table did not reduce as much. It appears as if ash stains need the mechanical action of the cotton swab to be reduced more.

### 7.3.3 Sebum

The un-aged sebum stains were so similar in colour to the reference samples, that

the colour difference while cleaning them are negligible in trying to understand which cleaning agents are effective for cleaning polyester fabric. However, the sebum stains changed colour considerably during ageing, from barely noticeable to around 11-12  $\Delta E$ . The aged stains can be used to understand what cleaning agents are suitable to use on sebum stains on polyester fabric.

Ethanol treatment changed the appearance of the stains, they turned whiter in some parts, and more translucent in others. The white parts gave the impression of being collected on top of the fabric, rather than inside of it. It might be possible to remove those parts with mechanical action, but this ought to be tested in further research. Ethanol is not the best solvent for solving fatty stains (Hylander 1956; Timár-Balázszy & Eastop 1998). Examples of solvents that are better at solving fatty stains are acetone and isopropanol. It would be interesting to examine other solvents, in combination with sebum stains and polyester fabric.

Timár-Balázszy & Eastop (1998, pp. 59-62) described that treatment with ethanol might cause desiccation of the fibre. The tensile strength testing showed that all but one sample treated with ethanol had a strain below 72 N and a stress less than 35%. This might be due to desiccation of the fibre, but it might also be due to the sebum staining, as the sebum stains treated with Orvus WA Paste show similar results. To get clarity in this question, tensile strength needs to be tested on a larger amount of replicas of each sample.

Polyester has an affinity for fatty, greasy soils according to Gohl & Vilensky (1980 p. 115). It can be hard to clean and reduce those stains on polyester. The results show that the cleaning power of Orvus WA Paste and Dehypon LS54 was low when cleaning the sebum stains. Both methods of cleaning the aged stains with Gall soap showed better cleaning power. This is probably due to the bile acid found in ox gall, which forms micelles and emulsifies lipids and absorbs fats. Gall soap is known for being good at cleaning fatty stains. Although the cleaning power was not as good as on coffee stains and ash stains, the cleaning power was good enough to make a visible difference.

The sebum stains treated with ethanol had a different appearance than the ones treated with surfactants, due to the different techniques of loosening the dirt from the fabric. Ethanol is a solvent and dissolves the fatty stains, while surfactants work with micelles and rolling up of the fatty dirt, and enzymes breaks down the dirt into smaller, dissolvable compounds (Timár-Balázszy & Eastop 1998, pp. 59, 233; Wolbers 2003, p. 57). Fatty stains might need to be partly dissolved by a solvent, but considering the appearance of the treated sebum stains (figures 32 and 37), a combination of solvent cleaning and wet cleaning might be used to get more aesthetically pleasing results.

When considering all types of stains and cleaning methods, the cleaning agent that was

most effective was Gall soap. Gall soap was most effective on 9 types of stains. Dehypon LS54 was most effective on two types of stains and Orvus WA Paste on one type. Triammonium citrate was the second most effective on 5 types of stains, Orvus WA Paste and Dehypon LS54 on 4 types of stains and Gall soap and ethanol on one type of stain. As mentioned, ethanol might not be the best solvent at reducing fatty stains. The other four cleaning agents were all effective at different stains and through different cleaning methods. Hence, it is important to have a variety of cleaning agents for stain reduction, and to try different ways to reduce the stain. As one of the interviewees said: Through trial and error, all of a sudden you find the cleaning agent that works on that specific stain. It is also possible to mix different cleaning agents to change their abilities and thus reduce the stain. Timár-Balázs & Eastop (1998, p. 225) explains that the combination of surfactants and sequestering agents can be very effective.

None of the cleaning agents appeared to be unsuitable to use on polyester fabric when looking at the results from the tensile testing. All of them give similar results. However, the appearance of the sebum stains after treatment with ethanol was not satisfactory, and ethanol should probably not be the only cleaning agent used on a fatty stain on polyester.

Orvus WA Paste, Dehypon LS54, Gall soap and triammonium citrate are all suitable to use when cleaning historical polyester textiles according to the results of this study. Timár-Balázs, Csányi, & Máréfys conclusions (1993 p. 335), that the most effective stain removal agent might not suite the conservators goal to preserve the fabric is not shown true by the results in this study. However, if Gall soap is used for cleaning stains on other types of fibres, it is important to be aware that the pH of Gall soap lies between 10-10,5 according to the producers, a pH that can cause further degradation of some types of fibres. Another issue to be aware of concerning Gall soap is that soaps can form lime soap when used in hard water, så before using Gall soap it is important to learn about the hardness of the water being used. No signs of lime soap was found on the samples cleaned with Gall soap.

#### **7.4 What cleaning methods are suitable for stain removal on polyester textiles?**

The tensile data shows that the un-aged stains treated with cotton swabs were inconsistent in the placements of the breakpoints (Figures 47, 50 and 53). Many of their breakpoints had a lower stress and strain than the other un-aged stains, while others had similar stress and strain. If the phenomenon only were seen with the ash stains, one could have imagined that parts of the ashes still were loosely bound to the textile, and moved with the cotton swab, creating more mechanical action. But as the phenomenon occurred with all three types of stains, and the other two were not bound to the textiles surface in

the same way, this theory is probably incorrect. Especially so, as the break point of the ash stains did not differ from the breakpoints of the other stains.

It was considered whether the amount of colour degradation affected the break points of the samples and if that could be the reason for the inconsistent data of the un-aged stains treated with cotton swabs. There were no trends found when comparing the breakpoint of the samples to the colour differences of the same samples, which showed that the amount of colour reduction was not the reason for the inconsistent breakpoint results.

The aged stains treated with cotton swabs were more coherent with the rest of the aged stains in the tensile strength plot than the un-aged stains were. They were a bit spread out in the plot with some treatments, but not as widely as in the samples with un-aged stains. It seems as if the second ageing caused the samples to become more stable, and that the mechanical action not affected the fibre more than the dripping through the textile did, as it had already degraded that far. It appears as if some of the textiles aged once were more sensitive to mechanical action than the samples aged twice.

The un-aged coffee stains treated on the suction table had a slightly higher colour difference between not cleaned and cleaned stains, than the stains treated with cotton swabs. With the aged coffee stains, it was the other way around, the stains treated with cotton swabs had a higher average  $\Delta E$  than the ones treated on the suction table. With the un-aged ash stains, Orvus WA Paste, Gall soap and triammonium citrate had higher average  $\Delta E$  when treated with cotton swabs than on the suction table. The ash stains were overall more effectively reduced with cotton swabs than on the suction table.

For the un-aged sebum stains, cotton swabs and suction table gave similar results concerning average colour difference between not cleaned and cleaned stains. The same goes with the aged stains and Orvus WA Paste, Dehypon LS54 and Gall soap. Ethanol reduced the colour of the aged stains less on the suction table than with cotton swabs. The sebum stains treated with ethanol on the suction table also became more visible than the ones treated with cotton swabs. The fatty stains probably need the mechanical action created with cotton swabs when they are being dissolved by a solvent, otherwise the solvent will spread in the fabric.

Tinkham (2001) investigated the colour differences when cleaning pre-soiled polyester fabrics with a number of surfactants, of which one was Orvus Wa Paste. She compared stains not agitated during cleaning with stains agitated. In this study we can compare the un-agitated cleaning to the cleaning on a suction table, and in some ways, the agitated cleaning to the cleaning with cotton swabs. Her results said that samples cleaned with Orvus WA Paste, and not agitated had a colour difference of 1,1  $\Delta E$ , and the samples cleaned with agitation had a colour difference of 7,3  $\Delta E$ . Tinkhams samples were not aged, so the results could be compared to the un-aged coffee and ash stains of this study (not sebum, because of the small colour difference when stained). Table 5 shows that all stains, the aged once as well, treated with Orvus WA Paste on a suction table (not agitated) were reduced less than

the stains treated with Orvus WA Paste and cleaned with cotton swabs (agitated). Even though the amount of  $\Delta E$  is different between this and Tinkhams study, the result shows that samples treated with Orvus WA Paste and agitated during cleaning have a higher reduction of colour than samples cleaned without agitation.

Both cleaning methods for stain reduction tried in this study showed good results. Sometimes cotton swabs were more effective and sometimes a suction table was a more effective tool. While not tried in this study, a combination of these methods would very likely be functional, effective and suitable for treating stains on polyester fabric.

## 8. Conclusions

The main degradation factors for polyester fibres are UV-light, oxygen, moisture and temperature. As polyester fibres have a high degree of crystallinity, and the deterioration mainly appears in the amorphous regions, it is a quite stable fibre, not prone to degradation. When degradation occurs, it can be either visible, with colour differences or hazing or invisible, with changes within the material. A pigmented fibre runs less risk of degradation due to UV-light as the light is prevented from reaching inside the fibres by the pigments.

The colour changes caused by ageing were barely visible to the naked eye, but were detected with colour measurements using spectrophotometry, with 1,2  $\Delta E$  in the samples aged once, and 1,7  $\Delta E$  in the samples aged twice. The tensile data showed that ageing did change the tensile strength of the fibres, which were weaker after ageing.

The aged alkaline ash stains created a higher tendency for breakage at the stain during tensile testing than the rest of the stains. It appears likely that alkaline stains left to age on polyester fabric increase the degradation of the fibre.

The ash stains faded with ageing, and were in most cases somewhat easier to clean after ageing. The coffee stains on the other hand, darkened with ageing and were harder to remove after ageing. The stains made from sebum changed appearance during ageing, they spread in the textile and turned darker. Gall soap was the most effective cleaning agent for the aged sebum stains, and removed around 44% of the colour of the stain.

All cleaning agents used in this study, Orvus WA Paste, Dehypon LS54, Gall soap, triammonium citrate and ethanol, gave good colour reduction results with some stains. However, ethanol created a messier stain, even though the colour of it was reduced. Gall soap was the cleaning agent that over all was most effective at reducing the colour of the stains. The results show that the same type of stain can be reduced with different types of cleaning agents. The tensile testing did not pinpoint any of the cleaning agents as unsuitable, as the breaking points of the cleaned samples were similar to the breaking points of the aged reference samples.

Both mechanical action with cotton swabs and cleaning agent and letting cleaning agent drip through the textile on a suction table were suitable cleaning methods for removing stains from polyester fabric. As both of them gave the best result with different stains and cleaning agents, it is suggested that they can be used in combination to perhaps give an even better result.



## 8.1 Further Research

The aim of this pilot study was to create means to map out a model that could be used in further research about stain removal on ecclesiastical textiles found in the Swedish church. The structure of this study could be used as a base for further research. The differences compared to this study should be to make separate samples for cleaning and for tensile testing, and to prepare at least 5 replicas of each sample. The steps to build the research around would be:

- Understand the fibre of the textile and how it degrades through
  - ◇ Literature review
  - ◇ Ageing of samples
  - ◇ FTIR measurements
  - ◇ Colour measurements
  - ◇ Tensile testing
- Investigate cleaning of stained samples through
  - ◇ Preparation of stained samples for cleaning according to ISO standard 105-E07:2010 Textiles - Tests for colour fastness
  - ◇ Ageing of samples
  - ◇ Cleaning of samples
  - ◇ Colour measurements
- Investigate degradation of stained and cleaned fibres through
  - ◇ Preparation of stained samples for tensile testing according to ISO standard 13934-1, Textiles - Tensile properties of fabrics
  - ◇ Ageing of samples
  - ◇ Cleaning of samples
  - ◇ Tensile testing

If stain removal on polyester textiles should be investigated further, it would be important to prepare more replicas of the samples for tensile testing. It would be interesting to prepare stained samples as well as immersed samples for tensile testing to compare the tensile data as well as the physical results of tear placement.

There are other stains, cleaning agents and cleaning methods that would be important to investigate in combination with polyester textiles to get a wider understanding of the behaviour of polyester fibres. For this, it would be interesting to do the same research as in this thesis with a plain woven polyester textile and learn if it gives the same results.

To get a further understanding of the degradation of polyester, an investigation with ageing using UV-light would be important to perform as textiles often are exposed to UV-light, and it is one of the most important degradation factors of polyester fibres. It would also be interesting to investigate ageing for a longer period of time, to gain further knowledge about the degradation of polyester fibres.

It would also be interesting to do a study that compare stain removal of stains pre-treated with glycerol with stains without pre-treatment.

## 9. Summary

The thesis was an experimental pilot study that investigated efficiency and effects of stains and cleaning agents on polyester textiles. The objective of the thesis was to, through literature studies, interviews, experiments and analyses, gain an understanding of the degradation process of polyester and to find suitable cleaning methods and cleaning agents to use for stain removal on polyester fabric.

The research questions used were:

- What causes degradation of polyester fibres and in what manner do they degrade? Do different types of stains degrade polyester further?
- What will be the effects if a stain is left to age?
- What cleaning agents are effective and suitable for cleaning polyester textiles?
- What cleaning methods are suitable for stain removal on polyester textiles?

The literature reviewed looked at textile fibres with a focus on polyester fibres, their history, composition, properties and degradation. Polyester is not very prone to degradation due to its high crystallinity. Degradation occurs foremost in the amorphous parts of the fibre. The main degradation factors for polyester fibres are UV-light, oxygen, moisture and temperature.

Cleaning processes and dirt were also reviewed through literature. The detergents used in textile conservation are mainly made from anionic and non-ionic surfactants. Sequestering agents, enzymes and solvents are also used to clean historical textiles.

12 textile conservators were interviewed and the answers about stain reduction, type of water used while cleaning, and methods to dry textiles were used, along with the literature review, to decide what stains, cleaning agents and cleaning methods to use during the experimental part of the thesis.

The experimental part contained aged and stained samples of polyester fabric. The samples were aged in a climate chamber at 95°C and 65% relative humidity. Half of the samples were aged once and the other half aged twice. After the first ageing, the samples were stained with acidic coffee, alkaline ashes or fatty synthetic sebum. Half of them were stained and then cleaned and the other half were stained, aged a second time, and then cleaned.

After careful consideration, five cleaning agents were tested. Anionic Orvus WA Paste, non-ionic Dehypon LS54, gall soap, which is a well known spot remover, but not yet investigated through a conservation point of view, triammonium citrate, which is a chelating agent, and for the sebum stains, triammonium citrate was replaced with ethanol, as fatty stains often require a solvent to be removed.

Two cleaning methods were used. Number one was cotton swabs dipped in cleaning agent and rolled over the stain. The cleaning was done on blotting paper, to help absorb the dirt. Number two was done by dripping the cleaning agent through the stain on a suction table.

The analytical tools used to analyse the experiments were a spectrophotometer to look for colour changes and evaluate the efficiency of the cleaning agents, infrared spectroscopy was used to look for molecular differences in the aged polyester fabric, and tensile testing was used to search for degradation through ageing, staining and cleaning.

Infrared spectroscopy did not show any molecular differences. Unaged polyester, polyester aged once and aged twice were tested, and the spectra were very similar. However, both colour measurements and tensile testing showed differences in polyester not aged, aged once and aged twice.

All cleaning agents used in this study, Orvus WA Paste, Dehypon LS54, Gall soap, triammonium citrate and ethanol, gave good colour reduction results with some stains. However, ethanol created a messier stain, even though the colour of it was reduced. Gall soap was the cleaning agent that over all was most effective at reducing the colour of the stains. The results show that the same type of stain can be reduced with different types of cleaning agents. The tensile testing did not pinpoint any of the cleaning agents as unsuitable, as the breaking points of the cleaned samples were similar to the breaking points of the aged reference samples.

Both mechanical action with cotton swabs and cleaning agent and letting cleaning agent drip through the textile on a suction table were suitable cleaning methods for removing stains from polyester fabric. As both of them gave the best result with different stains and cleaning agents, it is suggested that they can be used in combination to perhaps give an even better result.

The aim of the thesis was to create means to map out a model for further research of different textile fibres in combination with different types of stains, different cleaning agents and stain removal methods. The structure of this thesis could be used in a larger project about stain reduction.

## 10. List of References

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# 11. List of Illustrations

## 11.1 List of Figures

Photographs and illustrations by Karin Hindborg if not otherwise stated.

<b>Figure 1</b>	The universal recycling code for PET.	p. 21
<b>Figure 2</b>	The four most common types of surfactants.	p. 28
<b>Figure 3</b>	Hydrophobic tails and hydrophilic heads of surfactants.	p. 28
<b>Figure 4</b>	How micelles encapsule, roll up and solubilise soil.	p. 29
<b>Figure 5</b>	Interpretations of the tensile test curve.	p. 35
<b>Figure 6</b>	Samples prior to ageing.	p. 44
<b>Figure 7</b>	Staining with coffee.	p. 46
<b>Figure 8</b>	Stains from ashes and sebum.	p. 46
<b>Figure 9</b>	CTS climate chamber.	p. 47
<b>Figure 10</b>	Climate chamber settings.	p. 47
<b>Figure 11</b>	Stain reduction with cotton swabs.	p. 48
<b>Figure 12</b>	Stain reduction on suction table.	p. 49
<b>Figure 13</b>	Tensile testing.	p. 52
<b>Figure 14</b>	Tensile testing with slack mounting.	p. 52
<b>Figure 15</b>	Un-aged coffee samples.	p. 53
<b>Figure 16</b>	Un-aged ash samples.	p. 53
<b>Figure 17</b>	Un-aged ash samples.	p. 54
<b>Figure 18</b>	Un-aged sebum samples.	p. 54
<b>Figure 19</b>	Un-aged sebum samples.	p. 54
<b>Figure 20</b>	Un-aged coffee samples.	p. 55
<b>Figure 21</b>	Un-aged coffee samples.	p. 55
<b>Figure 22</b>	Blotting paper with residues from stains.	p. 55
<b>Figure 23</b>	Un-aged ash samples.	p. 56
<b>Figure 24</b>	Un-aged sebum samples.	p. 56

<b>Figure 25</b>	Coffee stains before ageing.	p. 57
<b>Figure 26</b>	Aged coffee stain.	p. 57
<b>Figure 27</b>	Ash stain before ageing.	p. 57
<b>Figure 28</b>	Aged ash stain.	p. 57
<b>Figure 29</b>	Sebum stain before ageing.	p. 58
<b>Figure 30</b>	Aged sebum stain.	p. 58
<b>Figure 31</b>	Aged sebum stain.	p. 58
<b>Figure 32</b>	Aged sebum stain.	p. 58
<b>Figure 33</b>	Aged coffee stain.	p. 59
<b>Figure 34</b>	Aged coffee stain.	p. 59
<b>Figure 35</b>	Aged ash stain.	p. 60
<b>Figure 36</b>	Aged sebum stain.	p. 60
<b>Figure 37</b>	Aged sebum stain.	p. 60
<b>Figure 38</b>	Delta E for coffee stains.	p. 62
<b>Figure 39</b>	Reduction of coffee stains.	p. 62
<b>Figure 40</b>	Delta E for ash stains.	p. 64
<b>Figure 41</b>	Reduction of ash stains.	p. 64
<b>Figure 42</b>	Delta E for sebum stain.	p. 65
<b>Figure 43</b>	Reduction of aged sebum stains.	p. 66
<b>Figure 44</b>	Total colour difference and lightness.	p. 67
<b>Figure 45</b>	FTIR overlay spectrum.	p. 68
<b>Figure 46</b>	Torn samples stained with coffee.	p. 69
<b>Figure 47</b>	Scatter plot on coffee stains.	p. 70
<b>Figure 48</b>	Torn sample stained with ashes.	p. 71
<b>Figure 49</b>	Torn sample stained with ashes.	p. 71
<b>Figure 50</b>	Scatter plot on ash stains.	p. 72
<b>Figure 51</b>	Differences between samples aged once and twice.	p. 72
<b>Figure 52</b>	Torn sample stained with sebum.	p. 73
<b>Figure 53</b>	Scatter plot on sebum stains.	p. 74

<b>Figure 54</b>	Line graph ageing of polyester.	p. 75
<b>Figure 55</b>	Line graphs comparing gradient of curve.	p. 75
<b>Figure 56</b>	Line graphs comparing gradient of curve.	p. 76
<b>Figure 57</b>	Line graphs comparing gradient of curve.	p. 76
<b>Figure 58</b>	Line graphs comparing unaged and aged stains.	p. 77
<b>Figure 59</b>	Scatter plot showing distribution of breakpoints.	p. 78
<b>Figure 60</b>	Reduction of all stains	p. 79

## 11.2 List of Tables

<b>Table 1</b>	Factors, versions and naming of samples	p. 43
<b>Table 2</b>	Standardised set-up of experiment	p. 43
<b>Table 3</b>	Average colour of not cleaned and reference samples	p. 67

## 11.3 List of Abbreviations

Numbers	0. Unaged	1. Aged once	2. Aged twice		
Capital letters	A. Coffee	B. Ashes	C. Sebum		
Lowercase letters	a. Orvus WA Paste	b. Dehypone LS54	c. Gall soap	d. Triammonium Citrate	e. Ethanol
Roman numbers	I. Cotton Swabs	II. Suction Table			

## Appendix 1

### Answers from interviews about stain removal

Type of stain	Interviewees who treated that stain	Cleaning solvents	Treatment methods
<b>Fatty, greasy</b>	10	<ul style="list-style-type: none"> <li>-Gall soap in water</li> <li>-Petrol</li> <li>-Ethanol</li> <li>-Spray containing enzymes called Pure Effect</li> <li>-Washing up liquid, Änglamark, Yes, Neutral</li> <li>-Detergents for machine wash, liquid and in powder form</li> <li>-different types of soaps, Marseille soap, olive soaps, silk soap</li> <li>-Marlipal-</li> <li>-Ampho</li> </ul>	<ul style="list-style-type: none"> <li>-Pre-treat the fatty stain with surfactants</li> <li>-Wet vacuum</li> <li>-Start with mechanical action if needed</li> <li>-Puts a soft towel under stain and treats the stain with a foam</li> <li>-Wet cleaning to rinse the textile after stain removal</li> <li>-Remove the stain with cotton swabs and blotting paper</li> </ul>
<b>Candle wax</b>	11	<ul style="list-style-type: none"> <li>-Residues removed with white spirit (can change colours of the textile), chemically clean petrol, ethanol</li> </ul>	<ul style="list-style-type: none"> <li>-Mechanical action</li> <li>-Heat in combination with blotting paper or other absorbing materials (can cause fatty stains on silk). Heat from heating spatula, steamer, iron</li> </ul>
<b>Coffee</b>	11	<ul style="list-style-type: none"> <li>-Water, distilled , tap, hot, cold</li> <li>-Detergents with enzymes, washing up liquid, Berol</li> <li>-Ammonia</li> <li>-Soap water; Mixture of hot water, soap/washing up liquid/ammonia</li> </ul>	<ul style="list-style-type: none"> <li>-Cotton swabs</li> <li>-Soak in cold water</li> <li>-Wet cleaning</li> <li>-Wets with detergent, removes with blotting paper (capillary forces), wet cleaning</li> </ul>
<b>Coffee with milk (protein)</b>	5	<ul style="list-style-type: none"> <li>-Cold water</li> <li>-Ampho</li> <li>-Enzymes</li> </ul>	<ul style="list-style-type: none"> <li>-Soak in cold water</li> </ul>
<b>Wine</b>	10		

Type of stain	Interviewees who treated that stain	Cleaning solvents	Treatment methods
Ring marks	10	-Water -If there is dirt in the ring mark, add detergent etc	-Localised suction table -Cotton swabs and blotting paper -Gels using capillary forces, keep a blotting paper under the textile -Moisten and dry quickly with a hairdryer from outer edges towards the middle -Fade the ring, dry with hairdryer, heat spatula -Anything with capillary forces -Moistened filter paper with dry filter paper on top, under pressure
Fruit/berries	3	-See wine -Water -Detergents	-Hot water
Degradation products	11	-Cold water -Detergents, anionic, non-ionic, mixtures -Percarbonate bleaching -Borohydride bleaching (reducing)	-Surface cleaning with vacuum cleaner -Soaking in cold water -Wet cleaning -Bleaching
Foxing	10	-Cold water -Hot water and soap water (see coffee) -Percarbonate bleaching -Borohydride bleaching (reducing) -Bicarbonate of Soda	-Wet cleaning -Beaching
Corrosion products	6	-Chelating agents -Water -Petrol	-Protect around stain with cyclododecane -Bleaching -Mechanical action

Type of stain	Interviewees who treated that stain	Cleaning solvents	Treatment methods
<b>Wine</b>	10	<ul style="list-style-type: none"> <li>-70% ethanol, 30% ethanol</li> <li>-Water; cold, hot, distilled</li> <li>-Soap water (see coffee)</li> <li>-Gall soap</li> <li>-Marseille soap</li> <li>-Bicarbonate of soda, sprinkled on the stain</li> <li>-White wine vinegar</li> <li>-Pre-treatment with glycerol</li> <li>-Salt to draw out the stain when it is new</li> </ul>	<ul style="list-style-type: none"> <li>-Localised suction table</li> <li>-Cotton swabs and blotting paper</li> <li>-Foam made from soap (can be hard to remove)</li> <li>-Soaking in cold water</li> </ul>
<b>Blood</b>	9	<ul style="list-style-type: none"> <li>-Saliva on fresh blood (enzymes)</li> <li>-Cold water</li> <li>-After treating with cold water; hot water</li> <li>-Soap water (see coffee)</li> <li>-Washing up liquid</li> <li>-Detergents</li> </ul>	<ul style="list-style-type: none"> <li>-Rinse in water</li> <li>-Foam made from detergent</li> </ul>
<b>Ink</b>	7	<ul style="list-style-type: none"> <li>-Ethanol</li> <li>-Ethyl acetate</li> <li>-IMS/IDA</li> </ul>	<ul style="list-style-type: none"> <li>-Cotton swabs</li> <li>-Localised suction table</li> <li>-Treat on the washing table, to be able to quickly wet to avoid the creation of ring marks</li> <li>-Treat it during wet cleaning, lift the textile and remove ink with cotton swabs</li> <li>-Retouching with seams</li> </ul>
<b>Soot</b>	11	<ul style="list-style-type: none"> <li>-Triammonium citrate /sodium citrate (weaker than triammonium citrate) (chelating agent)</li> <li>-Sodium Percarbonate</li> <li>-Ampho DSK 68, Berol, Orvus, Marseille soap</li> </ul>	<ul style="list-style-type: none"> <li>-Moisten a cloth with citrate and wipe the textile</li> <li>-Wet cleaning</li> <li>-Surface cleaning with vacuum cleaner, smoke sponges, PU sponges (polyurethane)</li> <li>-Wet microfibre cloth</li> </ul>



Type of stain	Interviewees who treated that stain	Cleaning solvents	Treatment methods
Sticky surface stains	6	-Water -Washing up liquid -Petrol, white spirit -Enzymes	-Surface cleaning; vacuum, cotton swabs -Mechanical action
Dark sticky stains	5	-Washing up liquid -Petrol, white spirit	-Mechanical reduction -Use Teas Chart to find suitable solvent
Chewing gum	6		-Mechanical action -Cool it
Grass stains	2	-Water and detergent -Gall soap, washing up liquid	
Make-up	7	-Belongs to the fatty/greasy category -Ethanol -Washing up liquid	-Cotton swabs -Wet cleaning
Un-known stains	9	-Water, distilled, tap -Water + detergent -Solvent; acetone, petrol -Cold water, hot water, soap water (see coffee), other soap mixtures	-Starts with the least invasive cleaning agent and proceeds to more invasive -Use Teas Chart to find suitable solvent -Tries to identify stain, uses microscope -Moist microfibre cloth
Sweat	6	-Water and detergent -Soap water (see coffee) -Acetic acid to make wash solution acidic	-Wet cleaning

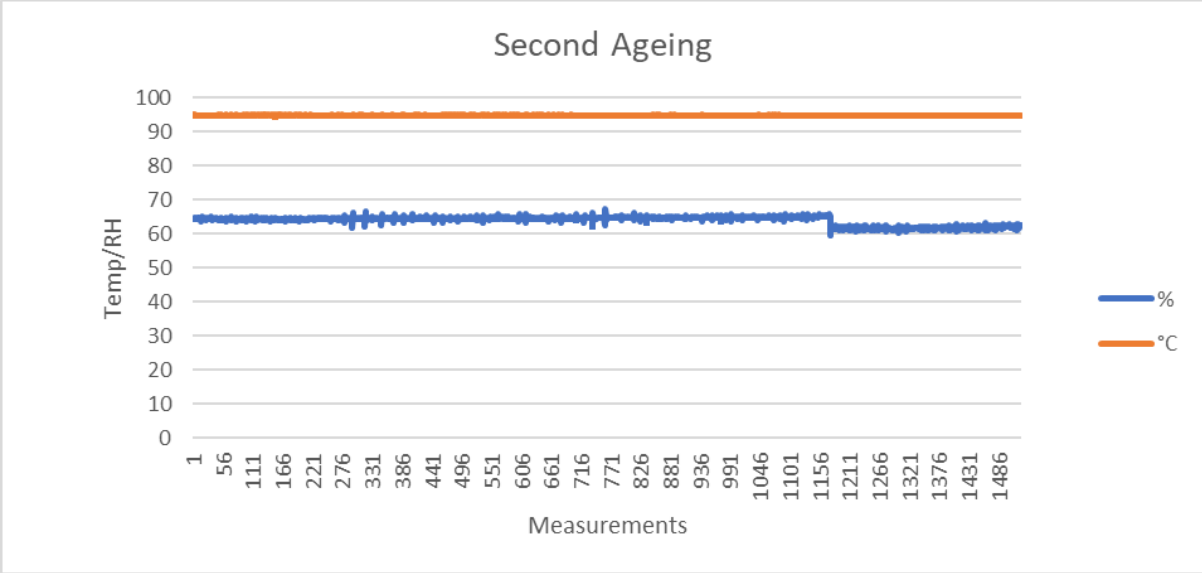
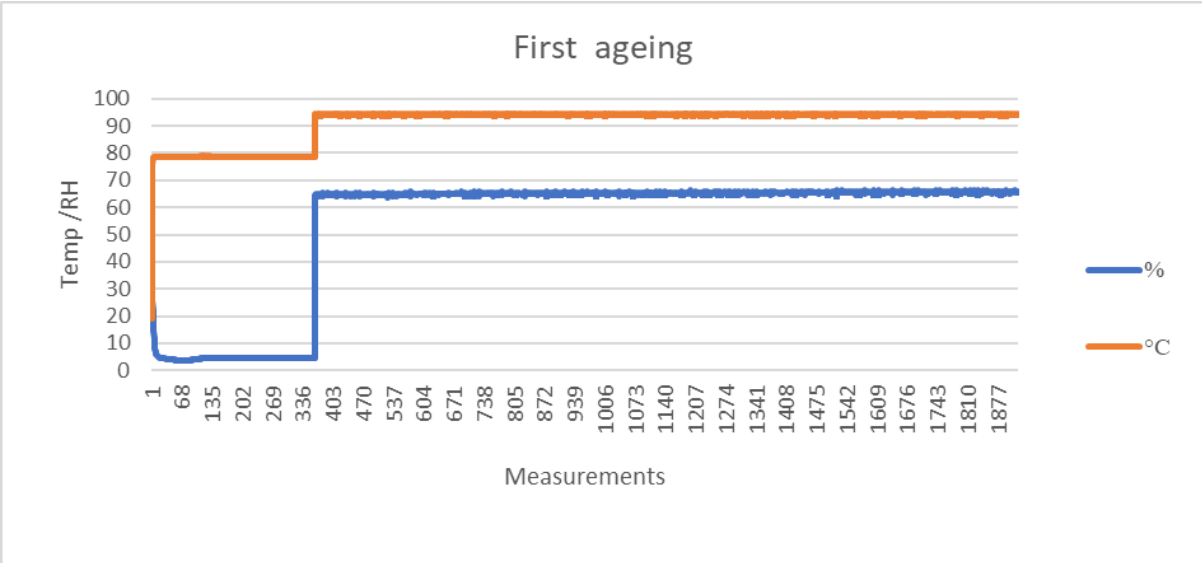
Type of stain	Interviewees who treated that stain	Cleaning solvents	Treatment methods
Flower pistils	5	-Ethanol	-Wet cleaning
Bird droppings	2	-Saliva	-Mechanical action -Vacuum cleaning -Wet cleaning

### Drying methods used by interviewees

Drying method	Interviewees who used that method	Comments
Flat on a washing table or other flat surface	12	-Can use glass slides with weights on the edges
Fan	7	
Dehumidifier	1	
Absorb moisture	11	-Suction table -Removes water with paint roller -Removes water with a natural sponge
Blows it dry	7	-On embroideries where the colour might bleed -3D objects
Towels	11	-White ones, washed without detergents -Can be used with weights on embroideries
Cotton sheets	5	
Blotting paper	3	-Often used on edges of the textile that dries first, residual dirt is drawn to dry areas
Nylon netting 3D	1	-Bulked up to a 3D shape to give access to air from all directions
Wet vacuum cleaner	2	-Used to remove extra moisture

# Appendix 2

Measurements of temperature and RH during the first and second ageing of samples.



# Tensile testing instrument report

Dnr RAÄ-2022-0924

Date of analyses 2023-04-04

Guest colleague Karin Hindborg, Gothenburg university

Analyst Karin Hindborg

Instrument report author Karin Hindborg

Instrument report date 2023-04-06

## Samples

Samples of polyester fabric, all details will be described in Karin Hindborgs master thesis.

## Purpose

Tensile tests were performed in order to assess the influence of stains and cleaning treatments on polyester fabric.

## Method

The standard ISO 13934-1:2013(E) was followed.

### Sample preparation

A white, satin weave polyester fabric for testing was purchased new at Gårda Textil and pre-washed. Each sample consisted of three specimens that were cut to shape and fringed, 5 x 25 cm, before accelerated ageing and the staining and cleaning treatments. The specimens were cut and tested in warp direction. The sample thickness was measured with callipers to 0,28 mm.

Samples named in table 1 and details about the treatments will be provided in Karin Hindborg's thesis (Gothenburg university, 2023).

Table 1. Naming system of samples

Ageing	0. Not aged		1. Aged one time		2. Aged two times	
Staining agent	A. Coffee		B. Ashes		C. Sebum	
Cleaning agents	a. Orvus WA	b. Dehypon LS54	c. Bile soap	d. Tri-ammonium citrate	e. Ethanol	
Cleaning method	I. Cotton swabs			II. Suction table		
Sample	× First sample		O Second sample		□ Third sample	

## **Instrument parameters**

SHIMADZU Autograph AGS-X with Trapezium Lite X software

With 100 N load cell

x 10 kN load cell

x flat grips with rubber pads

**Gauge length** 200 mm

**Speed** 100 mm/min extension

**Pre-test** the samples were clamped centrally and straight in the grips without using pre-tension (slack mounting). A pre-tension applied and the gauge length corrected accordingly.

**Environmental conditions** during measurement 20°C, 65% RH

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## **Results**

Table 2. Tensile test results giving all sample results and their averages for elastic modulus, breaking force, stress, strain, maximum displacement, and the respective standard deviations.

<b>Name</b>	<b>Elastic</b>	<b>Break_Force</b>	<b>Max_Stress</b>	<b>Max_Stroke(Strain)</b>	<b>Max_Displ.</b>
	Force 10 - 20 N	Sensitivity 10	Calc. at Entire Areas	Calc. at Entire Areas	Calc. at Entire Areas
Unit	N/mm <sup>2</sup>	N	N/mm <sup>2</sup>	%	mm
1Aall	82.6	1106.8	79.1	34.6	69.3
1Aall	71.3	1096.8	78.3	36.3	72.6
1Aall	78.1	1096.4	78.3	34.8	69.6
Average	77.3	1100.0	78.6	35.2	70.5
Standard Deviation	5.7	5.9	0.4	0.9	1.9
1Abll	75.9	988.4	70.6	33.2	66.4
1Abll	72.5	1169.9	83.6	36.4	72.8
1Abll	88.4	1186.6	84.8	35.9	71.8
Average	78.9	1115.0	79.6	35.2	70.3
Standard Deviation	8.4	109.9	7.9	1.7	3.4
1Acll	78.2	1130.0	80.7	35.6	71.1
1Acll	74.8	1142.0	81.6	37.3	74.6
1Acll	70.3	1096.4	78.3	35.1	70.1
Average	74.4	1122.8	80.2	36.0	71.9
Standard Deviation	3.9	23.6	1.7	1.2	2.4
1Adll	71.4	1129.8	80.7	38.0	76.1
1Adll	70.3	1073.1	76.7	36.8	73.5
1Adll	70.0	1098.8	78.5	37.7	75.4
Average	70.6	1100.6	78.6	37.5	75.0
Standard Deviation	0.7	28.4	2.0	0.7	1.3
1Ball	67.3	1070.3	76.5	37.2	74.4
1Ball	78.8	1029.8	73.6	34.7	69.3
1Ball	81.5	1054.7	75.3	34.3	68.5

Average	75.9	1051.6	75.1	35.4	70.8
Standard Deviation	7.6	20.4	1.5	1.6	3.2
1BbII	75.3	999.2	71.4	32.7	65.3
1BbII	78.9	1061.8	75.8	34.2	68.4
1BbII	70.6	1087.7	77.7	36.4	72.8
Average	74.9	1049.6	75.0	34.4	68.8
Standard Deviation	4.2	45.5	3.3	1.9	3.7
1BcII	70.3	1123.3	80.2	36.7	73.4
1BcII	70.7	959.5	68.5	33.7	67.4
1BcII	74.9	1147.7	82.1	37.4	74.9
Average	72.0	1076.8	77.0	35.9	71.9
Standard Deviation	2.6	102.4	7.4	2.0	3.9
1BdII	76.6	1158.6	82.8	37.7	75.3
1BdII	73.3	1082.6	77.3	35.7	71.3
1BdII	73.8	1126.4	80.5	37.3	74.5
Average	74.6	1122.5	80.2	36.9	73.7
Standard Deviation	1.8	38.2	2.7	1.1	2.1
1Call	84.0	941.0	67.2	31.8	63.5
1Call	72.4	1078.8	77.1	36.2	72.4
1Call	86.8	881.1	62.9	30.7	61.4
Average	81.1	967.0	69.1	32.9	65.8
Standard Deviation	7.6	101.4	7.2	2.9	5.9
1CbII	70.3	1051.1	75.1	35.8	71.7
1CbII	69.4	963.6	68.8	34.1	68.2
1CbII	71.1	1075.2	76.8	36.7	73.4
Average	70.3	1029.9	73.6	35.5	71.1
Standard Deviation	0.8	58.7	4.2	1.3	2.6
1CcII	70.8	1081.1	77.2	36.7	73.5
1CcII	74.2	1135.4	81.1	37.4	74.8
1CcII	73.5	985.8	70.4	34.1	68.1
Average	72.8	1067.4	76.2	36.1	72.2
Standard Deviation	1.8	75.7	5.4	1.8	3.5
1Cell	70.0	972.1	69.4	34.4	68.8
1Cell	84.3	1000.3	71.4	33.2	66.5
1Cell	75.7	839.5	60.0	30.6	61.1
Average	76.7	937.3	66.9	32.7	65.5
Standard Deviation	7.2	85.8	6.1	2.0	4.0
2AaI	65.4	839.2	59.9	32.8	65.7
2AaI	62.3	833.0	59.5	33.1	66.1

2Aal	62.2	728.4	52.0	30.7	61.5
Average	63.3	800.2	57.2	32.2	64.4
Standard Deviation	1.8	62.3	4.4	1.3	2.6
2Abl	65.2	837.0	59.8	32.6	65.2
2Abl	66.5	875.3	62.5	33.2	66.5
2Abl	64.6	869.7	62.1	33.5	67.0
Average	65.4	860.7	61.5	33.1	66.2
Standard Deviation	0.9	20.7	1.5	0.4	0.9
2Acl	62.7	842.3	60.2	33.3	66.6
2Acl	65.9	834.3	59.6	32.4	64.9
2Acl	64.4	931.3	66.5	34.7	69.4
Average	64.4	869.3	62.1	33.5	66.9
Standard Deviation	1.6	53.8	3.8	1.1	2.3
2Adl	68.4	848.5	60.6	32.1	64.3
2Adl	67.0	941.1	67.2	35.0	70.0
2Adl	64.6	863.9	61.7	33.7	67.4
Average	66.7	884.5	63.2	33.6	67.2
Standard Deviation	1.9	49.6	3.5	1.4	2.9
2Bal	67.4	854.9	61.1	32.1	64.1
2Bal	68.1	867.7	62.0	32.5	65.0
2Bal	64.1	861.0	61.5	33.3	66.5
Average	66.5	861.2	61.5	32.6	65.2
Standard Deviation	2.1	6.4	0.5	0.6	1.2
2Bbl	68.4	780.5	55.7	30.1	60.2
2Bbl	65.7	793.8	56.7	31.2	62.4
2Bbl	70.9	921.8	65.8	33.1	66.2
Average	68.3	832.0	59.4	31.5	63.0
Standard Deviation	2.6	78.0	5.6	1.5	3.0
2Bcl	70.4	817.2	58.4	31.3	62.5
2Bcl	67.9	798.3	57.0	30.6	61.2
2Bcl	66.6	942.4	67.3	34.7	69.4
Average	68.3	852.6	60.9	32.2	64.4
Standard Deviation	2.0	78.3	5.6	2.2	4.4
2Bdl	70.9	895.1	63.9	31.9	63.8
2Bdl	69.0	926.8	66.2	33.6	67.2
2Bdl	69.4	906.3	64.7	32.6	65.1
Average	69.7	909.4	65.0	32.7	65.4
Standard Deviation	1.0	16.1	1.1	0.9	1.7
2Cal	67.5	948.9	67.8	33.6	67.2

2Cal	69.0	961.5	68.7	34.0	68.0
2Cal	68.3	972.1	69.4	34.7	69.3
Average	68.3	960.8	68.6	34.1	68.2
Standard Deviation	0.7	11.6	0.8	0.5	1.1
2Cbl	70.1	958.1	68.4	33.7	67.4
2Cbl	69.9	985.6	70.4	35.0	70.1
2Cbl	71.1	987.2	70.5	34.8	69.6
Average	70.4	977.0	69.8	34.5	69.0
Standard Deviation	0.7	16.3	1.2	0.7	1.4
2Ccl	72.9	981.6	70.1	33.9	67.8
2Ccl	72.3	955.6	68.3	33.8	67.5
2Ccl	71.4	972.3	69.5	34.1	68.2
Average	72.2	969.9	69.3	33.9	67.8
Standard Deviation	0.8	13.2	0.9	0.2	0.3
2Cel	71.4	939.6	67.1	34.0	68.0
2Cel	70.5	977.2	69.8	34.1	68.3
2Cel	73.2	911.7	65.1	32.1	64.2
Average	71.7	942.8	67.3	33.4	66.8
Standard Deviation	1.4	32.9	2.3	1.1	2.2
2	66.7	904.3	64.6	33.8	67.7
2	67.1	948.7	67.8	35.1	70.2
2	67.0	943.0	67.4	33.9	67.8
Average	66.9	932.0	66.6	34.3	68.6
Standard Deviation	0.2	24.1	1.7	0.7	1.4
1B	76.5	1134.6	81.1	37.1	74.2
1B	73.9	1103.4	78.8	36.8	73.5
1B	72.2	1135.6	81.2	38.2	76.5
Average	74.2	1124.5	80.4	37.4	74.7
Standard Deviation	2.1	18.3	1.3	0.8	1.6
1A	84.7	1162.5	83.0	37.6	75.2
1A	72.9	1042.8	74.5	36.1	72.2
1A	74.2	1083.4	77.4	36.8	73.5
Average	77.3	1096.2	78.3	36.8	73.6
Standard Deviation	6.4	60.9	4.3	0.8	1.5
1C	69.7	1134.2	81.0	38.8	77.7
1C	69.3	940.7	67.2	33.9	67.9
1C	68.3	1093.5	78.1	37.9	75.7
Average	69.1	1056.2	75.4	36.9	73.8
Standard Deviation	0.7	102.0	7.3	2.6	5.2



2B	65.8	853.8	61.0	33.2	66.4
2B	65.2	897.9	64.1	34.8	69.6
2B	65.6	873.0	62.4	32.7	65.5
Average	65.5	874.9	62.5	33.6	67.2
Standard Deviation	0.3	22.1	1.6	1.1	2.2
2A	64.7	849.8	60.7	33.9	67.9
2A	67.5	920.1	65.7	35.1	70.2
2A	68.8	932.0	66.6	34.1	68.2
Average	67.0	900.6	64.3	34.4	68.8
Standard Deviation	2.1	44.4	3.2	0.6	1.3
2C	66.2	928.1	66.3	34.2	68.4
2C	65.7	875.8	62.6	33.4	66.8
2C	65.9	853.9	61.0	32.8	65.6
Average	65.9	886.0	63.3	33.5	67.0
Standard Deviation	0.2	38.1	2.7	0.7	1.4
1	71.8	1077.7	77.0	37.1	74.1
1	74.3	1078.8	77.1	36.8	73.6
1	69.3	934.9	66.8	33.6	67.2
Average	71.8	1030.5	73.6	35.8	71.6
Standard Deviation	2.5	82.8	5.9	1.9	3.9
1Aal	72.0	972.3	69.4	34.1	68.2
1Aal	78.5	881.9	63.0	31.8	63.6
1Aal	67.0	974.4	69.6	34.9	69.7
Average	72.5	942.9	67.3	33.6	67.2
Standard Deviation	5.8	52.8	3.8	1.6	3.2
1Abl	74.4	983.4	70.2	33.2	66.3
1Abl	74.0	1073.5	76.7	36.3	72.7
1Abl	73.5	919.4	65.7	32.3	64.7
Average	73.9	992.1	70.9	33.9	67.9
Standard Deviation	0.4	77.4	5.5	2.1	4.2
1Acl	70.7	1058.2	75.6	36.0	72.0
1Acl	79.6	880.6	62.9	31.1	62.1
1Acl	69.2	916.9	65.5	32.7	65.4
Average	73.2	951.9	68.0	33.3	66.5
Standard Deviation	5.6	93.8	6.7	2.5	5.0
1Adl	68.9	898.4	64.2	33.1	66.1
1Adl	72.4	1103.9	78.8	37.5	75.0
1Adl	74.7	953.4	68.1	33.6	67.1
Average	72.0	985.2	70.4	34.7	69.4

Standard Deviation	2.9	106.3	7.6	2.4	4.9
1Bal	74.6	837.6	59.8	30.7	61.5
1Bal	73.4	981.4	70.1	33.7	67.5
1Bal	75.0	1143.8	81.7	38.1	76.1
Average	74.3	987.6	70.5	34.2	68.4
Standard Deviation	0.8	153.2	10.9	3.7	7.4
1Bbl	71.9	980.4	70.0	34.2	68.4
1Bbl	75.7	1035.5	74.0	35.0	70.0
1Bbl	68.8	954.1	68.1	34.6	69.2
Average	72.1	990.0	70.7	34.6	69.2
Standard Deviation	3.4	41.5	3.0	0.4	0.8
1Bcl	68.6	928.1	66.3	33.2	66.3
1Bcl	68.2	810.3	57.9	31.4	62.7
1Bcl	70.0	977.6	69.8	34.0	68.1
Average	68.9	905.3	64.7	32.9	65.7
Standard Deviation	1.0	86.0	6.1	1.4	2.7
1Bdl	74.7	820.1	58.6	30.2	60.4
1Bdl	72.6	974.1	69.6	34.0	67.9
1Bdl	71.8	998.2	71.3	34.6	69.3
Average	73.0	930.8	66.5	32.9	65.9
Standard Deviation	1.5	96.6	6.9	2.4	4.8
1Cal	72.8	913.0	65.2	32.8	65.6
1Cal	72.3	766.6	54.8	29.9	59.8
1Cal	84.8	933.1	66.6	31.5	63.0
Average	76.6	870.9	62.2	31.4	62.8
Standard Deviation	7.0	90.9	6.5	1.5	2.9
1Cbl	68.8	904.1	64.6	32.5	64.9
1Cbl	70.8	1049.9	75.0	36.2	72.5
1Cbl	69.5	956.5	68.3	34.5	69.0
Average	69.7	970.2	69.3	34.4	68.8
Standard Deviation	1.0	73.9	5.3	1.9	3.8
Maximum	70.8	1049.9	75.0	36.2	72.5
Minimum	68.8	904.1	64.6	32.5	64.9
1Ccl	70.7	873.6	62.4	32.3	64.7
1Ccl	72.0	960.7	68.6	33.8	67.6
1Ccl	70.4	900.7	64.3	33.1	66.2
Average	71.0	911.7	65.1	33.1	66.1
Standard Deviation	0.9	44.6	3.2	0.7	1.5
Maximum	72.0	960.7	68.6	33.8	67.6

Minimum	70.4	873.6	62.4	32.3	64.7
1Cel	69.0	1113.3	79.5	38.4	76.8
1Cel	71.7	899.9	64.3	33.0	65.9
1Cel	79.7	855.7	61.1	30.9	61.9
Average	73.4	956.3	68.3	34.1	68.2
Standard Deviation	5.6	137.8	9.8	3.9	7.7
Maximum	79.7	1113.3	79.5	38.4	76.8
Minimum	69.0	855.7	61.1	30.9	61.9
0	84.6	1144.3	81.7	35.3	70.6
0	89.7	1086.0	77.6	33.7	67.5
0	84.3	1062.6	75.9	33.2	66.4
0	106.5	945.6	67.5	30.4	60.7
0	85.4	898.8	64.2	30.5	60.9
0	103.2	1068.2	76.3	33.1	66.2
0	83.0	1103.3	78.8	34.5	69.0
0	88.4	1004.0	71.7	32.1	64.1
Average	90.6	1039.1	74.2	32.8	65.7
Standard Deviation	9.1	83.2	5.9	1.8	3.6
2Aall	66.0	833.1	59.5	32.3	64.5
2Aall	62.0	737.1	52.6	31.4	62.8
2Aall	66.3	866.6	61.9	33.4	66.9
Average	64.8	812.3	58.0	32.4	64.7
Standard Deviation	2.4	67.2	4.8	1.0	2.0
2Abll	64.2	896.6	64.0	34.8	69.7
2Abll	66.1	965.1	68.9	35.6	71.1
2Abll	67.2	882.2	63.0	33.4	66.9
Average	65.8	914.6	65.3	34.6	69.2
Standard Deviation	1.5	44.3	3.2	1.1	2.2
2Acll	64.5	814.6	58.2	32.4	64.8
2Acll	62.9	855.9	61.1	33.4	66.8
2Acll	68.5	826.2	59.0	31.8	63.7
Average	65.3	832.3	59.4	32.5	65.1
Standard Deviation	2.9	21.3	1.5	0.8	1.6
2Adll	66.7	839.1	59.9	32.0	63.9
2Adll	66.2	921.8	65.8	34.6	69.3
2Adll	68.2	942.4	67.5	35.0	69.9
Average	67.0	901.1	64.4	33.9	67.7
Standard Deviation	1.0	54.7	4.0	1.7	3.3
2Ball	69.9	894.2	63.9	33.3	66.7
2Ball	67.3	848.6	60.6	32.3	64.5

2Ball	69.7	911.4	65.1	33.7	67.4
Average	69.0	884.7	63.2	33.1	66.2
Standard Deviation	1.4	32.4	2.3	0.8	1.5
2BbII	66.4	878.9	62.8	33.0	66.1
2BbII	68.9	698.5	49.9	28.9	57.7
2BbII	71.0	881.4	63.0	32.3	64.7
Average	68.7	819.6	58.5	31.4	62.8
Standard Deviation	2.3	104.9	7.5	2.2	4.5
2BcII	68.9	921.4	65.8	34.1	68.3
2BcII	69.3	931.0	66.5	33.9	67.9
2BcII	66.6	902.5	64.5	34.5	68.9
Average	68.2	918.3	65.6	34.2	68.4
Standard Deviation	1.5	14.5	1.0	0.3	0.5
2BdII	72.9	928.4	66.3	33.0	66.0
2BdII	73.3	887.5	63.4	31.4	62.7
2BdII	72.4	888.3	63.5	32.5	65.0
Average	72.9	901.4	64.4	32.3	64.6
Standard Deviation	0.4	23.4	1.7	0.8	1.7
2Call	67.8	927.5	66.3	33.7	67.5
2Call	68.5	936.1	67.0	33.6	67.1
2Call	68.8	930.6	66.5	34.2	68.5
Average	68.4	931.4	66.6	33.9	67.7
Standard Deviation	0.5	4.4	0.4	0.4	0.7
2CbII	73.3	936.8	66.9	34.2	68.5
2CbII	70.9	847.1	60.5	31.7	63.5
2CbII	70.0	912.9	65.2	32.9	65.8
Average	71.4	898.9	64.2	33.0	65.9
Standard Deviation	1.7	46.4	3.3	1.3	2.5
2CcII	68.2	863.0	61.6	32.8	65.6
2CcII	67.8	880.4	62.9	33.1	66.2
2CcII	71.5	948.5	67.8	33.1	66.3
Average	69.2	897.3	64.1	33.0	66.0
Standard Deviation	2.0	45.2	3.2	0.2	0.4
2Cell	71.2	932.0	66.6	33.5	67.1
2Cell	70.4	750.7	53.6	29.7	59.5
2Cell	71.6	827.6	59.1	31.1	62.1
Average	71.0	836.8	59.8	31.4	62.9
Standard Deviation	0.6	91.0	6.5	1.9	3.9

## Appendix 4

### Colour measurements, delta E, L, a and b.

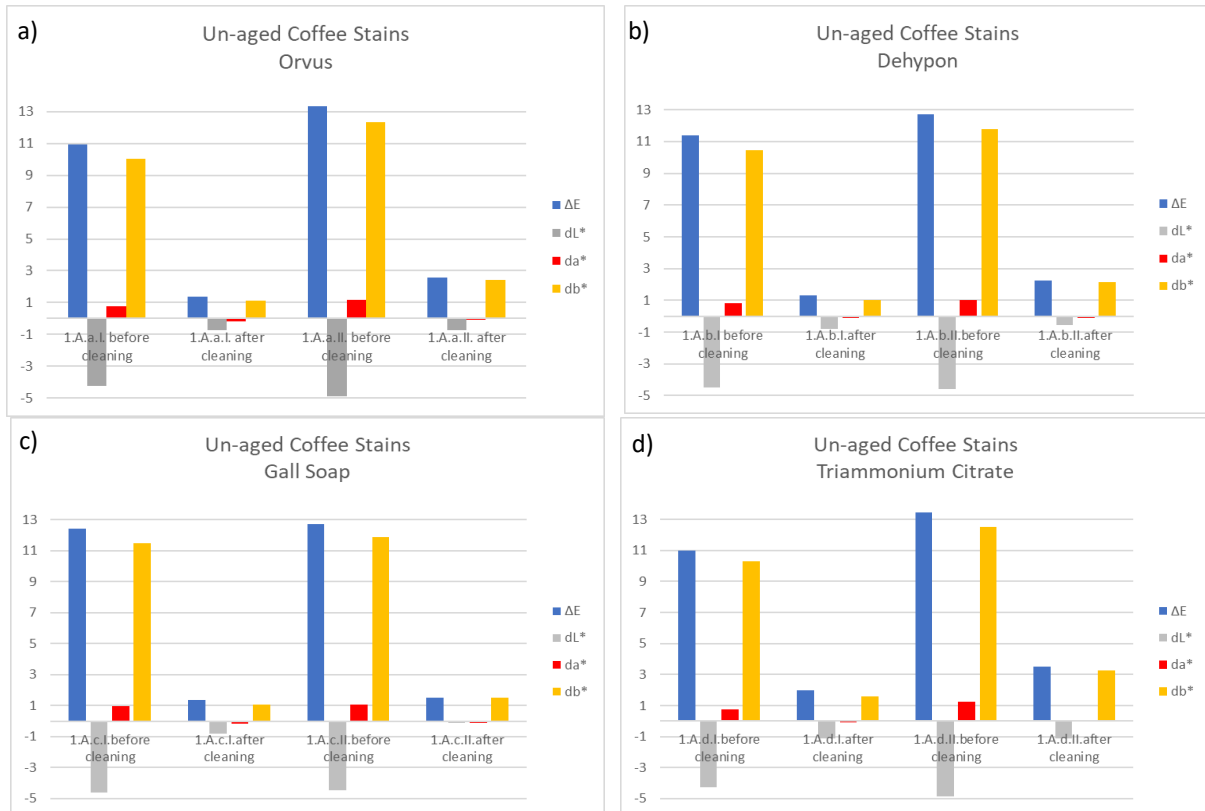


Fig. 38. a) Colour measurements of un-aged coffee stains treated with Orvus WA Paste.

b) Colour measurements of un-aged coffee stains treated with Dehypon LS54.

c) Colour measurements of un-aged coffee stains treated with Gall soap.

d) Colour measurements of un-aged coffee stains treated with triammonium citrate.

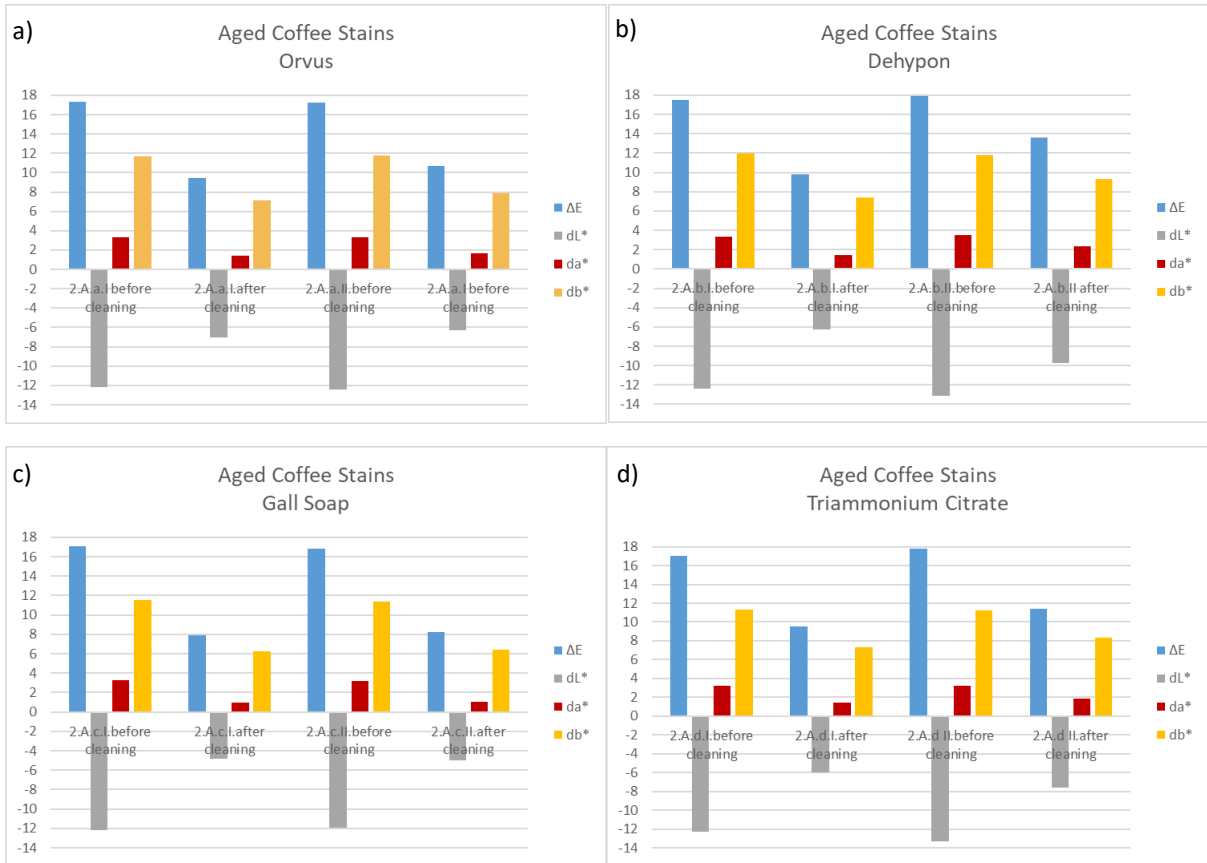


Fig. 39. a) Colour measurements of un-aged coffee stains treated with Orvus WA Paste.

b) Colour measurements of un-aged coffee stains treated with Dehypon LS54.

c) Colour measurements of un-aged coffee stains treated with Gall soap.

d) Colour measurements of un-aged coffee stains treated with triammonium citrate.

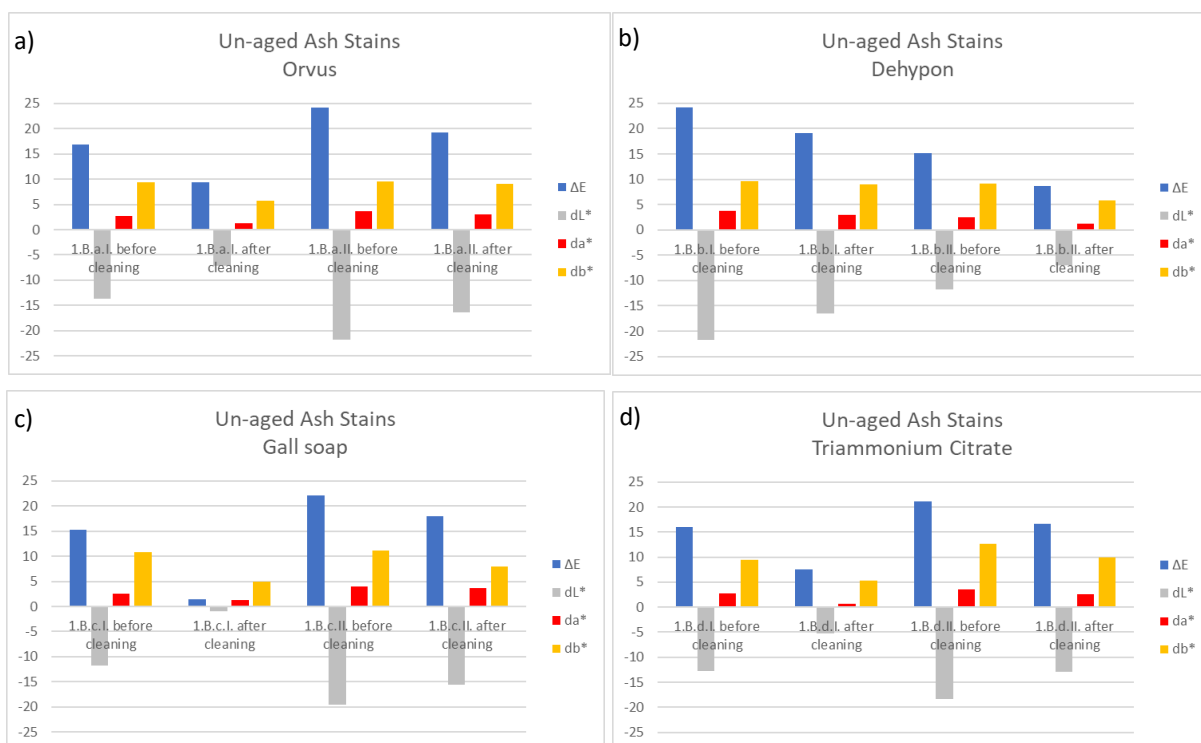


Fig. 40. a) Colour measurements of un-aged ash stains treated with Orvus WA Paste.

b) Colour measurements of un-aged ash stains treated with Dehypon LS54.

c) Colour measurements of un-aged ash stains treated with Gall soap.

d) Colour measurements of un-aged ash stains treated with triammonium citrate.

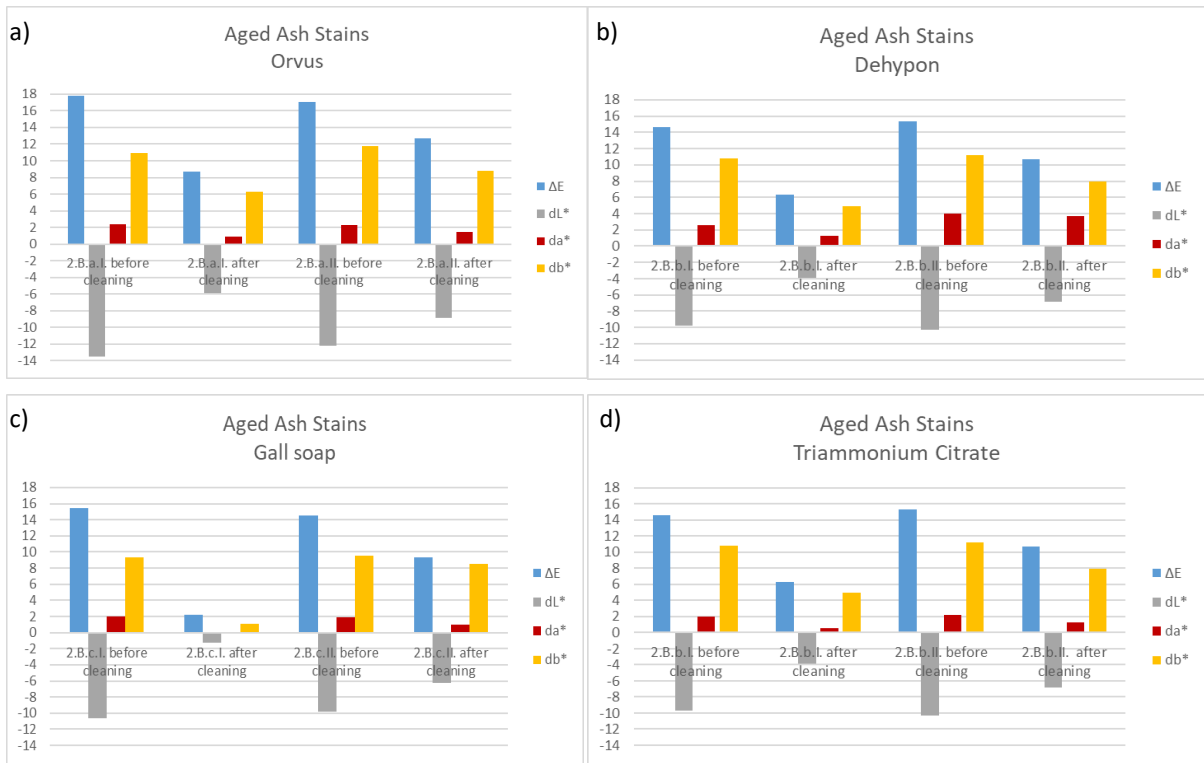


Fig. 41. a) Colour measurements of aged ash stains treated with Orvus WA Paste.

b) Colour measurements of aged ash stains treated with Dehypon LS54.

c) Colour measurements of aged ash stains treated with Gall soap.

d) Colour measurements of aged ash stains treated with triammonium citrate.



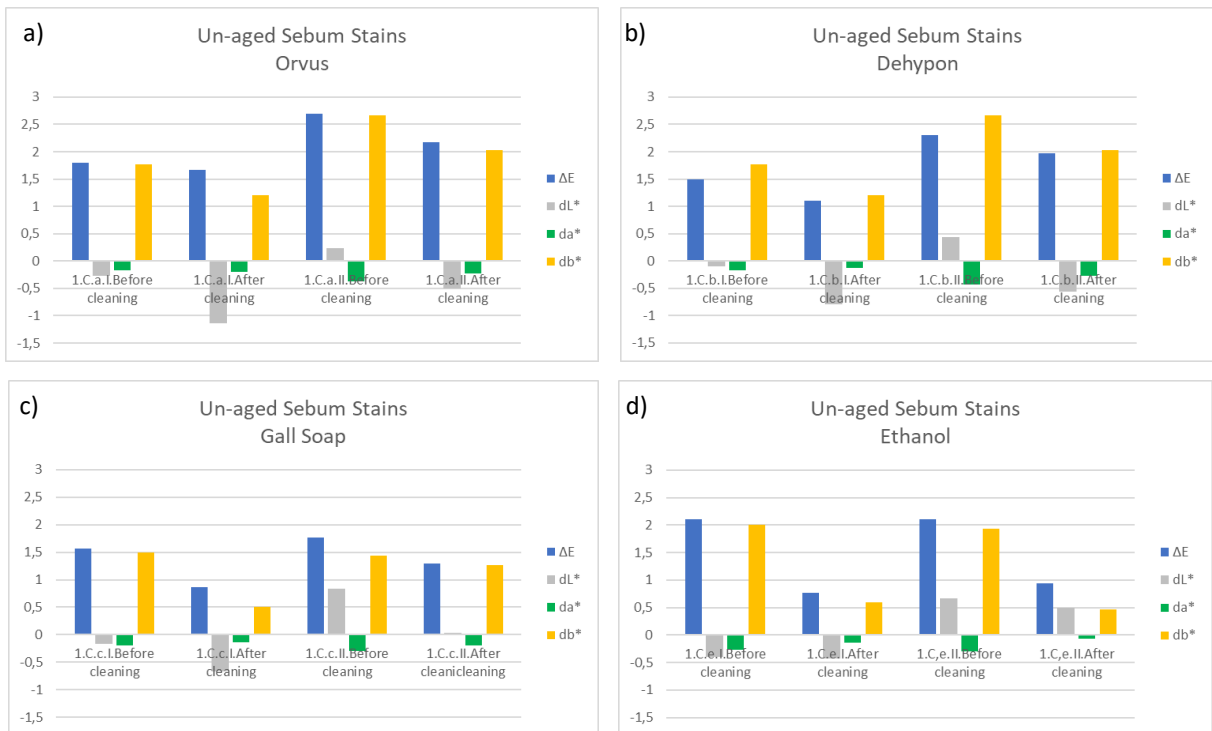


Fig. 42. a) Colour measurements of un-aged sebum stains treated with Orvus WA Paste.

b) Colour measurements of un-aged sebum stains treated with Dehypon LS54.

c) Colour measurements of un-aged sebum stains treated with Gall soap.

d) Colour measurements of un-aged sebum stains treated with ethanol.

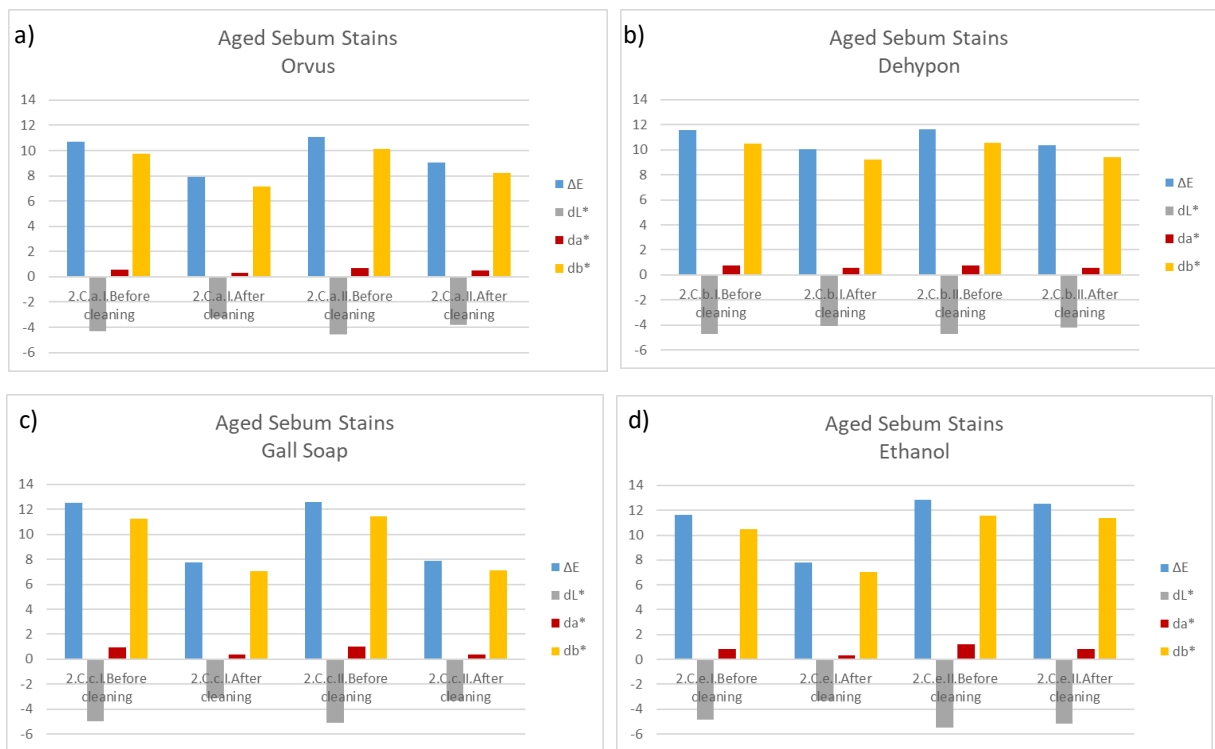


Fig. 43. a) Colour measurements of aged sebum stains treated with Orvus WA Paste.

b) Colour measurements of aged sebum stains treated with Dehypon LS54.

c) Colour measurements of aged sebum stains treated with Gall soap.

d) Colour measurements of aged sebum stains treated with ethanol.