

AGEING AND CONSERVATION OF SILK

Evaluation of Three Support Methods
Using Artificially Aged Silk

Johanna Nilsson



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ISBN 978-91-7346-851-0 (printed)
978-91-7346-852-7 (pdf)
ISSN 0284-6578

The publication is also available in full text at: http://hdl.handle.net/2077/40524

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Cover: Detail of Karl X Gustav's cloak from 1654, inv no Lrk 21998. Courtesy Tove Nilsson. Portrait of author by Jens Mohr. Page 16: Detail of Karl X Gustav's cloak from 1654, inv no Lrk 21998. Courtesy Tove Nilsson. Pages 20 and 42: Details of Gustav II Adolf's coronation doublet, inv no Lrk 25605. Courtesy Göran Schmidt.

Layout: Jonathan Westin. Print: Ineko, Kållered 2015.

Abstract

The general aim of this thesis is to evaluate common remedial conservation support methods used in the conservation treatments of fragile silk costumes that have experienced physical damage. It is based on five papers. Paper I surveyed the methods textile conservators use to conserve historic textile costumes and their criteria for a successful intervention. It also investigated artificial ageing of modern silk and wool fabrics with exposure to ultraviolet radiation to create material for laboratory-based experimental research. Furthermore, it evaluated mechanical methods to imitate natural wear in silk and wool fabrics, to simulate the handling of conserved costumes, and to find a method to evaluate the effect of the conservation methods. The most common conservation method conservators reported using was to insert a support fabric between the outer fabric and the lining of a costume, which was then sewn on by the couching method over the outer fabric. The most important criterion for a successful conservation was aesthetical appeal. Abrasion by Nu-Martindale and tensile testing were found promising to use to achieve natural accelerated wear and to evaluate conservation methods.

Paper II aimed at finding an optimal accelerated ageing protocol to simulate the nature and degree of degradation found in naturally aged seventeenth century silk fabric in order to produce surrogates for experimental research. Tensile tests, Attenuated Total Reflection - Fourier Transform Infrared spectroscopy (ATR-FTIR) and Size Exclusion Chromatography (SEC), were investigated as methods for evaluating the results. Four environmental parameters were tested: relative humidity (RH), acidity (pH), ultraviolet irradiation (UV) and thermo-oxida-

tion. For Paper III, further investigations were carried out to establish analytical markers for aged silk by additional analytical methods. The investigations were successfully complemented and verified using amino acid analysis, and measurement of pH and brightness. In both Papers II and III it was established that thermo-oxidation at 125°C was the most suitable ageing method.

In Paper IV two types of experimental damage on silk surrogates were conserved with three different methods: brick couching, laid couching, and crepeline. The conserved surrogates were further subjected to accelerated wear by using a combination of washing and tumbling, followed by tensile testing. The three interventions increased the surrogates' strength from three to more than five times. Surrogates with a tear conserved with laid or brick couching were the least affected by wear; and, after the conservation was removed, the abraded surrogates

conserved with crepeline were stronger than those conserved with the other two methods. Paper V explored the factors that determine aesthetic quality of conservation interventions. The study, based upon examinations performed by Swedish textile conservators, resulted in two factors coherence and completeness, that describe aesthetic quality.

TITLE: Ageing and Conservation of Silk: Evaluation of Three Support Methods Using Artificially Aged Silk

LANGUAGE: English

ISBN: 978-91-7346-851-0 (printed)

978-91-7346-852-7 (pdf)

ISSN: 0284-6578

KEYWORDS: Artificial ageing of silk, textile conservation, historic silk costumes, stitching techniques, aesthetics

List of Papers

The present doctoral thesis is based on the following five papers, which will be referred to in the text with their Roman numerals.

- I. Johanna Nilsson. "A survey of the most common support methods used on historical costumes and a preliminary investigation of tests assessing the quality of conserved fabrics." Post print at the conference *Scientific Analysis of Ancient and Historic Textiles*, 2005, University of Southampton, eds. R. Janaway & P. Wyeth, p. 79-85.
- II. Johanna Nilsson, Francisco Vilaplana, Sigbritt Karlsson, Jonny Bjurman, and Tommy Iversen. "The validation of artificial ageing methods for silk textiles using markers for chemical and physical properties of seventeenth century silk." *Studies in Conservation*, 2010, 55 (1) p. 55-65.
- III. Francisco Vilaplana, Johanna Nilsson, Dorte Vestergaard Poulsen Sommer, and Sig-

britt Karlsson, "Analytical markers for the degradation of historic and artificially aged silk in different environments.", *Analytical and Bioanalytical Chemistry*, 2015, 407, (5) p. 1433-1449. DOI 10.1007/s00216-014-8361-z

- **IV**. Johanna Nilsson. "Evaluation of stitched support methods for the remedial conservation of historic silk costumes." *e-conservation Journal*, 2015, 3. DOI 10.18236/econs3.201506
- V. Johanna Nilsson and Östen Axelsson. "Attributes of aesthetic quality used by textile conservators, evaluating conservation interventions on museum costumes.", *Perceptual and Motor Skills*, 2015, 121, (1), p. 199-218. DOI 10.2466/27.24. PMS.121CIOX7

Paper II was written in collaboration with Dr Francisco Vilaplana, Professor Sigbritt Karlsson, Professor Jonny Bjurman and Professor Tommy Iversen, all chemists. Vilaplana and Karlsson were at that time at the Swedish Royal Institute of Technology, KTH, Stockholm, Department of Fibre and Polymer Technology, School of Chemical Science and Engineering. Bjurman is at the Department of Conservation, University of Gothenburg, and Iversen was at the time at Innventia AB.

The chemical analyses, ATR-FTIR, and SEC were made in collaboration with Dr Francisco Vilaplana and Professor Sigbritt Karlsson. Nilsson carried out the ATR-FTIR analyses under the supervision of Francisco Vilaplana who analysed the results. The SEC analyses were performed by Vilaplana under supervision of the staff at Innventia AB.

Paper III was a collaboration between Dr Francisco Vilaplana, Dorte V. P. Sommer, Professor Sigbritt Karlsson and Johanna Nilsson. Assistant Professor V. P. Sommer is at the Royal Danish Academy of Fine Arts Schools of Architecture, Design and Conservation - School of Conservation.

The ATR-FTIR analyses were performed by Francisco Vilaplana who also performed the SEC analyses under the supervision of the staff at Innventia AB. The pH measurements were performed at the laboratories of KTH Biotechnology/Glycoscience Division and Wallenberg Wood Science Centre, AlbaNova University Center in collaboration between Vilaplana and Nilsson. The amino acid analyses were made by Anne Blicher at The Technical University of Denmark, Department of System Biology, and the analyses were interpreted by V. P. Sommer.

Paper V was made in collaboration with Dr Östen Axelsson at Gösta Ekman Laboratory, Department of Psychology, Stockholm University. Axelsson designed the experimental part and performed the statistical analyses.

Sammanfattning på svenska

Livrustkammaren har unika samlingar av kungliga dräkter alltifrån sent 1500-tal till nutid och en stor del av dräkterna är tillverkade i siden, ett material som är komplicerat att konservera. Många av dräkterna är efterfrågade till utställningar i Sverige och utomlands. Detta medför att redan mycket sköra dräkter utsätts för slitage. Om dräkterna ska hålla och kunna bevaras måste de konserveras. Då många av dessa dräkter har konserverats ett flertal gånger med olika metoder väcktes frågan om vilken metod som är bäst att använda för skört siden.

Huvudsyftet med avhandlingen är att utvärdera konserveringsmetoder för historiska dräkter i siden genom att experimentellt pröva tre vanliga konserveringsmetoder; (1) underlagning med läggsöm, (2) underlagning med förstygn och (3) crepeline.

För att kunna göra utvärderingen behövde jag nå ett antal delmål:

- I. Kartlägga vad textilkonservatorer anser vara det viktigaste kriteriet för en lyckad konservering.
- 2. Kartlägga vilka de vanligaste sömnadsmetoderna för konservering av dräkt är.
- 3. Ta fram metoder för att åldra siden artificiellt så att de kemiska och mekaniska egenskaperna liknar 1600-talssiden och det artificiellt åldrade sidenet kan ersätta det historiska sidenet i testerna enligt punkt 4-7.
- Undersöka hur de vanligaste sömnadskonserveringarna påverkar styrkan hos en konserverad provlapp.
- 5. Undersöka hur en konserverad provlapp påverkas av accelererad nötning.
- 6. Undersöka hur sömnadskonserveringarna påverkat provlappen efter accelererad nötning då konserveringen avlägsnats.

- 7. Undersöka hur lång tid det tar att utföra de olika sömnadskonserveringarna.
- 8. Undersöka vilka egenskaper som gör att en sömnadskonservering uppfattas som estetiskt tilltalande av textilkonservatorer.

Dessa undersökningar har genomförts i ett antal studier som presenteras i fem artiklar, vilka utgör basen för avhandlingen, och i denna svenska sammanfattning redogör jag för resultatet från dessa. Jag har valt att inte sammanfatta inledningen som innehåller beskrivningar av textilkonserveringens utveckling och villkor, materialet siden, konserveringstekniker och material. De fem artiklarna finns bifogade i slutet av den tryckta avhandlingen.

Den första studien (Paper I) består av två delar, en enkät och en serie experiment. Textilkonservatorer från konserveringsateljéer i Europa och Nordamerika besvarade en enkät som bl a gällde vilka sömnadsmetoder som användes för dräktkonservering och vilka kriterier som ansågs viktiga bland textilkonservatorer för att en konservering ska bedömas som lyckad. Den experimentella delen inbegrep ylle och siden som åldrats artificiellt med en kombination av ultraviolett strålning, fukt och temperatur (fortsättningsvis som helhet förkortat till UV) för att få ett skört material att använda i de följande experimenten där olika mekaniska metoder skulle prövas i sin förmåga att orsaka naturlig nötning och accelererat slitage (för att åstadkomma samma slitage som ett föremål utsätts för under lång tid). Jag ville också hitta en metod för att utvärdera effekten av olika konserveringsmetoder. Enkäten visade att den vanligaste konserveringsmetoden var underlagning med läggsöm och det viktigaste kriteriet för en lyckad konservering var att den är estetiskt

tilltalande. För den experimentella delen användes standardtyg i siden (ISO 105-F06:2000) och ylle (ISO 105-F01:2001). Av standardtygerna tillverkades åldrade och oåldrade provlappar, konserverade med läggsöm över olika stora ytor och okonserverade provlappar. Provlapparna utsattes sedan för fem olika mekaniska tester varvid nötning med Nu-Martindale och dragtest var de metoder som då bedömdes kunna fungera för att nöta och slita tygerna samt för att utvärdera effekten av konserveringsmetoderna.

Den andra studien (Paper II) syftade till att hitta en metod för att framställa ett siden som påminner om historiskt siden med prover från två 1600-talsdräkter ur Livrustkammarens samling som referenser. Syftet med att artificiellt åldra siden var att kunna använda det som försöksmaterial i kommande experiment. Avhandlingsämnet hade då begränsats till att bara gälla siden. Både kemiska och mekaniska analyser användes för att finna markörer för nedbrytning av siden. Attenuated Total Reflectance - Fourier Transform Infrared spectroscopy (ATR-FTIR) användes för att analysera molekylstrukturen i sidenets yta, och Size Exclusion Chromatography (SEC) användes för bestämning av den relativa molekylvikten. Mekaniska egenskaper undersöktes genom att mäta töjning, styrka och materialets motstånd vid sträckning (Young's modulus) genom dragtester. Fyra olika metoder för artificiellt åldrande prövades i ett antal variationer på vitt standardsiden (ISO 105-F06:2000): UV (samma metod som i Paper I), värmebehandling, olika pH-värden samt olika fukthalter. Genom ATR-FTIR-analyserna kunde det konstateras att en markör för nedbrytning är förekomst av karbonylgrupper i de historiska dräkterna, i de provlappar som åldrats genom UV-strålning och i de

som värmebehandlats i 125°C. Markören saknades hos de prover som exponerats för hög luftfuktighet och de som utsatts för buffertlösningar med högt pH-värde. Tyrosin (Tyr) är en aminosyra i silkets amorfa delar och visade sig också vara en markör för nedbrytning. I det historiska silket var intensiteten något lägre för tyrosinets dubbelband än i det oåldrade sidenet. Liknande värden som för historiskt silke hade också siden som åldrats med pH 13 eller värmebehandling i 125°C i 28-56 dygn. Inga nämnvärda förändringar fanns i de prover som utsatts för olika fukthalter eller låga pH-värden. UV-strålning däremot gav kraftig minskning av Tyr. En storleksfördelning med två maxima kunde observeras med SEC-analyser för det oåldrade sidenet men ännu tydligare för det artificiellt åldrade och för de historiska referenserna. Detta kan bero på silkets uppbyggnad med två dominerande peptidkedjor. Generellt visade de historiska proverna en lägre relativ molekylvikt än de artificiellt åldrade proverna. Molekylvikten påverkades inte nämnvärt för de prover som åldrats genom olika fukthalter. Behandling med sur buffertlösning (pH 1) och basisk buffertlösning (pH 13) gav däremot en minskning av molekylvikten. Resultaten av SEC-analyserna för de UVexponerade proverna är något osäkra då silket löste upp sig annorlunda än i de andra proverna. De värmebehandlade proverna hade en lägre relativ molekylvikt än det oåldrade sidenet. För de mekaniska egenskaperna användes dragtester. De visade att åldrande i 100% RH i 28 dygn eller i lågt pH-värde (pH 1) hade mycket liten effekt på de mekaniska egenskaperna. De prover som behandlats i pH 13 däremot förstördes totalt och kunde inte testas. Värmebehandling i 125°C och UV visade en reduktion av styrka och töjning som påminner om resultaten för de historiska proverna. Däremot är värdena för motstånd vid sträckning (Young's modulus) inte lika de historiska proverna som har högre värden.

I den tredje studien (Paper III) utvidgades undersökningarna från den andra studien (Paper II) med fler analysmetoder för att ytterligare fastställa markörer för silkes nedbrytning: mätning av pH, mätning av ljusstyrka och aminosyraanalyser. Dessutom redovisades alla data från samtliga dragtestade prover och resultaten signifikanttestades. pH-mätningarna visade att proverna från de historiska dräkterna och de prover som UV-exponerats i tio dygn och proverna som värmeexponerats i 125°C i 28 dygn liknade varandra. Mätningar av ljusstyrkan som talar om hur mycket tyget har gulnat och därmed färgförändrats utfördes. Mätningarna kunde inte genomföras för de historiska proverna. De UV-exponerade proverna förlorade i ljusstyrka särskilt de första 2-4 dygnen med ett liknande resultat för proverna som utsatts för pH 1 och pH 13. Värme i 125°C orsakade linjärt avtagande ljusstyrka vartefter exponeringstiden ökade. Fukt påverkade inte ljusstyrkan på proverna. Aminosyraanalyserna bekräftar resultaten av de tidigare analyserna med ATR-FTIR. UV-exponering orsakar en märkbar minskning av Tyr, detta gäller även för de värme-exponerade proverna om än inte i samma omfattning. Även proverna från de historiska dräkterna visar på lägre halter av Tyr, vilket tyder på att oxidering sker vid naturligt åldrande av siden. Inga påvisbara förändringar finns hos de prover som utsatts för olika pH och fukthalter. I jämförelse med oåldrat standardsiden hade de historiska sidenproverna endast något högre modulusvärden men var kraftigt reducerade vad gällde styrka och töjning. Det historiska sidenets styrka var i genomsnitt 17% av styrkan hos oåldrat standardsiden. UV-exponering höjde provernas modulus-värden kraftigt de första dygnen men efter 5-6 dygn hade de sjunkit till ungefär samma värde som oåldrat standardsiden. Däremot sjönk värdena för styrka och töjning från första dygnet. Värme i 125°C minskade styrkan och töjningen direkt men planade ut efter 28 dygn medan modulus-värdet förblev högt. Utifrån de olika analyserna i Paper II och III kunde vi fastställa att värme i 125°C var den metod som bäst lämpade sig för att åldra standardsiden så att det liknar referensproverna från 1600-talsdräkterna.

I den fjärde studien (Paper IV) undersöktes genom dragtest hur styrkan påverkats på artificiellt åldrade provlappar med reva eller nötskada när de konserverats med antingen läggsöm, förstygn eller crepeline. Provlapparna tillverkades av standardsiden som åldrades i 125°C i 56 dygn och skadorna framställdes antingen genom ett snitt med skalpell för att skära en reva eller genom Nu-Martindale för att framställa en söndernött yta. Provlappar dragtestades efter att de konserverats med de tre olika konserveringsmetoderna, efter att de konserverade provlapparna utsatts för accelererat slitage genom tvätt och torktumling och efter att konserveringen tagits bort. Metoden för att simulera slitage liknar förstås inte den nötning som uppstår vid naturlig hantering men jag behövde uppnå en stor effekt på relativt kort tid. De tre konserveringsmetoderna ökade provlapparnas styrka, före slitage, med tre till mer än fem gånger. Läggsöm på reva var den metod som gav den starkaste provlappen. Provlappar med reva konserverad med läggsöm eller förstygn var mindre påverkade av accelererat slitage än

de som konserverats med crepeline. Efter att konserveringen tagits bort var provlapparna med nötskada som konserverats med crepeline starkare än de som konserverats med läggsöm och förstygn. Konservering av en nött yta med crepeline skadade provlappen mindre än läggsöm och förstygn. I filmen "Sömnadskonservering med läggsöm, förstygn och crepeline" kan man se hur provlapparna konserverades (YouTube-film Nilsson), Appendix 2.

Frågan om hur lång tid de olika konserveringsmetoderna tar kräver mer omfattande undersökningar då förutsättningarna kan variera så mycket, t ex vad som ska konserveras och vem som utför konserveringen.

I den femte studien (Paper V) undersöktes vilka faktorer som enligt textilkonservatorer i Sverige avgör en konserverings estetiska kvalitet. I det första experimentet deltog 24 konservatorer som fick till uppgift att rangordna 33 fotografier på konserveringar utförda på dräkter ur Livrustkammarens och Hallwylska museets samlingar. Konserveringarna var utförda med läggsöm, förstygn eller crepeline. Deltagarna sorterade fotografierna i tre grupper utifrån sina uppfattningar om hög, medel och låg estetisk kvalitet. Därefter intervjuades de om vilka kriterier de hade beaktat under sorteringen. Utifrån intervjuerna skapades sju beskrivande meningar som återgav konservatorernas uppfattning om konserveringarna i de tre grupperna. I påföljande experiment deltog en ny grupp av tio textilkonservatorer med syfte att få fram de viktigaste faktorerna för estetisk kvalitet. De fick till uppgift att sortera samma 33 fotografier från det första experimentet sju gånger utifrån de beskrivande meningarna från det första experimentet. Genom en analys som

rangordnar svaren utkristalliserades två faktorer som representerade estetisk kvalitet. Min medförfattare och jag tolkade dessa som samstämmighet och fullständighet. Ytterligare en analys som talar om förhållandet mellan de olika faktorerna visade att det endast är samstämmighet som är signifikant. Detta innebär att en lyckad konservering från ett estetiskt perspektiv innebär att konserveringen är välintegrerad med föremålet vad gäller material och metod. Fullständighet innebär att hela skadan på ett föremål är konserverad.

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Foreword

It has been a privilege to get the opportunity to dig into and deepen my knowledge in a subject that really engages me but also is a challenge. I have always felt confidence in my choice of subject: evaluation of conservation methods for historic costumes. After many years as a textile conservator with my own experiences but building on other conservators' experiences I had a need to get a more science based knowledge of the preserving capability and effect that different conservation interventions have. But also to find out what criteria conservators have for aesthetic interventions. It was natural to me to narrow my studies to concern fragile silk costumes, as silk is a common material in the collections of the Royal Armoury and the Hallwyl Museum where I work and it is a material that is difficult to treat. To fulfil the aim with this thesis it was necessary to establish an interdisciplinary way of working. A challenge, but also a joy, has been the cooperation with people in other disciplines. Mechanical tests have been carried out at Swerea IVF AB in Mölndal, a textile research company. The company generally performs tests for the textile industry, and a challenge was to communicate my aims and the limitations and complexity of my material. Furthermore, a challenge for me was to understand the limitations and possibilities of their equipment, as well as to interpret the tensile tests and to analyse the results correctly. It has given me the possibility to work in an advanced textile research lab with an equipment of high standard and an experienced staff. Further, the chemical studies have been made in cooperation with several chemists and with an expert in leather conservation who masters amino acid analyses. I have also cooperated with a psychologist, an expert in psychological

analyses of aesthetic experiences with an interest to test his methods on aesthetics in textile conservation. This cooperation has given me the possibility to broaden my research project with methods I do not master myself and to develop my skills in performing experiments and writing scientific papers. Another challenge has been to find journals with an understanding of these types of interdisciplinary studies. Interdisciplinary studies are encouraged in general, but to many reviewers they might seem too complex and even bewildering. It has been difficult to find publications with an understanding of the broad issues, the methods used, and the statistics presented and therefore the papers, which form the basis of this thesis, are published not only in journals of conservation, but also in journals of chemistry and psychology.

Acknowledgements

This research was made possible with grants from Agnes Geijer's Foundation for Textile Research: Scholarships, Gunvor and Josef Anér's Foundation, Märta, Gunnar, and Arvid Bothén's Foundation, The Barbro Osher Pro Suecia Foundation, The Gyllenstiernska Krapperup Foundation, The Royal Patriotic Society, The Royal Society of Arts and Sciences in Gothenburg. The printing of this thesis was sponsored by Berit Wallenberg Foundation.

I am grateful to my employer LSH - a museum authority under the Government - comprising the Royal Armoury, Skokloster Castle and the Hallwyl Museum Foundation with its Director General Magnus Hagberg, and former Director General, Associate Professor Barbro Bursell, who have generously allowed me to pursue my research during work hours. A great thanks to

Professor Ola Wetterberg from the Department of Conservation, University of Gothenburg, for his valuable support as my examiner, and for the economical agreement between LSH and the university for my studies. Thanks also to my supervisors Professor Elizabeth Peacock and Professor Jonny Bjurman, who have helped me with clever advice, and with the language of both English and chemistry. I am very grateful to Assistant Professor Dorte V. P. Sommer, Dr Francisco Vilaplana, Professor Jonny Bjurman, Professor Sigbritt Karlsson, Associate Professor Tommy Iversen, and Dr Östen Axelsson, that you agreed to be a part in this project as authors and also for being invaluable advisors. Professor Rolf Sandell is thanked for support of the statistical analyses. Dr Marianne Nygren, Professor Arne Nygren, and Dr Erik Nilsson are thanked for valuable comments on Paper IV. Textile engineer Karin Christiansen at Swerea IVF AB is thanked for being so helpful during the cooperation with the tensile tests.

I also want to thank all Swedish textile conservation colleagues; I have always felt a great support from you, which has been very important to me during the process. Many of you textile colleagues have willingly volunteered to my studies and experiments, thank you all. Augusta Persson and Margit Forsberg are especially thanked for the conservation of surrogates, Anna Ehn-Lundgren for the pre-test of the aesthetic attributes, Anna Stow for the language check and English voice in the film. I am grateful to my LSH colleagues for their patience with me, their encouragement and help to keep the project economy and applications in order, especially Milla Springfeldt, who also helped me with the final proof of the thesis. A special thanks goes to "old KoF" colleagues and my friends at the Department of collections in LSH - work is so much easier and enjoyable with you. Jens Mohr and Erik Lernestål, LSH's photographers, are two of these important colleagues who have helped me a lot. It has been a pleasure to get to know my colleagues at the Department of Conservation, University of Gothenburg, who are very supportive. Special thanks go to Maria Hörnlund for helping me finding references, and to Katarina Östling for correcting the reference list, to Dr Maj Ringaard, Fil. lic. Tina Westerlund, and Dr Ingalill Nyström for helpful comments on the thesis at the final seminar. Thank you to Jonathan Westin for the formatting and layout of my thesis. I am grateful to Jonathan Sköld for his work with the film. I am also in debt to the textile conservators Ann French, Francis Lennard, Mary Brooks, and Sarah Benson for guidance of the English terminology of stitches. Marie-Louise Wulfcrona, Marie Wallenberg, and Agneta von Essen are thanked for introducing me to the profession of textile conservation.

Last but not least, my family is thanked, in many ways this project has been run as teamwork with my family as my closest and most devoted team. My parents, Boel and Carl-Otto Jonsson, for their never-ending patience, competence and help, cannot be thanked enough. My brother Jakob Jonsson is thanked for statistic support, and my father for the statistical analyses. My children, Tove who has acted as photographer, Isak who has made advanced illustrations and film music, amongst many other things, and Hillevi who has contributed with image editing, they are all greatly thanked. To my husband Robban Nilsson I do not know what to say, really, he is always saying yes when I ask for help, no matter when or what. Dear family, I am very fortunate to have you to share any kind of moments during our daily life.



Introduction

My thesis is based upon a series of five published studies attached at the end. I will first give you a very short nontechnical introduction to the methodology of the studies followed by a broader background in order to increase the understanding of what I sought to achieve and to give an overview of textile conservation in Sweden, and of some of the problems conservators deal with. My thesis evaluates three remedial conservation methods commonly used on historic costumes, especially the strength of the conservation expressed as maximum force at break. However, historic costumes cannot be used in a study that has destructive effects on the material. It was necessary to produce surrogates for the historic silk as a testing material. For that reason methods for artificial ageing of silk were experimentally studied. To set the foundation of my research a pilot study, Paper I, included two sections with the aim to map circumstances in costume conservation and to find suitable evaluation methods for remedial conservation. A questionnaire was sent to textile conservators in Europe and North America mapping some background information, and most important was to establish facts concerning which conservation methods they use, and which criteria they find important to regard a conservation as successful. To find suitable evaluation methods for textile strength I had to evaluate a set of mechanical tests of aged standard fabrics. The result from the pilot study was followed up and developed in further experiments, Paper I and Paper II, with the aim to produce surrogates for the historic silk as a testing material. Two seventeenth century silk costumes from the Royal Armoury's collections served as examples when setting a degradation level for the silk surrogates in standard silk (ISO 105-F06:2000). Standard silk was exposed to a series of extreme environmental conditions such as high temperature or high humidity to find an ageing method that made modern silk similar to historic silk. Both the historic and the artificially aged silk were analysed with mechanical and chemical methods to establish the degree of similarity with historic silk that the ageing methods could produce. High temperature gave the most similar results both in mechanical and chemical respects. In the next study, Paper IV, a set of silk samples was aged in high temperature. The surrogates were either cut with a scalpel to achieve a tear or mechanically abraded to achieve a worn damage. The damaged surrogates were conserved with three different stitching methods and submitted to experimental interventions, such as accelerated wear and removal of all stitches. Tensile strength was tested after every treatment, for instance after accelerated wear. The film "Stitched support methods using laid couching, brick couching and crepeline attached by running stitches" is available. It explains how the methods were carried out for the study in Paper IV (YouTube-film Nilsson), Appendix 2.

In a following study, Paper V, Swedish textile conservators were engaged in an experiment to establish the factors that determine aesthetic quality. The three stitching methods were represented on photographs of conserved costumes from the Royal Armoury's and the Hallwyl Museum's collections. However, no comparison between the three stitching methods was

performed in this study that concerned the basic question of what is meant by the aesthetic quality of textile conservations. More thorough information of the experimental studies is given in Papers I-V.

Now to the background of the studies. The Royal Armoury in Stockholm, where I have been working as a textile conservator since 1990, has a unique collection of royal costumes, both in quality and quantity, approximately 7,000 individual pieces. A majority of them are in delicate silk, often in combination with metal threads. Furthermore, which makes the costume collection exceptional, there is a thorough inventory made by the keepers through the centuries. There are notes made about the garment, its material, techniques, and colours. Sometimes one can even find whether the garment has been used at a special occasion, from where the material was ordered, and the price. The collections at the Dresden Rüstkammer and at the Rosenborg in Copenhagen also have this thorough documentation (Rangström, 2003).

The Royal Armoury has had its conservation studio since 1890 and from 1907 there are records of conservation treatments in the form of conservation reports in the archive of the Royal Armoury's conservation studio in Stockholm. In 1978 Skokloster Castle and the Hallwyl Museum were merged, and together with the Royal Armoury they were formed into a government authority. Many of the costumes are in great need of conservation even though they have been treated several times with different methods during the years. These recurrent treatments and the variety of interventions that have been used were an important reason for studies on conservation methods for historic costumes.

The conservator's task is to protect the museums' items from further degradation and, if possible, to make them available for exhibition. This task involves being able to control the climate, choosing storage materials, carrying out regular pest control, cleaning, and providing fragile materials with a support by conservation treatment and appropriate mounting when necessary. Correct decisions in the choice of treatment are expected in line with ethical considerations. According to the International Council of Museums' Code of Ethics (ICOM 2013, 2.24):

"The principal goal should be the stabilisation of the object or specimen. All conservation procedures should be documented and as reversible as possible, and all alterations should be clearly distinguishable from the original object or specimen."

From maintenance to textile conservation

Modern textile conservation has its roots in the nineteenth century, when simple care and repair turned into a vocation. Before that time, many textiles continued to be used in daily life, with little or no concern at all for their historical preservation. Gradually, techniques for preserving textiles were developed, based on ideas that some textiles are unique, culturally and historically interesting, and because of this require special handling and treatment. The art of textile conservation probably has its origins in the everyday mending and maintenance of textiles and the beginning of today's general conservation philosophy has been attributed to a change in attitude.

Muñoz Viñas (2005) suggests that this happened at the turn of the twentieth century and describes it like this:

"Conservation began when it became clear that the views, approaches, and skills required to treat a painting were different from those required to treat the walls of a common peasant house; or when it was apparent that cleaning a Neolithic axe required a different attitude and knowledge from that needed to clean a household lamp." (p. 2)

According to Brooks (2000) there is a long tradition of caring for highly valued textile collections, and conservation of historic and artistic works emerged in the 1920s. Brooks also states that textile conservation as a professional activity is a young field that had its founding at the textile conservation conference organised by the International Institute for Conservation of Artistic and Historic Works (IIC) in Delft in 1964. The council of the IIC found that a book about textile conservation was needed and gave Jentina Leene the commission to edit the first book on this topic.

The development of the textile conservator profession in Sweden and the Royal Armoury in Stockholm

The conservation studio of the Royal Armoury has its origin in the Royal Wardrobe from the 16th century. The keeper was responsible for the costumes and this is an early example of the care and maintenance of highly valued textiles. King Gustav II Adolf initiated the collections that later became the museum of the Royal Armoury. He demanded in 1628 that his costumes from two important occasions should be saved as a perpetual memorial (Sundin & Tegenborg-Falkdalen, 2003). In 1851 the costumes were transferred from the Royal Wardrobe to the Swedish Royal Armoury and became the responsibility of the Master of the Royal Armoury who probably had help from people familiar with textile handicraft.

In 1892 Carl Anton Ossbahr (1859–1925) was promoted to deputy, and finally, in 1896, to superintendent. Ossbahr initiated the permanent exhibition of the Royal Armoury in the Royal Palace in Stockholm, and is considered the father of collections management in Sweden (Brunskog & Nilsson, 2013). With the employment of Ossbahr, the first steps towards today's conservation treatments started at the museum. The fabric he used to preserve the collection of banners was not very successful, however, as the net being used as a support for the fragile fabrics was too coarse. After some time the net broke down and transformed the fabric into small pieces, the size of postage stamps. This net was also used in Switzerland (Flury-Lemberg, 1988).

With Baron Rudolf Cederström (1876–1944), who was appointed amanuensis for the Royal Armoury in 1899 and became its superintendent in 1904, the methods and profession developed further. The museum's activities expanded and improved under the directorship of Cederström, and he was later acknowledged as a leading museum technician. It has been claimed that Ossbahr served as Cederström's mentor and encouraged him to take a scientific approach to conservation. Cederström was aided and inspired by exchanges of information with European colleagues including Rathgen in Berlin. He visited almost every important armoury and textile collection in Europe at the time. As a result, he brought back with him new material and restoration techniques that he introduced and tested, and he urged that the results of the methods should be demonstrated to be safe prior to implementation (Brunskog & Nilsson, 2013). In

1925, Cederström found and started to use the semi-transparent silk fabric known as crepeline. In Sweden during this period, the method was known as the Cederström method. Later on during Cederström's active period, several textile assistants were employed and the conservation department slowly started to grow. The association Pietas was established in Stockholm 1908 by Agnes Branting (1862-1930) and Cederström was a member of the board. The mission of Pietas was to supervise the conservation of ecclesiastical and other historic textiles to secure the professional level of the conservation work while assistants carried out the practical work. Pietas was led by Branting from 1912-1930 and thereafter by her niece Agnes Geijer (1898-1989) until 1949. Geijer became a prominent figure in the field with many international contacts (Lundwall, 2000).

In Sweden, as in other European countries, the conservators reported to the curators who were responsible for the objects; they decided how the objects should be treated and wrote the conservation reports. The first curator Gudrun Ekstrand wrote a book chapter "Historic Costumes" about the conservation at the Royal Armoury in Stockholm; but, in fact, only dealt with the costumes in theoretical manner (Ekstrand, 1972). In Sweden, this situation led to trade unions encouraging the conservators in Stockholm to establish an association in order to achieve influence and status. A survey was made in 1964 to map the education level of the textile conservators in Stockholm (SFT 2012). The survey indicated that many of the conservators had studied art, design, and art history and many had craft skills from education and training in

different handicrafts. This was the starting point for the conservators to unite, and the Swedish Association for Textile Conservation (SFT) was established in 1967 with members from all over Sweden. The association aims at: a high textile professional standard, further education, developing conservation methods and to see that the knowledge and experience of conservators are efficiently used in work. At SFT-meetings and workshops experiences are shared, learnt and passed on amongst the conservators (SFT website, 2015). The title "conservator" at the Royal Armoury's conservation studio was used for the first time in 1973 when Eva Möller was employed (Nilsson & Enhörning, 2003). During her first year as conservator, she was given a one month study journey through Europe to visit some of the leading conservation studios and met people including Judith Hofenk de Graaff, Ágnes Tímar-Balázsy and Karen Finch. The meeting with Karen Finch who was founder of the Textile Conservation Centre in the U.K. inspired Eva Möller to contact the Ministry of Education in 1976 to point out the need of an education in conservation and the need for more positions for conservators in Sweden (SFT, 2012). The academic conservation programme at the Department of Conservation at the University of Gothenburg started in 1985. The knowledge about textile conservation, built largely from experiences of which methods and materials are appropriate to be used, has been widely shared and transferred between conservators in the SFT-meetings and through the teaching of students at the University of Gothenburg. Textile conservation in Sweden has also been influenced by the conservation culture of other countries. as some conservators are educated at the Textile Conservation Centre in England or Abegg-Stiftung in Bern and several conservation students at the University of Gothenburg have undertaken internships in countries such as Canada, England, France, and Switzerland. Since many of the conservators at the Royal Armoury's studio through the years have taken an active part in the conservation community, both nationally and internationally, the studio has followed the development of the profession and is thereby open to test new material and methods.

The literature as a common platform

An important common platform for the conservators in many countries in Europe (and beyond) is the literature that has influenced generations of textile conservators and has spread the knowledge of textile conservation by way of conference pre- and post-prints, articles in journals, and books. Of significant importance for textile conservators from 1970 onwards is a handful of books such as Textile Conservation edited by Leene (1972), Caring for Textiles by Finch and Putnam, (1977), Textile Conservator's Manual by Landi, (1985) with a second edition in 1992, and Textile Conservation and Research by Flury-Lemberg, (1988). With Chemical Principals in Textile Conservation by Tímar-Balázsy and Eastop, (1998) a further step of development in textile conservation was taken as it deals with the science of textiles; this is also dealt with in Leene's book, but not in such depth. The chapter "Restoration and conservation" by Lodewijks and Leene (1972) ends with the reservation that the chapter is only a guide to the methods most commonly used in the conservation of textiles. Also, Finch and Putnam (1977) underline that their book is a practical guide for those responsible for old textiles and can be used for certain areas, but is not meant to be a handbook in conservation interventions as this type of work demands a qualification. Landi's book (1985) on the other hand, is written for textile conservators and students in textile conservation; it soon became established in many countries as the main literature in textile conservation. It is much more detailed than the earlier books, and the second edition (1992) is an updated version with reflections over earlier case studies that have been revisited. Flury-Lemberg (1988) does not cover as much that concerns practical conservation as Landi; rather, it covers more textile history than textile and conservation technology.

More recent publications specialising in textile conservation are Tapestry Conservation Principles and Practice by Lennard and Hayward (eds), (2006), Textile Conservation Advances in Practice by Lennard and Ewer (eds), (2010a) and Changing Views of Textile Conservation by Brooks and Eastop (eds) (2011). In Lennard and Ewer (2010a), one can follow all steps in tapestry conservation but from different perspectives, as the authors represent slightly different approaches and experiences. The book is built up of three parts: changing context, treatment options and the future, supplemented with chapters by authors sharing their experiences from the last 25 years. Lennard and Ewer claim that the textile conservator's understanding of object significance has changed and that the preservation of the object's own history has become a more important aspect of conservation. Earlier, conservators were keener to reverse changes to an object in an attempt to return it to its original state. In efforts to document and understand an object's history, the development of instrumental analysis is of great importance. Lennard and Ewer state that during the 1980-90s new technical solutions were found and the focus was on *how* and not *why* an object was conserved. Again focus changed during the late 1990s and the object's context became more important.

Lennard and Ewer (2010b) identify four different areas they find important for collaboration between scientists and conservators: (1) materials analysis of the object, (2) mechanics of degradation including the effects of the environment, (3) analysis of new materials for their preservation considerations and as treatments aids and (4) analysis that informs the treatment processes. (p. 230).

The authors conclude that conservation treatments have not changed fundamentally during the two last decades, but that the development of techniques and conservation material has progressed significantly, as has our understanding of how treatments work. Lennard and Ewer (2010b) find that it is important to evaluate previous treatments. When evaluating previous treatments, one possible problem is that the condition of the object prior to treatment was not investigated and documented thoroughly. Moreover, many conservation reports describe the interventions and material used in vague terms. Possibly science and increasing collaboration with conservation scientists have opened the way for new solutions and knowledge. There is still much to explore in this subject, as the variations are almost endless. Textiles are made with different materials using different techniques and often combined with other non-textile materials (e.g. metal). Their condition and damage vary. The list can be long. In addition, new material will be attached as a support, which in turn has a great variety of more or less unknown properties, and to attach this material a technique

has to be chosen. It is important to understand the effect of the chosen technique. The book by Brooks and Eastop (2011) describes textile conservation from a retrospective, contemporary, and future perspective. It contains a selection of important articles from 1956 to 2008 of which some were published in English for the first time and thereby available to a broader public. The new challenges and changes the authors point out for the profession are: the social context, technical development, new materials, contemporary art, and philosophical questions. What the majority of the above-mentioned books have in common is that they all contain a wide range of experience in the field of textile conservation as they enable so many textile conservators to be heard.

There are three books about textile conservation published in Sweden during the two last decades. SFT has published two. Textilskatter i svenska museer [Textile Treasures in Swedish Museums, translation by Anna Stow], (2000) in which textile conservators describe the development of textile conservation in Sweden as well as their own experiences from case studies. The second, Textilkonservering: att vårda ett kulturarv [Textile Conservation: To Care for Cultural Heritage, translation by Anna Stow], (2012), also written by textile conservators, reflects the role of the textile conservator and describes preventive conservation, conservation, exhibition, working with church textiles, mounting textiles, and health risks. The third book is written by Eva Lundwall, (2003), Den ljusskygga textilkonsten: textilkonservering under 1900-talet [The Light Shy Textile Art: Textile Conservation During the 1900s, translation by Anna Stow] is a history of textile conservation at Pietas (which later was incorporated into the National Heritage Board) and the National Heritage Board's conservation studio, and describes their methods and the materials used during the period 1908-1999.

Decision making

At a certain stage a decision is taken whether an object is to be conserved or not. The opinion on the pros and cons of remedial conservation has varied over time. Some have advocated caution and preventive conservation (Flury-Lemberg, 1988); while others have claimed that interventions are necessary if we want to preserve the objects (Landi, 1992; Ashley-Smith, 1994; Appelbaum, 2007). An important task for museums is to exhibit and preserve their collections and the conservator's responsibility is to make this possible. To me both preventive conservation and remedial conservation are essential in this mission. If we were to neglect remedial interventions many textiles would lose their meaning. But it is important that the interventions used in remedial conservation fulfil the needs, and this thesis is a contribution to our knowledge how three common stitching methods affect a degraded silk that is treated.

According to Jedrzejewska (2011) there are two main reasons why conservation should be carried out: to prohibit further deterioration of the object's substance, and to make it look satisfactory. She presents a long record of ethical principles and concludes that the main ethical principle in the choice of treatment and the care of the object, is the respect for the object's authenticity of evidence. According to Lennard and Ewer (2010b) there has been a change in the museum conservator's tasks in the US and UK in the last decades. Remedial interventions have taken a step back in favour of preventive conservation due to cost cutting. The museum

conservators are more involved in overall collections care and preparing exhibitions instead of performing extensive remedial interventions on single objects. The situation is similar in Sweden. Nevertheless, objects are still subjected to remedial conservation. In the decision making for treatments, several conservators maintain that every object presents its own unique conservation problems, that the choice of treatment is dependent on the object's condition and intended future, and that there are many questions to be answered before a decision is taken (Finch & Putnam, 1977; Flury-Lemberg, 1988; Landi, 1992; Caple, 2000; Appelbaum, 2007). Finch and Putnam (1977) state that "...rules for treatment are almost impossible to formulate beyond agreeing that most old textiles will require cleaning, many will also need some support and, if they are to be displayed, will need to be made as attractive as possible." (p. 69). Eastop (2011) claims that it is not a textile object's condition that determines the conservation treatment; it is its role and context. Muñoz Viñas (2005) claims that conservation of objects is made for those people for whom they are important and therefore important to preserve, and that their desire must be considered in the decision making of how to treat an object. The opinion of Lodewijks and Leene (1972) is that the importance of the object affects the choice of method and that time consuming methods should be saved for the more valuable objects is possibly not something that contemporary conservators agree with. But there is an awareness and a discussion about efficiency with the time aspect as an important factor in the choice of methods (Caple, 2000; Appelbaum, 2007; Lennard & Ewer, 2010a; Marçal, Macedo & Duarte, 2014). The Com-

monwealth of Australia has on behalf of Heritage Collections Council of Australia published a guide to be used when assessing the significance of cultural heritage collections (Russel & Winkworth, 2010). MacLeod and Car (2014) use this guide in their work to make a treatment priority score considering the significance of textile collections and every textile's need of conservation. MacLeod and Car also considered whether a volunteer could carry out the work or if it must be done by a conservator. By using this information in a mathematical model they produced a figure of the estimated costs, and treatment of the objects could be prioritized. Ashley-Smith (1994) raised the question about what he saw as an increasing aversion against intervention. He advocates the importance of the intervention for the object's aesthetic appearance as well as the preserving effect, and he states that we should better know the long-term outcome. Appelbaum (2007) also argues for the necessity of conservation intervention, as preventive conservation alone is not enough and that it is necessary to know the consequence of methods. An object's expected future and its on-going deterioration rate must be considered as well as its appearance. Flury-Lemberg (1988) opposes this attitude stating that it sometimes is better to do nothing, and wait for the development of new techniques. While according to Landi (1992), this attitude only postpones problems.

A first decision to be made is if an object should be conserved or not. If yes, the next decision is how it should be treated. In the questionnaire from 2005 (Nilsson, 2005) textile conservators stated that the most common reason for conservation was an approaching exhibition. The decision of how to conserve the objects was most

often made mainly by the conservator alone, less often as a joint decision by conservator and a senior curator. The topic of decision making has been studied by several authors, and methods have been developed for the decision making process (Caple, 2000; Appelbaum, 2007). Caple (2000) advocates the decision tree method in which expected outcomes depending on method and associated costs can be calculated, and also that it can be a part of risk assessment. Appelbaum (2007) has developed a thorough eightstep system for how to plan and implement a conservation treatment. The first four steps are necessary as foundation for the more technical decisions. Her method starts by characterizing the object, followed by reconstructing its history, determining its ideal state and deciding on a realistic goal of the treatment. Thereafter, the conservator shall choose treatment method and conservation material, document the object, carry out the treatment, and document again after treatment. She states that at the end of the treatment the conservator has to judge the result from the custodian's point of view, according to the first impression of the object before the treatment was started. Otherwise there is a risk that the conservator does not see the object as a whole, and focuses on the damage only, which might not dominate the impression of the object any longer.

How willing is the conservator community to reflect over available methods, and maybe reject familiar methods in favour of others? Appelbaum (2007) states that many conservators are eager to get knowledge of which type of conservation method works best; but, she is doubtful about their willingness to change their methods, if the alternative departs from the conservator's

own experiences. This is in line with Marçal, Macedo, and Duarte (2014) who discuss the "anchoring effect" —that a conservator continues to use a known and habitual method despite new, better methods being available. The authors have studied the decision making of conservators from a psychological perspective and claim that the conservator's interaction with an object provides an individual experience that influences future decisions. They state that people preferably choose to make the easiest decision, and with increasing complexity in the choices, there is a risk that no decision or action is taken. I am inclined to agree with this. My own experience is that there are several aspects involved when a decision of how to make a conservation intervention is taken and the willingness to adapt new methods, the time aspect is the crucial point. It is necessary to practice before proceeding to the "real object" when adapting a new method. That requires time and if the conditions are good with generous time limits I believe most conservators would take the opportunity to at least test new methods. If there is a time limit, which most often is the case, the choice of method and material must be affected. It is quicker to work with a familiar method and to use a material that is already available. But there are also high demands on the quality, the aesthetic as well as the preservation, that a conservator always should strive at.

Silk

Silk can be produced from wild moths as *Antheraea pernyi* and *Antheraea mylitta* or cultivated moths as *Bombyx mori* (Shoeser, 2007), which is the silk, explored in this thesis. Many of the royal costumes in the Royal Armoury in Stockholm were made for special occasions such as



Figure 1. Gustav II Adolf's coronation costume, inv no Lrk 25605-6. Courtesy Göran Schmidt.

coronations and weddings and had by necessity aimed to show the royals in splendid situations. Several costumes are made of white silk from Bombyx mori. Silk is and has always been a precious material with special properties that are due to its chemical structure. It is regarded as a fibre with fairly high strength and breaking extension (Morton & Hearle, 2008). It is a highly oriented and crystalline proteinaceous fibre with amorphous regions. It consists of two 7-12 µm wide filaments that the larva produces to make its cocoon. Consisting mainly of fibroin, the filaments are stuck together with sericin as a glue (Tímár-Balázsy & Eastop, 1998). Fibroin is not a single protein as it consists of two main components called H-fibroin, which is large and the dominating protein in the silk fibre, and Lfibroin, which is considered small (Shimura, Kikuchi, Ohtomo, Katagata & Hyodo, 1976). The molecular weight of H-fibroin is about 350,000 and that of L-fibroin about 25,000 (Takahashi, 1993). A third component is P25 (Coublé, Moine, Garel & Prudhomme, 1983). P25 is a glycoprotein, which is present in a much smaller proportion than L- and H-fibroin (Inoue, et al. 2000). The gene sequence is known for these three and they are present in single copies per genome, and each generates a single polypeptide (Shoeser, 2007).

The very smooth surface of the fibre makes silk quite resistant to dirt which does not deposit so easily and is one explanation to why the white silk costumes in the Royal Armoury after almost 400 years still are quite white and clean. Following stretching, the polymers in the silk fibres do not go back to their former state as the stretching ruptures hydrogen bonds. This phenomenon can be seen as creases in a silk fabric. The handle of silk is



Figure 2. Karl X Gustav's coronation costume, inv no Lrk 25597-9. Courtesy Göran Schmidt.

described as medium due to its crystalline structure that gives it certain stiffness (Gohl & Vilensky, 1983). There are several reasons why silk deteriorates. In the case of costumes one of the first things that likely will happen to cause a physical defect is when the costume is being worn. Many of the Royal Armoury's ceremonial costumes, such as Gustav II Adolf's and Karl X Gustav's coronation costumes have only been worn once at a special occasion, figures 1 and 2. Therefore one can conclude that costumes like those have had their physical damages later on during occasions such as noble men taking the opportunity to try them on while visiting the court (Miranda, 1950; Reenstierna, 1946-1953), souvenir theft or when the costumes were used as masquerade costumes at court festivals. But damage and wear has certainly also occurred since the costumes have become museum objects, for example during exhibitions with damage caused by tension from inappropriate mounting or colour fading due to the light. Damage can also occur because of a textile's construction. A delicate silk fabric folded over coarse linen will by time break, figure 3. In addition to degradation by physical impact silk is degraded by other factors such as oxidation which the two amino acids tyrosine and threonine are especially sensitive to. Photo oxidation through daylight and more often ultraviolet irradiation is reported to be harmful to silk as is hydrolysis under extreme humidity conditions as well as high temperatures and extreme pH (Yanagi, Kondo & Hirabayashi, 2000; Luxford, Thicket & Wyeth, 2010; Vilaplana, Nilsson, V. P. Sommer & Karlsson, 2015).

Through the Silk Road trade route silk spread beyond China and during the 12th century, a silk industry was established in Italy and



Figure 3. A damage on the shoulder wing of Gustav II Adolf's doublet, inv no Lrk 25605. Courtesy Matti Östling.

France and later also in Germany and England (Shelagh, 2004). For the analyses in this thesis, samples of both satin and tabby silk weave were taken as references from Gustav II Adolf's coronation costume from 1617 and Karl X Gustav's coronation cloak from 1654. Unfortunately, there are no records listing from where the fabrics originate. It is presumed that many of the fabrics have their origin in Italy, and that many of the costumes were ordered from France (Cyrus-Zetterström, 2009). Fabrics for the royal wardrobe were bought through merchants at the Stockholm market. Many of the deliveries of fabrics seem to come via merchants from Northern Germany and the Netherlands, but they were probably middlemen. Examples of terms used for fabrics that were handled at the royal wardrobe include "genueseratlask" and may be interpreted as indicating that the origin of the fabric is from Northern Italy (Aneer, 2009). Genueser could stand for Genua and atlask for atlas or satin. The name atlas is said to come from the mountain Atlas (Morocco, Algeria and Tunisia) that has a magic lustre in a certain light (Ekenberg & Landin, 1894). A reason why the silk fabric can be so shiny is that the silk strands in older costumes have a negligible twist. Silk can be tightly packed in a weave, and if the yarn only has a slight twist the fibres will spread out and give a shiny lustre. This is enhanced by the silk filament's triangular cross-section that reflects the light. This is the reason why the fabric in both Gustav II Adolf's and Karl X Gustav's coronation costumes have an extremely smooth and shiny surface, which is enhanced by the satin weaving technique. When handling and touching the costumes the thick satin in the outer fabric feels quite robust even though the satin in Gustav II Adolf's doublet has worn and damaged areas. While the lining in tabby in his doublet, which is much thinner than the satin, feels and looks fragile.

Interventions

When studying the textile conservation reports at the Royal Armoury's conservation studio it is obvious that the categories of objects being treated have varied over time, as has the number of objects. There is also a clear development of the reports themselves; the early ones being quite brief, and from the 1960s and onwards being more detailed and accompanied with photographs.

From 1884 and onwards, much effort was put into pest management, using pesticides regularly and by airing all textiles every spring (Nilsson & Enhörning, 2003). Despite these efforts the collections in wool suffered from insect damage and, therefore, many hours of conservation were spent on woollen objects in the early years of the conservation studio. But also objects such as banners and costumes bearing witness to Sweden's great period were treated. In the 1950s different types of horse equipment were most common for treatment. During the 1960s costume conservation was at its peak and almost the only category to be treated in 2000, figure 4.

Three types of interventions have been particularly common to use on costumes in the conservation studio of the Royal Armoury: laid couching, brick couching, and a cover of crepeline. There are a few exceptions where adhesive treatment and stitching have been used. When using laid couching the fragile textile is laid on top of a support fabric, placed aligned with the fabric grain, and then the damage is secured onto the support fabric. Laid couching is a long straight

stitch, laid in line with either the warp or weft of a fabric and then fastened in place with short perpendicular holding stitches, inserted at regular intervals. Brick couching is carried out in the same way as laid couching, it is the stitching technique that differs. The stitch in brick couching is a short stitch laid perpendicularly over one or several warp or weft threads sewn to form a regular pattern like brick-work. The crepeline method protects a damaged and frayed area by using a covering layer of silk crepeline (a light organdie) that is attached to the fabric with running stitches along the edges of the crepeline. Usually, when used on costumes, the shape of the crepeline is adapted to be able to fix it along its edges at existing seams or lines in the fabric. When a piece of crepeline covers a larger area it is common to further attach it with spaced rows of running stitches to keep it close to the underlying fabric to avoid bagging or wrinkling in the crepeline. There are variations in the description and naming of the couching stitches in the English culture, and the Swedish language has no expression for brick couching. In some of the conservation reports from the Royal Armoury's conservation studio the word sinka is used for laid couching, generally läggsöm in Swedish.

An example of conservation during the early 1960s is the court dress of Lovisa Ulrika from 1751 (inv no Lrk 31247-9), which was quite radical. The skirt was washed and worked over with sponges and thereafter partially treated with chalk, washing detergent, and ammonia. 165 hours were spent on the skirt alone. Also many of Gustav III's costumes were treated during this period. One example is his wedding costume from 1766 (inv no Lrk 31255-7), in which the weave consists of silk and flat silver strips.

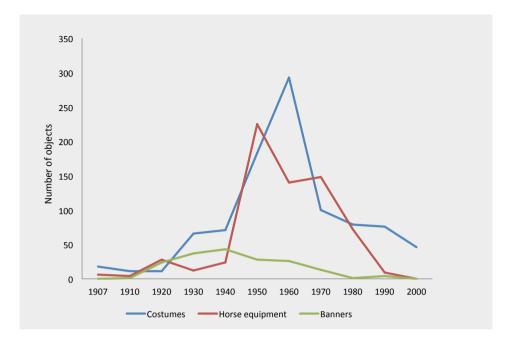


Figure 4. The most common type of textile objects to be conserved in the Royal Armoury's studio can be divided into three main groups: costumes including accessories and shoes, horse equipment including textiles for carriages, and banners.

Figure 5. Karl X Gustav's silk breeches, inv no Lrk 19322. Courtesy Tove Nilsson.

Figure 6. Detail of the conserved right corner of the right leg of Karl X Gustav's silk breeches, inv no Lrk 19322. In the upper half it is brick couching and in the lower part it is laid couching. Courtesy Tove Nilsson.

The loose silver strips in the weft were stitched down two by two as part of the conservation, using brick couching. The coat alone required 990 hours.

Material choice has not always been successful over the years. Coarse net has been mentioned earlier, but tulle or tarlatan (a thin cellulose fabric) was also used on banners and on costumes such as King Gustav II Adolf's doublet from 1627 (inv no Lrk 31193), which had its fragmentary outer fabric in silk completely covered in tulle in 1911. After Cederström's introduction of crepeline, the coarse materials were replaced, but banners were often given a full support onto which the banner was stitched and thereafter given a cover of crepeline. Since Cederström's period, most of the conservation work in the studio is made in preparation for exhibitions and loans. During the years before the reopen-

ing of the Royal Armoury in the Royal Castle in Stockholm in 1978, a comprehensive conservation work started to prepare the objects for the new exhibition. A large number of the objects to be displayed were costumes, and the methods used were often one of the three most common ones. The above mentioned doublet of Gustav II Adolf is an exception in the choice of method. It was re-conserved and the tulle was removed along with the original outer silk fabric, except where it was hidden under the braids. The original silk was put into storage in envelopes and substituted with a new silk satin. A buff-jerkin of Gustav II Adolf from 1627 (inv no Lrk 31194) had sleeves in fragmentary silk that were instead kept and sewn onto crepeline. The domino of Gustav III's masquerade costume, the costume he wore at the ball when he was mortally wounded 1792, is a reconstruction as the original was judged to be





too fragile to be exhibited. The costume of Karl X Gustav from 1650 (inv no Lrk 19321-2), earlier conserved in 1948, was re-conserved in 1985 with three stitching methods. The seams of the breeches were almost totally unstitched. A full support was attached under the breeches front pieces leaving the old patches from the earlier conservation untouched. The loose weave of the front pieces was stitched with both laid couching and brick couching, figures 5 and 6. Edges of the pockets were covered with crepeline and some of the ribbons were treated using adhesive with crepeline as support. During the early 1990s, the radical methods were discussed and questioned at the conservation studio, and crepeline came to be more frequently used to avoid extensive stitching and opening of seams. Gustav III's breeches from 1777 (inv no Lrk 21447), with a bleaching damage on the left leg, was partially covered with crepeline that was attached with running stitches. Another example of a crepeline conservation, made late in the 1990s, is a bodice from 1907 (inv no Hwy XXII:I:A.09) made of weighted silk having the typical damage of tears all over the surface almost impossible to stitch into, figure 7. Degraded weighted silk is often recognized by sharp cracks or slits usually following the line of the warp and in extreme cases recognized by total fragmentation. Landi (1992) states that tin-weighted silk can only be supported by the adhesive method followed by complete immobility. The bodice described above, from the Hallwyl Museum, seems nevertheless an example where adhesive treatment was not necessary. It has successfully been supported with a cover of silk crepeline attached by rows of stitches to keep the crepeline tight to the surface and keep the fragile silk in place.



Figure 7. The bodice to a dress conserved using a cover of crepeline, inv no Hwy XXII:I:A.09. Courtesy Tove Nilsson.

Landi's (1992) recommendation in the choice of method is most often to use a support fabric inserted under a frayed area that is then couched down, preferably made without unstitching seams; but, if that is not possible, exceptions may be made or the frayed area could be covered with a new fabric or crepeline. Her recommendations vary depending on which piece in a costume she describes, but support fabric with couching is the overall theme. Nylon net is often mentioned in contemporary publications as a material (Gill, 2010; Lennard & Ewer, 2010c), but this is a material that has not been used in Sweden until just recently being then introduced by interns and colleagues from abroad. Maybe the reason for this is the earlier bad experiences of tulle and other coarse net resulting in damages and impressions (Brunskog & Nilsson, 2013) and the negative reports by Lodewijks and Leene (1972).

Needle techniques

In the textile conservation literature on case studies and in conservation records, the needle techniques used are rarely described in detail. Often the interventions are described as "... supported by stitching..." (Gentle, 2010 p. 64) or "...the bad areas were reinforced with couching..." (Landi, 1988 p. 272). Information is lacking concerning the type of stitch, the spacing or the length of the stitch, and there is very little guidance of how to execute the stitching. Lennard and Ewer (2010b) point out that stitching techniques, so far, have not been the subject of many studies. The lack of research and evaluation is probably due to the fact that stitching still is regarded as something natural in combination with textiles, while adhesive treatments do not have the same tradition as stitching and have been more scrutinised because they are

less familiar. Lodewijks and Leene (1972) state that "...sewing methods are more natural to a textile" (p. 151) and should be chosen before an adhesive method. Landi (1992) has the opinion that the most important result using needle technique is that the textile's special property of drape still remains; it is important, however, not to stitch too tight or with too small stitches, as the stitches may cut into the textile and cause damage. Finch and Putnam (1977) also highlight that the tension of the stitching is of importance. Flury-Lemberg (1988) advocates laid couching, she states that it requires few stitches. Lodewijks and Leene (1972) suggest that different techniques can be used while mending with stitches and that determining which one to choose is a matter of the form, condition, and historical and aesthetic value of the textile. Several authors agree that the conservator's skills are crucial. The conservator should be a master of needle techniques, have a sense of colour, and have an overall artistic feeling to maintain the balance of the work of art (Lodewijks & Leene, 1972; Landi, 1992).

There are recommendations and descriptions on what type of stitches to use in textile conservation and in specific situations (Landi, 1992; Winslow Grimm, 2002; Gill, 2006). Couching, running stitches and tacking are the most commonly mentioned stitches. Couching is highly recommended to use on worn areas with loose warp or weft and is stitched either as brick couching or as laid thread couching. Landi (1992) states that laid couching "...gives the flattest form of join when piecing in new fabric or rejoining an object which has been cut through." (p. 117). She also states when describing how to carry

out the stitching that: "The lines should always extend into the stronger fabric surrounding the area under repair. Spacing should be varied according to both structural needs and aesthetics, the holding stitches being staggered to form a brick pattern." (p. 117). This is a recommendation that is well established amongst the professionals.

Conservation material

Thread

The choice of conservation material is complicated as there are many aspects to take into consideration. Flury-Lemberg (1988) claims that reeled silk is the best material to use for stitching. Silk is easy to dye, it adapts best to each fabric, which makes the stitches virtually invisible and it will break before it damages the fabric. Some recommendations are not very specific, but state that the thread should suit the object's texture and be fine enough not to make holes in the conserved fabric, and that the colour match is important (Lodewijks & Leene, 1972; Landi, 1992). Several authors state that the recommendation has been to use like with like both in the choice of support fabric and of consolidating threads with which to stitch (Landi, 1992; Lennard & Ewer, 2010b). In Landi's study of threads of natural fibres versus synthetic fibres she concludes that the conservation material to be used should resist stress longer than the object to be conserved and that it might be an idea to develop the optimal material ourselves (1988). Still in 1992, Landi is uncertain about what is best as she claims that synthetic fibres are more resistant to environmental factors than natural fibres and it is not clear what to recommend. She states that it is a question of the tension of the thread and not its tensile strength, and concludes that the governing factor is that the material is compatible with the object (Landi, 1992). Ellis (1997) studied the effect of dyeing and artificial ageing on the mechanical properties of a variety of silk and polyester threads from a fabric with trade names as Tetex or Stabiltex, commonly used in textile conservation. It was found that polyester thread, which sometimes is feared to be too strong, had a similar tensile strength as fine hair silk. A more recent survey investigates threads that are commonly used in the consolidation of weak areas in textiles and studies how threads in natural fibres or in the synthetic fibres affect historic textiles in natural fibres (Benson, Lennard & Smith, 2014). They obtained no clear difference between natural and synthetic threads, while the thread's fibre constitution, being filament or staple, affected the result, as did the stitching technique. A thread with staple structure recovered better after load than a thread with fine filament. The result is interesting, but as the amount of samples was limited this needs to be investigated further.

Support fabric

Flury-Lemberg (1988) states that the supporting fabric should correspond with the object, but also that the first requirement is that it should be as transparent as possible so that the object can be studied from both front and back. She mentions crepeline as being the gentlest treatment. Landi (1992) suggests fine net or silk crepeline as support for very delicate and fragile textiles, but she also claims that the crepeline is too weak to give sufficient support and not heavy enough to restore the former drape of the fragile textile. Lodewijks and Leene (1972) state that a textile

in a supple fabric supported by even a very thin fabric would harm the drape. Finch and Putnam (1977) propose that:

"The most effective support one can give an old and fragile textile is to back it with suitable material, and choosing the type of backing is of great importance. It should be compatible with the textile it will support and suitable in weight, weave, colour and type of fibre." (p. 70).

Effects of interventions

There are very few investigations of how different fabrics act as support material and how they affect the textile to be supported. One exception is Coulter's study (2007) that presents an investigation of how two types of fabrics with different stiffness (Young's modulus) used as patches affect a woven tapestry. With a patch of flax as a support, the location of the largest stresses has moved to the corners of the patch. With a stiffer support patch the stresses have decreased in general. This uneven distribution will almost not allow the weakened area to move while the tapestry is hanging, which may weaken the previously strong areas which the patch would now pull on.

Appelbaum (2007) has pointed to the need of such studies: Little data exist on the extent to which specific treatments protect against various loads, particularly dynamic ones, and there is no widespread agreement about an ethical standard for any particular level of protection. Conservators need to know more about *how* to protect objects, and they need to make decisions about the degree they *should* protect them. (p. 287).

Evaluation of conservation methods and their effect on degraded silk costumes is an unexplored field. Paper IV is a contribution to this

neglected area. More has been done in the field of tapestry conservation as in the British three year project "Monitoring of Damage to Historic Tapestries" 2007-2009 (Historic Royal Palaces website).

Theory

I started my career as a textile conservator in the middle of the 1980s, and after completing an undergraduate degree in conservation with a textile specialization I have worked with the textile collection at the Royal Armoury and the Hallwyl Museum, and mainly with costumes. When I started this employment we were seven textile conservators, and our manager encouraged us to share experiences and to discuss conservation treatments. As already mentioned, knowledge was built at society meetings, between generations of conservators and via conservation literature. This type of knowledge is called vernacular by Kaiser (2000) and he claims that vernacular knowledge is something that everyone has and uses daily but that this knowledge is limited to your own group, which, in my case, is the textile conservators. Today's conservation profession requires that one has several of the knowledge types proposed by Kaiser (2000), preferably all four of them: scientific expert knowledge, vernacular knowledge, silent knowledge, and practical proficiency. One of my colleagues in my early career was considered to be an expert in costume conservation. As a recent graduate conservator I was keen to partake of her experiences and knowledge, and was privileged to be able to learn from her. After having gained some more experience during the following ten years I reflected on the fact that many of the costumes had been conserved a number of times and that different methods often had been used. I wanted to know if any of the methods was preferable and to take a step further and combine my vernacular knowledge and practical proficiency with a more scientific knowledge. At this time I could be considered to be a "practitioner with a certain proficiency" according to the Dreyfus and Dreyfus (1986) model, that Kaiser (2000) relates. The model describes development of competence from novice to expert in five stages. The stages are: novice, advanced beginner, competence, proficiency and expertise (Dreyfus & Dreyfus, 1986). All through, experience is stressed in this list and the professional practitioner has by experience reached a level of unconscious knowledge, called intuition (Dreyfus & Dreyfus, 1986; Kaiser, 2000). The expert practitioner according to Kaiser has, thanks to all earlier experience, almost no need to consciously analyse, but instead according to the model the work is carried out automatically, the knowledge is in the body of the expert. However, within conservation where in principle every object is considered unique and the decision making is based on individual objects' needs and context you can never work automatically. An expert practitioner in costume conservation must have a considerable experience but also a scientific basis for consideration of conservation methods, properties, and effects.

According to White (1978) the contemporary historians could only be taken seriously if they were serious in asking the type of questions that contemporary art and science require them to ask of the material chosen to study. White asks why research questions should come from other disciplines, and how an area can develop if those working in the field do not pose the questions. Within conservation those with practical experience must be those best equipped to ask the questions that will lead to solutions regarding

conservation, and according to Lassen (2014) practice is an investigating tool important for the final results. The benefit of my research work has been questioned by one of my textile conservator colleagues, and by a conservator acting as a peer reviewer of an article. I received comments for example that my research questions are about common sense, and that I am researching topics that are already well known to those working with conservation. What colleagues might regard as common sense and obvious knowledge through tradition and therefore unnecessary to survey is to me characterized by lack of scientific knowledge. According to Kaiser (2000) common sense is a term used to describe what the majority of a group of people see as obvious based on assumptions about what is right and true. The weakness of common sense is that many believe something to be true, and it is not the basis for what may be true. Kaiser states that common sense is in strong contrast to the other knowledge types described above, and I agree with him. - My studies comprise both basic and applied research. For example Paper V is of the first type; it describes the attributes behind a conservation being perceived as aesthetically pleasing. The result may seem self-evident when reached, but until something is researched there is no scientific basis for a detailed answer.

It has been important for me to write a thesis based on my published articles in order to share my results and make them available for other conservators or researchers in the field. When possible they have been published with open access copyright.

The studies in this thesis are empirical and based on the tradition of natural science. Concerning studies in conservation, this tradition was founded in the first half of the twentieth century, starting in 1930 when a new *scientific conservation* school arose described as *hard* science by Muñoz Viñas (2005) even though several scientists, including Rathgen whom Cederström learnt from, worked with scientific conservation issues even earlier. This era of *hard* science came to flourish even more under the second half of the twentieth century, and according to Muñoz Viñas it became the supreme approach at that time. In conservation, issues related to the knowledge of the properties of materials and of the deterioration processes also came to be included in this *hard science*.

The issues that have arisen in my own work as textile conservator, and that I have investigated in Papers I-IV, relate very well to this approach and therefore it is natural for me to adopt *scientific conservation* and *hard* science as my approach. The work in Paper V, however, has a different, psychological approach, which deviates from scientific conservation described and summed up in four principles by Muñoz Viñas (2005) and might be considered as *soft* science as it is partly based on experience-based interviews. Muñoz Viñas (2005, p. 78) claims that there has been very little theoretical discussion about the basis for conservation science since the early 20th century.

Muñoz Viñas (2005) suggests however that there are strong underlying principles that he calls a "material theory of conservation" and they are summed up as follows:

I. Conservation should attempt to preserve or restore the true nature of objects. This is its most important principle, which is common to all classical theories of conservation.

- 2. An object's *true nature* relies mainly upon its material constituents (material fetishism).
- 3. The techniques and target state of the conservation process should be determined by scientific means. Conservation techniques should be developed, approved, selected, performed and monitored in accordance with scientific principles and methods, and particularly in accordance with those emanated from the hard, material sciences. Subjective impressions, tastes or preferences should be avoided; instead, decisions should be based upon objective facts and hard data.
- 4. Scientific conservation methods and techniques actually produce results that are objectively better than those provided by traditional, non-scientific techniques. (p. 90)

I have some comments. Interdisciplinary research is of great importance. If conservation scientists, scientists in other disciplines, and conservators cooperate in joint projects, methods can be developed which are down-to-earth and useful to conservators. If we (the conservators), for example, accept that a conservation treatment is ultimately made for the persons the object is important to, and if we want our work to be relevant, and accept an approach shared by many contemporaries as Muñoz Viñas, (2005), Appelbaum, (2007) and Ashley-Smith, (2009), the subjective impressions must be taken into account, in contrast to what is expressed in the third principle that subjective data should be avoided.

Evaluation of the issue of aesthetic quality that is presented in Paper V is one example of cooperation between two professionals, a psychologist and a conservator, who have studied qualitative data in

a systematic way that should satisfy common scientific demands. Both hard data and soft data can be treated in a scientific or non-scientific way; there is no reason to reject qualitative data a priori.

According to Richmond and Bracker (2009) the theory of conservation is a current topic and there are changes within the conservation theory away from the "scientific objective materials-based conservation to the recognition that conservation is a socially constructed activity..." (p. xv-xvi). They also state that many contemporary stakeholders have questioned the previously existing opinion that science-based conservation would ensure to be true and neutral. According to my opinion, however, conservation practice must have a ground of scientific studies of conservation methods that satisfy common scientific demands and are reliable. In this way what is "true and neutral" may in the long run be approached.



Five empirical studies

Aim

The general aim of this thesis is to evaluate remedial conservation used in common conservation treatments of fragile silk costumes. To reach the aim the decision was taken to:

- Map what textile conservators judge to be the most important criteria for a successful conservation.
- Map what the most common methods are when using needle techniques in costume conservation.
- Develop methods to artificially age silk to achieve chemical and mechanical properties similar to seventeenth century silk, and to produce a surrogate for naturally aged silk for experimental research.
- Investigate how the most common needle techniques affect the strength of conserved silk surrogates.
- Investigate how silk surrogates conserved by needle techniques are affected by wear.

Investigate how conservation by needle technique may affect fragile and damaged silk after the stitches have been removed.

Investigate the time it takes to execute the most common needle techniques.

Investigate the properties of a conservation intervention that determine visual aesthetic quality among textile conservators.

These studies have resulted in five papers of which the most important results will be accounted for and discussed.

In search of test methods

A pilot study, Paper I, was made to find suitable methods to evaluate the preserving effect of common conservation methods, and to find a method to achieve accelerated wear to imitate the handling a textile undergoes for instance in a museum over the years. It was also made to build a platform of information about conservators' experiences of

costume conservation, methods used, opinion of important criteria for successful conservation, and the history of the conservation studio.

To gain information from the conservation community a questionnaire was sent by letter to 56 museums and private conservation studios, which were expected to work with costume conservation in Europe and North America. The questionnaire was sent to 19 textile conservation studios in Sweden, and was answered by 17 conservators representing 16 studios. The conservation studios were found on the members list of the Swedish Association for Textile Conservation. The 37 foreign studios were found on the ICOM Conservation Committee Textile Working Group members list. In total 18 foreign conservators gave exhaustive answers representing 16 studios. A total of ten conservators from ten studios responded that they were not able to answer the questionnaire as they had no costume conservation or that the conservator was newly appointed. No answer at all was received from a total of 14 conservation studios.

The Swedish respondents were informed that they could answer by letter or be interviewed on the telephone two weeks later if they preferred that. Four answered by letter, the rest were interviewed by me and I subsequently filled in the form. The foreign respondents were instructed to answer by letter as soon as possible. The participants were promised to receive a copy of the article after it had been published. The questionnaire (Appendix 1) had the following questions:

Since when is conservation pursued at your institution?

Please estimate the number of costumes that have been conserved at your institution during its history.

From which year dates the first conservation report?

Which criteria determine if the costume is going to be conserved?

Who makes the decision of method of conservation?

Please mention some of the criteria that you consider important for a successful conservation, for example durability, making it invisible, aesthetic etc.

Mark those methods that have been used/are in use at your institution.¹

Which one of the methods (mentioned above or added by you) is/has been the most common in your institution?

An answer was obtained from the oldest textile conservation studio, also having the earliest conservation reports, in the Museum für Kunst und Gewerbe in Hamburg, Germany; the oldest reports date from 1880-90. Data on the number of conserved costumes varied considerably between 1 and 13,000; the largest number was declared from the Hermitage in St Petersburg. The most often mentioned criterion for considering conservation successful was aesthetical appeal, and second most that it should give durable support preventing the object from further damage. The most common conservation method was to insert a support fabric, between the outer fabric and the lining, which is sewn on by couching

^{1.} Fourteen methods were described and the respondents could also add others.

over the outer fabric but not through the lining. The most common reason for treating a costume was an approaching exhibition, and the decision of which method to use was primarily made by the conservator.

The experimental part of this study involved both silk and wool as they are common materials in the costume collections of the Royal Armoury. As museum objects cannot be used in experimental research, artificially aged fabrics were used as substitute. Standard fabrics, the silk ISO 105-F06:2000 Bombyx mori and the wool ISO 105-F01:2001 were aged in a Weather-ometer with ultraviolet (UV) exposure at 313 nm W.m² at 95% RH and at 50°C ±2°C, (henceforth, this ageing method, with the same RH and temperature parameters will be shortened to UV). Both fibre types were tentatively aged for 1-4, 7 and 14 days. After ageing, the residual strength of the samples was tested according to standard EN-ISO 13937-2:2000. Four days UV exposure for the silk and seven days for the wool was deemed to give a reasonable degree of degradation, meaning that the material had become fragile but still in such a condition that it could be evaluated in the selected testing equipment.

The samples were exposed to five mechanical methods to see if they could achieve wear similar to long-term museum handling or be used in the evaluation of conservation interventions: abrasion by Nu-Martindale (as per SS-EN ISO 12947-2:1999), tensile strength test (as per EN ISO 13934-2:199), flexometer abrasion (as per ASTM D 6182, DIN draft 53351), flexometer bending (as per DIN 53 359:1957, 100,000 folds), and shear stiffness (as per Kawabata SS EN 13780:2003). The tested samples were unaged, aged without conservation, or aged and

conserved with laid couching. Unaged silk and wool were used as support fabrics, which had the same size as the samples, and the stitching was carried out with two single plies of silk.

Abrasion by Nu-Martindale, in which the surface of the samples is abraded against a rather coarse woollen fabric, was tested on unaged, aged, and conserved silk and wool samples in triplicates. The silk samples were checked every 100 turns and the wool samples every 500 turns. The abrasion was discontinued when one thread on the sample had broken. The samples were conserved in two variants: with laid couching either covering 20 mm of the sample centre or the entire area (sample size 40 mm Ø, abraded area 28 mm Ø). The Nu-Martindale gave a quite clear difference between the unaged and the aged samples, both silk and wool; the aged samples were much weaker than the unaged samples. The comparison between the un-conserved samples and the conserved samples was somewhat surprising as the conserved samples were weaker than the un-conserved, especially the samples in wool. The reason for this could be that the conserved samples have been handled more, that the laid couching was caught by the abrasion and caused further friction of the aged sample or both. The preliminary results showed that the woollen samples with laid couching over the entire area were stronger than the ones stitched over a smaller area. The use of Nu-Martindale was deemed at this point to be suitable to use in further tests.

The tensile strength test, which measures the force needed to break a sample, was used on another set of unaged, aged, and conserved samples (size 100 x 200 mm, gauge length 50 mm). The conservation was made in two variants. Two samples of each material were conserved with

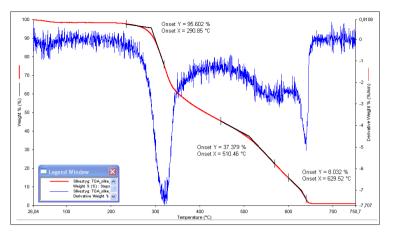


Figure 8. Thermo gravimetric analysis (TGA) of *Bombyx mori* ISO 105/F-1985.

the long stitch of the laid couching following the warp or with the long stitch following the weft direction; the laid couching covered 80 x 110 mm of the sample. Each sample was fastened between clamps including the support fabric, and the force was applied in the warp direction of the sample. The tensile tests, as opposed to the results with Nu-Martindale, showed a clear difference between the unaged samples, aged samples without conservation, and aged samples with conservation. The conserved samples were much stronger than the un-conserved ones and the same as the unaged samples. The direction of the long stitch only affected the silk samples, where the ones with the long stitch in the warp direction were stronger. Tensile properties seemed promising to use for evaluating conservation interventions. However, further tests with a larger number of samples were suggested to further evaluate the methods.

Flexometer abrasion, flexometer bending and shear stiffness did not show any effect when investigated with the samples, implying that they were not useful in this connection.

Significant outcome of Paper I: the most common conservation method was to insert a support fabric between the outer fabric and the lining and to sew it on by couching over the outer fabric but not through the lining. The most important criterion for a successful conservation was its aesthetical appeal, the second that the conservation should give durable support preventing the object from further damage. No tested method was found acceptable to imitate natural accelerated wear, but abrasion by Nu-Martindale (SS-EN ISO 12947-2:1999) and tensile test (SS-EN ISO 13934-2:1999, grab test) were deemed to be suitable to use for evaluating conservation methods. The information from Paper I constituted a stepping stone for the following studies.

Ageing of silk

To be able to evaluate the physical effects of textile conservation methods by material destructive experiments, it was necessary to produce a substitute similar to the fragile fabrics in historic costumes. It was decided to limit the study to concern only white silk with two seventeenth century costumes as references for the degra-

dation level to achieve. The Royal Armoury's collections consist of a unique amount of royal costumes from this time period with silk as a common material. Moreover, silk is a vulnerable material that often is very difficult to conserve when degraded. White silk was chosen in order to reduce interference from dyes and to make the results of studies on historic materials more comparable to standard silk. Paper II focuses on developing a suitable ageing method. Samples from the historic costumes had to be taken to be able to establish the silk's condition and to find markers for degradation through mechanical and chemical analyses. The samples were taken from Gustav II Adolf's coronation costume, 1617, from the lining in tabby of the doublet (inv no Lrk 25605), the satin in the breeches (inv no Lrk 25606), and the satin in Karl X Gustav's coronation cloak (inv no Lrk 25599) from 1654, figure 2. For the tensile tests, weft yarn was taken from cut edges in the seam allowance and for the chemical analyses pieces of approximately 5 mm² were taken from the seam allowance.

Although the silk ISO 105-F06:2000 has a given tex value, it was decided to measure the tex value of the yarn in the silk. The tex value was calculated by carefully removing a number of threads of the silk samples as well as from the historic costumes, and their un-crimped length was determined under a tension of 4 cN. The threads were weighed together, and from their total length and total weight the linear density was calculated. This method was suggested by Swerea IVF to give the same starting-point for all the samples as the ageing process affects the silk's tex value. The method may not be exact but as all the samples were treated in the same way, it was judged to be acceptable. The standard silk is said to be free from finishes and residual chemicals (ISO

105/F-1985) but to investigate this further thermo gravimetric analysis (TGA) was performed using a PerkinElmer Pyris 1 TGA in air atmosphere, with a heating rate of 10°C/min. The sample weight was about 1.4 mg and the instrument was calibrated against the magnetic transition temperatures of alumel (154.2°C) and iron (780°C).

According to the thermo gravimetric analysis presented in figure 8 most of the organic material was burnt off at about 650°C and evaporated. As the sample was not conditioned before the analysis, about 3 wt% of water evaporated before the analysis started. Thus, the sample probably had a water content of 4-5 wt%. The decomposition of the silk started at about 150°C. Low molecular weight compounds such as CO, CO, and NH, start to be released at around 150°C (Yanagi, Kondo & Hirabayashi, 2000). The result of the analysis showed that the tested sample contained 0.5-1 wt% of an inorganic unidentified substance, though the declaration of contents from the ISO standard states that it is free from finishes and chemical residues. The obtained content can be due to the ion exchange properties of silk, which might result in accumulation of salts from the handling of the sample or from the washing process in the production. Nevertheless, this was not perceived to influence the analyses.

The standard silk fabric ISO 105-F06:2000 *Bombyx mori* was treated by four artificial ageing methods considered to degrade silk: (a) accelerated UV exposure at 313 nm W.m² at 95% RH and at 50°C ±2°C for 1-10 days; (b) thermoxidation in dry air at 125°C for 14, 21, 28, 35, 42, 49 or 56 days; in air with 53% RH at 25°C or ≈42.5% RH in 60°C for 21 or 28 days; (c) exposure to 0, 53, 75, 86, 100% relative humidity (RH) at 25°C or 0, ≈42.5, 74.5, 80.25% RH at

60°C for 28 days; (d) immersion in and exposure to solutions of pH 1, 3, 7, 11 or 13 at 25°C or 60°C for 21 or 28 days. The methods all represent environmental conditions recommended to avoid in the preservation of textiles. The RH value using Mg(NO₃)₂ in 60°C is uncertain and was approximately 42.5% instead of 53%.

Accelerated UV exposure is a common method in the textile industry when testing the ageing properties of a fabric and was recommended already for the tests in Paper I by Swerea IVF who also made this second batch. Thermo-oxidation was used in spite of earlier findings that the fibres from Bombyx mori silk display an exceptional thermal stability as measured by changes in a number of physical parameters (Tsukada, Freddi, Nagura, Ishikawa & Kasai, 1992). Extreme humidity conditions, both at high and low levels, are known to have negative effects on silk by aiding deterioration (Morton & Hearle, 2008). Silk and other protein fibres are known to be sensitive to alkali and extreme acidic pH conditions (Tímár-Balázsy & Eastop, 1998).

Analysing effects of accelerated ageing

To assess analytical markers for the historic silk, to compare it with the artificially aged silk, and make it possible to evaluate the four ageing methods, the silk was analysed with Attenuated Total Reflection — Fourier Transform Infrared Spectroscopy (ATR-FTIR), Size Exclusion Chromatography (SEC) and tensile tests.

A series of attempts to use FT-Raman as an analytical method for the unaged and artificially aged silk samples were performed at Chalmers University of Technology at the Department for Applied Physics by Dr Holmlund. The results though, did not give clear answers; the yellow

colour of the silk aged by UV and thermo-oxidation caused problems and a very long time span was needed to run the analyses in order not to burn the samples. No further FT-Raman tests were performed. UV irradiation and heat causes yellowing of silk. According to Setoyama (1982) photo yellowing increases the amount of tryptophan and heat yellowing increases the loss of hydroxyl amino acids. According to Shao, Zheng, Liu, and Carr (2005) yellowing is primarily caused by the photo oxidation of tyrosine as it forms yellow chromophores residues. Residues of tryptophan also causes this effect but not to the same extent.

Formation of carbonyl groups is a common signature of the occurrence of oxidation processes in natural and synthetic macromolecules. This signature was found in the ATR-FTIR analyses for the historic samples and for the samples exposed to UV and thermo-oxidation at 125°C over different time periods. The samples immersed in low pH buffer environments (pH 1 and pH 3) showed a slight increase in the carbonyl absorbance. Tyrosine is one of the amino acids in silk's amorphous region and a slight decrease in the intensity of the tyrosine doublet bands was observed for the historic samples, the silk samples subjected to high alkaline conditions (pH 13), and to thermooxidation in dry air at 125°C. UV radiation was responsible for an extensive decrease in the tyrosine related peaks in the ATR-FTIR spectra at prolonged exposure.

In general, the SEC analyses of the historic samples showed lower molecular weight distributions than the artificially aged silk. Thermo-oxidation at high temperatures caused a decrease in the molecular weight distribution, as did immersion in acidic (pH 1) and alkaline (pH 13) environments.

This effect was more noticeable for the alkaline environment as expected for proteins (Hallett & Howell, 2005). UV radiation may induce complex alterations in the chain structure of silk macromolecules; when dissolving UV-exposed samples in DMAc, a slight suspension could be observed instead of the clear solution obtained for the rest of the samples. This fact might indicate the presence of cross-linked silk chains induced by UV radiation. Of the historic samples, the ones from Gustav II Adolf's breeches show a slightly higher relative molecular weight than the other two historic samples. The molecular weight distributions were calculated by comparison with pullulan standards (Agilent, USA).

The tensile tests were performed by an automatic test procedure with a Vibrodyn® CRE-type according to IN EN ISO 5079, ASTM D 3822. Five yarns from each ageing method were tested. The results showed that the historic samples have lower modulus, tenacity and extension than unaged standard silk, as expected. With respect to acidity and alkalinity, low pH caused a slight decrease in the modulus, tenacity and extension; whereas, exposure to strong alkaline conditions (pH 13) caused a total disruption. Thermo-oxidation at 125°C in dry air and UV irradiation at some exposure times resulted in reductions in extension and tenacity at break that are comparable to the effects of the natural ageing factors in the historic samples. The results of the modulus was surprising as the values, at the same exposure times as referred to above, were far under the historic samples. Short exposure time while ageing by thermo-oxidation (14-21 days) and UV irradiation (2-3 days) resulted in a reduction which was "too low" in tenacity and extension, but results in similar reduction in modulus as in the historic samples. Ageing in an environment with different relative humidities did not cause any effect on the silk.

The most important aim of Paper II was to achieve a method to artificially age silk to a state similar to the reference silk from Gustav II Adolf's and Karl X Gustav's costumes and to find analytical markers for naturally and artificially aged silk. Of the four environmental methods that were tested, both UV exposure and thermo-oxidation proved to give satisfactory results. With the results presented above of the different analytical methods, it was concluded that thermo-oxidation in dry air at 125°C for 28-56 days gave a silk most similar to the tested samples of the historic costumes. Thermo-oxidation at 125°C was to be preferred as it is easy to use and the sample size and shape is not fixed. Exposure to UV (in combination with 95% RH and 50°C ±2°C) had good qualities but seemed too harsh and has the disadvantage that it does not affect the sample homogeneously as the UV irradiation exposes only the surface of the sample.

The study in Paper III was made to further investigate the influence of physical-chemical mechanisms of ageing on silk fibres. To further establish the analytical markers for the ageing process, the historic silk samples and the artificially aged silk from Paper II were analysed at both molecular and macroscopic levels, and the experimental results were correlated using multivariate data analysis. The properties of modulus, elongation at break, and tenacity at break of all variants of ageing are reported and their significance is calculated in Paper III. Additional analyses were made through amino acid analyses, pH measurements and brightness. Furthermore the semi-quantitative results of the carbonyl and Amide II crystalline indexes were

calculated from ATR-FTIR measurements. In order to carry out the amino acid analyses and the pH measurements of the historic costumes additional samples were taken from the historic costumes, in their seam allowances.

These expanded data were used for exploratory principal component analysis (PCA) to obtain correlations between the effect of the different artificial ageing environments and the properties of the historic silk samples.

The result of the pH measurements showed that the historic samples have similar pH values to the samples exposed to accelerated UV for ten days and the samples subjected to thermo-oxidation at 125°C for 28 days.

Brightness, which is a degradation marker (yellowing), was not measured for the historic silk and could therefore not be compared with the artificially aged samples. The UV-aged samples had an exponential initial decrease in brightness, which stabilised after 2-4 days, and pH 1 and pH 13 gave similar results. Thermo-oxidation at high temperatures (125°C) caused a progressive linear decrease in brightness with exposure time while RH did not affect the brightness.

In Paper III, the secondary conformation of the aged silk samples was analysed in more detail with the ATR-FTIR analyses. Calculating the Amide III crystalline index using the relative height intensities from the bands at wave numbers 1260 and 1227 cm⁻¹, reveals subtle changes in the fibre conformation. UV irradiation caused an increase in crystallinity as did thermo-oxidation at 125°C, but less than UV irradiation. Also, pH exposure at pH 1 and pH 13 increased the crystallinity index while exposure to different RH conditions did not seem to alter the

conformation at the Amide III region. Only the sample from Gustav II Adolf's doublet shows a slight increase in the crystalline index while the other historic samples have similar values to the unaged reference silk. Accelerated ageing by UV caused a drastic reduction in the intensity of the tyrosine (Tyr) doublet band; there was also a reduction for the historic samples but not as drastic as the one caused by UV exposure. A similar decrease as for the historic samples is observed for the high temperature thermo-oxidized samples exposed for 56 days.

The result of the amino acid analyses confirms the result of the ATR-FTIR analyses of the effect on tyrosine stated in Paper II. Accelerated UV exposure causes a marked decrease of Tyr, and thermo-oxidation also causes a progressive decrease but less than UV exposure. No decrease of Tyr can be observed in the samples exposed to pH 1, pH 13 or in those exposed to 100% RH. The historic samples also exhibit lower Tyr values indicating oxidation during natural ageing. An even better marker for increasing oxidative changes may be found in the decreasing Tyr/Ala ratio, where Tyr in many cases is transformed into Ala. Again the samples exposed to more moderate pH as well as the RH exposed samples are unaffected. The results of how Tyr has affected the samples exposed to pH I and pH I3 do not agree with the brightness measurements, which showed a decrease in brightness.

The analyses with SEC chromatograms in Paper III support the earlier results of Paper II.

In Paper III, the tensile tests were used to compare the mechanical properties of the artificially aged silk with the historic silk and to put it into relation with the other analyses. Elongation at break and tenacity were judged to be the most

important mechanical properties to consider since these properties are relevant when a specimen is subjected to stretching, as when a historic costume is being mounted on a mannequin which is often the case for costumes at museums.

Accelerated ageing by UV exposure resulted in a sudden increase of the modulus after 1-3 days and a progressive decrease thereafter, reaching the modulus of unaged silk after 5-6 days, while the tenacity and elongation at break decreased from day one. Thermo-oxidative exposure at higher temperatures (125°C) reduced break extension and tenacity already from the shortest time exposure with some levelling effects after 28 days while the modulus was high and significantly higher than the modulus of the unaged reference silk. This could also be seen for the samples exposed to lower temperatures (60°C).

Relative humidity (RH) seems to have very little effect on the mechanical properties. Only one difference in break extension was significant: 0% RH in 125°C (p= .016); note the high temperature. Immersion of the silk in extreme pH conditions caused severe effects on their mechanical properties, therefore, only samples immersed in pH 1, 3, 7, 11 at 25°C and pH 3, 7 and 11 at 60°C could be tested. None of them gave a result similar to the historic samples.

Historic silk samples showed slightly higher modulus values and much reduced elongation at break and tenacity in comparison with the reference silk. Indeed, the seventeenth century silk showed on average a tenacity that is 17% of the reference silk. This is a clear indication of the brittleness of the historic silk fibres caused by combined physical and chemical structural effects during their natural ageing.

So far, results describing similarities in individual analytical markers between the historic samples and the samples exposed to different environmental conditions have been commented on. But there has not been any analysis of the similarities when all the 24 analytical markers and all samples are analysed together. Such analyses were tentatively performed by using Principal Component Analysis (PCA) and clustering methods. In PCA correlations indicated similarities (when the correlation is positive, high scores in one variable tend to follow high scores in another variable, and low scores in one variable tend to follow low scores in another variable), and in the clustering methods similarities were expressed as degree of numerical differences between variables.

In PCA, the historic samples, the two high temperature samples (125°C for 28 and 56 days) and the short time UV (4 days) sample were similar to each other. On the other hand, the samples subjected to RH (100% at 25°C for 28 days), pH 1 (25°C for 28 days) and pH 13 (25°C for 28 days) had the character of outliers. Moreover, the samples subjected to UV degradation for ten days were less similar to the historic samples. In a similar way, when clustering, the methods grouped the three historic samples together, while unaged silk, 100% RH, pH 1 and pH 13 were never combined with the historic samples. However, although the short time UV samples and the high temperature exposure samples tended to be similar to the historic samples, the results of the different clustering methods were not consistent in this respect.

The conclusion is that thermo-oxidation at 125°C is the most suitable method to use when simulating naturally aged silk similar to the references of the seventeenth century costumes,

and that the degree of degradation can be controlled by the exposure time.

To prepare for the next step, the experiments in Paper IV based on surrogates of the historic silk, it was necessary to find suitable equipment, as the number of samples required an oven generous in size. It was also important to make sure that the desired degree of ageing as in the previous tests would be achieved resulting in the same tenacity as the yarn in the historic costumes in Paper II. Therefore, several pre-tests were made with different ovens. In Paper III it was found that the decrease in the mechanical properties (elongation and tenacity at break) correlated well with structural markers such as the decrease of the Tyr content and the molar mass, and the increase of the carbonyl and crystalline indexes. To establish the effect of the ageing in the pre-tests the carbonyl indexes were analysed by ATR-FTIR, and the yarns' tenacity was measured by tensile testing.

The correct equipment and competence was found at SP Technical Research Institute of Sweden with ventilated and calibrated ovens large enough to accommodate the silk samples. It could be established that 42 days was the required time to age the samples to achieve surrogates with fragility similar to the historic samples.

Effect of three stitched interventions

In the study presented in Paper IV, three common stitched support methods were investigated with regard to their effect on the property maximum force at break. Tensile tests were executed on silk surrogates with similar properties as seventeenth century silk, obtained with the high temperature ageing method developed earlier.

In this study, 60 rectangular and 60 circular silk specimens were stamped out in standard silk 105-Fo6:2000 and were dried in a desiccator, 55 specimens at a time, over silica gel until the relative humidity stabilised at 4%. Ten specimens were kept as references while the others were suspended in 1000-ml borosilicate glass bottles. To suspend the specimen in the bottles a silk thread was attached 5 mm down onto the middle of the short end of the rectangular specimen and 5 mm down onto the edge of the circular specimen. The thread was placed under the screw-top polypropylene caps to secure the suspending thread so that the specimen hung vertically in the warp direction. To the bottom of each bottle 42 g calcium chloride (CaCl₂) was added to maintain a low relative humidity. The bottles were placed standing in the oven at a mean temperature of 125°C for 42 days.

Based on the answers to the questionnaire in Paper I and the consulted conservation reports at the Abegg Foundation (Switzerland) and the Royal Armoury (Sweden) that concern costumes, it was decided to evaluate and compare three methods that according to the questionnaire study, Paper I, and the conservation records were the most common: (I) - a damage is secured onto a support fabric with laid couching, (2) - a damage is secured onto a support fabric with spaced brick couching, (3) - a damage is covered with silk crepeline which is attached with running stitches.

It was decided to test these interventions on two typical types of damage for silk costumes: *tear* and *wear*. To achieve a tear, a scalpel was used cutting a 50 mm slit across the warp in the middle of the rectangular surrogate. How to achieve wear was a more difficult decision. Different methods to achieve wear were tested by different techniques in the study in Paper I. Asai, Biggs, Ewer, and

Hallett, (2008) who made a study involving tapestries used a glass bristle brush to create weak areas on tapestry samples, but their description was not detailed enough to be able to replicate for this study. It was decided to achieve wear by abrasion with Nu-Martindale, a mechanical test used in Paper I to achieve accelerated wear (called "museum handling" in Paper I). This equipment required circular samples and achieved a damaged area of 80 mm². A textile conservator with nine years of professional experience and employed at the Royal Armoury's conservation studio at the time executed the conservation interventions. Fine hair silk no 11 was used for the stitching with the tenacity 39 [cN/Tex] which is in line with the tests by Benson, Lennard, and Smith (2014). Standard silk 105-F06:2000 was used as support fabric in method (1) and (2), and silk crepeline was used in method (3). The intention was to treat the surrogates as if they had been real objects and to do the stitching that was found necessary. A film explaining in detail how the methods were carried out for the study in Paper IV is available (YouTube-film Nilsson), Appendix 2.

A number of the surrogates were subjected to accelerated wear after conservation in order to make it possible to investigate the resistance of the conservation to accelerated wear. Prior to degrading to achieve accelerated wear, the surrogates were given a temporary lining of standard silk, ISO 105-F06:2000, attached by running stitches close to the surrogates edges. The lining was there to make the wear more natural; the silk costumes in the Royal Armoury's collection have a lining that protects and supports the costume's outer fabric. Before a decision was taken on which method to use for this purpose a pre-test was made testing both pilling and Nu-Martindale. Nu-Martindale per SS-EN ISO 12947-2:199 was also tested in Pa-

per I, amongst other methods. It was found promising but was rejected in this study as the method was too hard to control and time consuming. The uncertainty of some results in Paper I also contributed to its rejection. Finally, it was decided to use standard methods for washing and tumble drying. It was somewhat surprising that it took quite a long time to achieve the desired result, which was to make the old intervention lose its supporting effect and to be considered unacceptable to the conservator's eye. The advantage with this method was the even handling without any particular pressure or mounting issues. The tumbling can also be interrupted and the result checked at any point.

Tensile testing was used to measure the mechanical property maximum force at break of the surrogates according to EN ISO13934-2:1999, the grab method using an Instron 5966. The available choice of testing methods was quite limited due to the shape and construction of the surrogates subjected to testing, and Swerea IVF suggested the grab method. In the tensile strength test of the conserved samples made in Paper I, both the support fabric and the aged samples were fastened together in the clamps of the equipment. As this was likely to affect the result it was decided not to fasten the support fabric or the crepeline but only the aged silk in the tests in this study, focusing on the strength of the aged silk when it was supported by the conservation.

Surrogates were subjected to tests of maximum force at break in a cumulative series of treatments: before damaging, after damaging, after conservation of the damaged surrogate, after accelerated wear of the conserved surrogate and, finally, after these treatments and removal of all stitches.

The tested conservation methods greatly augment the surrogates' maximum force at break

Table 1. Time required executing interventions on surrogates

Conservator					
Conservation method	I	2	Mean		
(A) laid couching	88 min	43.5 min	66 min		
(B) brick couching	43.5 min	47.5 min	45.5 min		
(C) crepeline	56 min	33 min	44.5 min		

of damaged areas in fragile silk; between more than three and more than five times depending on conservation method. Still, even the best method, laid couching, did not restore the damaged surrogate to more than 27% of the strength of undamaged surrogates. This seems to me like a well-balanced intervention: the risk for the damage to increase is reduced, but it still breaks before the undamaged area is harmed.

After conservation, the surrogates with tear were significantly stronger than crepeline with both laid couching and brick couching. The abraded surrogates with laid couching were significantly superior to both brick couching and crepeline. After accelerated wear there were significant differences only for surrogates with tear. Both laid couching and brick couching had significantly higher force at break after accelerated wear than crepeline. When the conservation had been removed, after accelerated wear there were significant differences only for abraded surrogates. Surrogates with laid couching and brick couching were significantly weaker than the surrogates conserved with crepeline.

In the choice of conservation method it is important that the silk fabric to be conserved is in such condition that it withstands extensive stitching if laid couching or brick couching is chosen. If museum textiles are to be conserved, it is feasible to use an intervention that gives the object sufficient support while at the same time causes very little negative influence. The suggestion here is that a museum object, which is expected to be handled with great care, could manage well with a treatment that makes it three times stronger even though this means that it is much weaker than when it was as new. Therefore, if a very fragile silk is to be conserved, crepeline is suggested to be the best method as the number of stitches is low and the crepeline still has a good protective effect for surrogates with tear and abraded surrogates. In addition, crepeline affects the fragile silk the least as shown after removal of the conservations. A conservation method that results in minimal harm is especially important when conservation may have to be replaced after a time. Also, it is likely that textiles treated with crepeline can benefit from their weakness being more obvious than textiles treated with laid couching or brick couching and therefore might be handled with more care.

Textiles that are in use, such as ecclesiastical textiles, are often managed differently as the common stitched interventions do not give enough support. Worn areas, on textiles such as chasubles and antependia, are therefore often supported with laid couching and covered with a new fabric that complements the original, and when possible a worn fabric is folded to hide and protect the damage.

Table 2. Time required executing interventions on baby bonnets

		Conservator		
Conservation method	I	2	3	Mean
(A) laid couching	75 min	90 min	135 min	100 min
(B) brick couching	75 min	150 min	150 min	125 min
(C) crepeline	135 min	150 min	135 min	140 min

Time required executing interventions

During the work with Paper IV and a yet unpublished study of aesthetic preferences in three conservation methods, two tests were made to measure the time taken to execute the three types of conservation interventions.

In the first test, surrogates similar to those damaged by abrasion in Paper IV were used. Before conservation, the surrogates were lined with standard silk (ISO 105-F06:2000). Thereafter, the damage was conserved with laid couching, brick couching, or crepeline. The interventions were made by textile conservator 1, with nine years of professional experience and conservator 2, the author, with twenty-three years of experience, both employed at LSH's conservation studio at the time. They treated two surrogates each with every method. The time required is presented in Table 1.

In the second test, three textile conservators were given the task to conserve three copies of baby bonnets, using the same methods as above. The damaged area to be treated was made in a fabric of plain woven monochrome silk, 25 x 25 mm² and mechanically produced with a scalpel by removing warp and weft to create a lacuna similar to a natural worn area. The bonnets had an interlining of the same silk as the outer fabric to be conserved and a lining in cotton. The interlining was given the function of support fabric, to which the damage was secured with laid couching and

brick couching respectively. The crepeline was to cover the whole left sidepiece in order not to form a patch, with the edges folded inwards and attached with running stitches. It was also secured to the damaged silk by a few rows of short running stitches. The conservators were instructed to use the stitching spacing they found best and were instructed not to stitch into the cotton lining. They were supplied with the crepeline and thread. The result of the time required is presented in Table 2. The conservators treated three bonnets each, using the three types of interventions. Two of the conservators did the work on commission and the third conservator was the author. Conservator 1 has twenty-six years of professional experience, conservator 2 has ten years and conservator 3 has twenty-four years of experience.

The result from the tests does not give a clear answer to which method is the quickest to execute or if any of the conservators has a favourite method which they are used to use. In Test 1 laid couching on average took the longest time to execute and in Test 2 it was the quickest. The variation between the conservators is considerable in both tests. The conservators with the longest experience are the quickest in both tests but in Test 2 the years of experience does not differ more than two years between the most experienced conservators. An explanation to this

Table 3. Short description of the seven attribute scales.

- 1. Increasingly invisible interventions.
- 2. Increasingly orderly and structured interventions.
- 3. Increasingly adapted interventions integrated with the textile item.
- 4. Best craftsmanship.
- 5. Increasingly good execution: coherent, taut and tight, professional impression.
- 6. Increasingly good adaptation of the conservation material to the item.
- 7. The damages are repaired and the object looks complete.

is probably that conservator I who works on commission is more skilled in stitching than conservator 3.

Aesthetic attributes in textile conservation

The criterion "aesthetically appealing" was found to be the most important to consider when deeming a conservation successful according to the conservators' answers to the questionnaire of Paper I. The underlying factors to determine aesthetic quality of conservation interventions were explored in Paper V. Textile conservators from the Swedish Association for Textile Conservation were engaged in the study.

In Experiment 1, a group of 24 conservators were asked to rank 33 examples of conservation interventions. For practical reasons, and in order not to harm the textiles through excessive handling, the experiment was performed by using photographs. They consisted of matte colour prints of digital photographs, each depicting an individual conservation intervention on a textile object from the collections of the Royal Armoury or the Hallwyl Museum in Stockholm. The interventions were authentic except for three that were temporary and arranged for the purpose of the study, as examples of less aesthetic interventions, to increase the range of quality. All interventions were photographed under exactly the same conditions - including identical lighting and printed at 1:1 scale.

The interventions represented three common methods: laid couching, brick couching, or cover with crepeline attached with running stitches. The 24 participants sorted the photographs into three groups of (1) low, (2) intermediate and (3) high aesthetic quality based on their own understanding of the concept. The participants were then interviewed about what criteria they had developed and used for sorting the interventions. From the interview results, seven descriptions were distilled by me in order to express seven underlying attributes of aesthetic quality found amongst Groups 1–3. The construction of the descriptive sentences was made in cooperation with the participants in Experiment 1 by e-mail. The seven attributes are described in Table 3.

In Experiment 2 the objective was to identify the most important attributes of aesthetic quality, as developed in Experiment 1.

A new group of ten conservators sorted the 33 photographs used in Experiment 1 seven times, each sorting representing one of the seven attributes from Experiment 1. In order to avoid scaling-order effects, each participant used the seven attribute scales in a unique irregular order. A full session lasted for at most 2.5 hours.

The sorting was analysed by rank-order correlations that revealed two latent factors that the authors interpreted as Coherence and Com-

pleteness. Coherence was represented by the attribute scales 3, 5 and 6 while Completeness was represented by attribute scales 2, 4 and 7. Ordinal regression analysis revealed that Coherence was the only significant predictor of aesthetic quality. This means that a successful conservation from an aesthetic point of view is well integrated with the textile in terms of the material and method and does not leave damage area untreated; the descriptions in Table 3 can be regarded as a whole.

Discussion

In this section I will discuss limitations, short-comings and ethical problems of the studies that my thesis is based upon.

A shortcoming in Paper I is a linguistic one. It would have been valuable to have introduced an inventory of types of stitches used in textile conservation and the corresponding names in the English and Swedish languages before formulating the questionnaire. As the English expression "couching" can be used for both brick couching and laid couching, misunderstanding might occur. Furthermore, in Swedish, there is no corresponding expression for "brick couching". In the questionnaire, the word couching was used and it is not always possible to tell whether the non-Swedish respondents meant brick couching or laid couching.

There is an ethical problem in Papers II and III. To be able to perform the analyses samples were taken from historic costumes though this should be avoided. Sampling by cutting a piece from a fabric or pulling a yarn from the weave, means some destruction of the object even though it is made at a hidden place in the seam allowance. Yet, it was decided to take samples as the result was expected to give information that

would justify the sampling. A serious limitation, however, is the small number of historic samples available for the analyses.

Have the studies in Papers II and III given information that justifies the sampling of the historic costumes? The studies have resulted in an ageing method achieving a silk similar to early seventeenth century silk which can be used as an adequate experimental substitute for such historic silk. This ageing method was applied in the study reported in Paper IV. The gained data are accessible for anyone and can also be used for other purposes such as assessing deterioration levels in silk collections. Appelbaum (2007) states: "...relevant to preservation is information bearing on an object's ageing. This includes its likely physical response to its environment..." "Such information helps us predict the behaviour of the object during treatment and develop recommendations for its future care." (p. 18).

The historic samples used in Papers II and III must be regarded as examples of the condition of seventeenth century silk and not as general representatives from this time period. The condition of silk can vary enormously depending on many circumstances such as how it has been produced, used and exposed, just to name a few examples. As the historic samples have been taken from the seam allowance it can be discussed how representative they are for the general condition of the specific costume. The fabric in the seam allowance must have been somewhat protected, other parts of the costumes might be even more fragile. This protection may also be an advantage, however; the silk in the seam allowance has been somewhat shielded from substances that might have affected the analyses such as the amino acid analyses.

The analysis methods used in Papers II and III have all given valuable information about the silk's properties and degree of degradation, but their disadvantage is that they are destructive. ATR-FTIR is often regarded as non-destructive but in this study the pressure of the crystal left marks and the degraded samples crumbled. Tensile tests are destructive in the sense that they tear the material apart.

Surrogates that were used in the experiments reported in Paper IV instead of real historic objects are sometimes criticised because a surrogate never can be the same as a unique object (Muñoz Viñas, 2005). It is of course true that real objects are more complex than artificially produced surrogates but systematic studies require a series of objects, and a series of real historic objects introduces random variance that is difficult to deal with. This is also discussed by Kerr (1989) who claims that it is important that scientists and conservators listen to each other to understand each other's possibilities and shortcomings. She argues for the necessity of using surrogates when developing methods and carrying out experiments before they are used on original objects. Also Garside (2012) states that surrogate materials are of great value in conservation as they can provide information about the current condition of an object, help us to understand degradation factors, and help to develop preserving methods.

The intention with the conservation interventions investigated in Paper IV was to make them in a way that was regarded as natural as possible by conservators. The brick couching and the laid couching were adjusted to cover the damage of the abraded surrogates; the shape of the damage varied somewhat. An alternative to this could have been to make the stitching regular and even

all over the damage area, but this alternative was rejected as it would have made the work less genuine. An advantage of a less genuine but more systematic performance is that it might permit systematic comparisons of, for instance, the effect of different number of sewn rows, length of rows, or distances between rows. The decision to use a naturalistic approach was made as the results should shed light on the choice of complex conservation methods. An advantage of the study in Paper IV was that the number of sewn rows was similar for laid couching and brick couching, supporting the comparison of the two methods.

The time needed to execute the three stitching methods has also been studied in this thesis. No clear result is obtained. The time consumption seems to depend on several factors such as what is being conserved and by whom. Conservators may have a favourite method that they often practice which may effect the time required. This issue has to be further investigated.

The method to age silk using different salts to achieve certain RH was uncertain for the samples exposed in 60°C, especially the batch with the intention to keep 53% RH which probably gave a RH closer to 42.5%. KCl was used to achieve 86% RH but gave instead 80.25±0.41%. NaCl was used to achieve 75% RH and did instead give 74.5±0.30% which must be regarded as quite close to the aim. This deviation does not change the interpretation of the result that the humidity did not affect the silk, instead it is the temperature that is crucial. Another shortcoming is that the salts' effect on the silk has not been surveyed in this study.

An unsatisfactory result with the ageing methods used in Papers I-IV concerns how modulus is affected. The samples exposed to thermo-

oxidation or UV at short exposure time, have significantly higher tenacity and extension than the historic samples. Modulus is affected differently; at short exposure time both thermo-oxidation and UV produce modulus values similar to the historic samples, while at longer exposure time modulus generally is significantly lower. It appears that it is not possible to find exposure times for the two ageing methods that make the results similar to the historic samples in all three measures, tenacity, extension and modulus. In a study of accelerated ageing of cellulose versus natural ageing, Erhardt and Mecklenburg (1995) present results that conclude that the major effect of ageing cellulose is loss of strength but leaving the modulus unchanged. Their tests also show that raised temperature speeds up the ageing process without fundamentally altering it, but that changing relative humidity alters the ageing process itself. This has not been found in the present studies made on silk, different RH levels have not shown any clear effect on the ageing of the material. Erhardt and Mecklenburg (1995) have studied the distribution of reaction products and that might also be a reason why the results do not agree, the samples also comprise two quite different materials.

The conclusion from Papers II and III was to use thermo-oxidation at 125°C to produce a silk similar to seventeenth century silk, and it was also the method used to produce the surrogates in Paper IV. It is clear from the results that the time periods have a big influence on the result and that prolonged time spans made the samples less similar to the historic samples concerning modulus. Also with UV, the modulus was affected after short time. The number of samples tested, both historic and artificially aged, was

limited. Also the variants of the two best ageing methods were limited, in time and effect (UV and high temperature). Further investigations using a broader spectrum of historic samples, more varied and expanded samples of UV and thermo-oxidation as well as UV and high temperature together will further develop this issue.

A limitation in the tests is that only a few conservation materials were tested; silk crepeline, hair silk and standard silk. But as this thesis focuses on evaluating the stitching methods, rather than the material, this choice felt natural.

Assessing the property maximum force at break by the grab test, as done in Paper IV, is convenient. A limitation though is that the method does not assess extension at break and Young's modulus when as in this case several layers of fabric are tested.

Paper V explores the attributes that determine aesthetic quality of conservation interventions, the criterion most mentioned to describe successful conservations. The study resulted in two predictors of aesthetic quality according to Swedish textile conservators, Coherence and Completeness, Coherence being the only statistically significant. This result implies that the experiences of Coherence and Completeness are not independent of each other: the experiences are one-dimensional. The seven scales describing Coherence and Completeness can be used together forming one broad scale of aesthetic quality. They are described in Table 3. The study is to be followed by other studies concerning evaluation of the aesthetic appearance of methods used in textile conservation.

Conclusions

The three most common methods when using needle techniques when conserving costumes are laid couching, brick couching with a support fabric inserted under the damage, or a cover of silk crepeline.

The most important criterion for a successful conservation was aesthetical appeal.

The best method, to achieve a silk similar to examples of seventeenth century silk is thermo-oxidation in 125°C.

Crepeline increases the surrogates' strength three times and is the gentlest method for the abraded surrogates according to their state after accelerated wear and removal of the conservation. However, the capacity of crepeline to make damaged surrogates stronger is less than the protective effect of laid couching and brick couching.

Laid couching made the abraded surrogates the strongest and increased their strength more than five times.

Laid couching and brick couching are the methods that make the surrogates with tear withstand accelerated wear the best.

Further investigations are necessary to be able to draw any conclusions about the time requirements for the interventions.

The most important factor that determines an aesthetic intervention is that it is coherent.

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Appendix

Appendix 1: Inquiry – Conservation methods for historical costumes

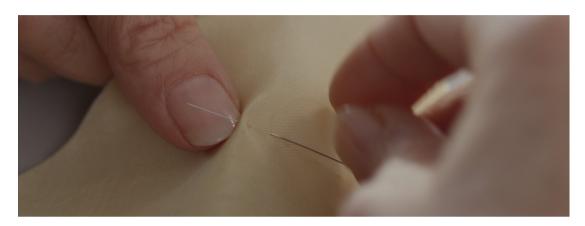
name of conservator:
name of institution:
e-mail-address:
telephone number:
1. Since when is conservation pursued at your institution?
Please estimate the number of costumes that have been conserved at your institution during its history.
3. From which year dates the first conservation report?
4. Which criteria determine if the costume is going to be conserved?
5. Who makes the decision of method of conservation?
6. Please mention some of the criteria that you consider important for a successful conservation, for example durability, making it invisible, aesthetic etc.
7. Please mark with X those methods that have been used/are in use at your institution. Please complete with other methods used/in use.

	Mark with X	
A. A support fabric is put in between the outer fabric and lining and is sewn on by couching over the outer fabric and through the support fabric, but not through the lining .		
B . A support fabric is put in between the outer fabric and lining and is sewn on by running stitches , thread by thread on the support fabric, but not through the lining .		
<i>C</i> . A support fabric is put in between the outer fabric and lining and is sewn on by couching through all fabric layers.		
D. A support fabric is put in between the outer fabric and lining and is sewn on by running stitches through all fabric layers.		
<i>E</i> . A broken or fragile area is covered with silk crepeline , and only the outer edges of the crepeline part are sewn on.		
<i>F.</i> As A, B, C, or D and is covered with silk crepeline. Please note if method A, B, C or D is used in combination with silk crepeline.		A B C D
G. A broken or fragile area is covered with polyester crepeline (Stabiltex) and only the polyester crepelines outer edges are sewn on.		
<i>H.</i> As A, B, C or D and is covered with polyester crepeline (Stabiltex). Please note if method A, B, C or D is used with polyester crepeline.		A B C D
<i>I.</i> A new fabric , similar to the original, is sewn on and covers the whole or parts of a broken/fragile area, which is first conserved by sewing . The area beneath is left without measure.		
J. A new fabric, similar to the original, is sewn on and covers the whole or parts of a broken/fragile area, which is first conserved by sewing. Please note with which method it has been conserved by sewing.		
K. A new fabric , similar to the original, is sewn on and covers the whole or parts of a broken/fragile area, which is first conserved by adhesive treatment . Please note which material has been used.		
L. Conservation method is change of way of storing/keeping. Please describe in what way.		
M. The costume has been left without measure, but a reconstruction/copy has been made.		
N. The costume has been conserved and a reconstruction/copy has been made.		

Since I may have omitted some method, please complete here with other methods used:

Appendix 2: Stitched support methods using laid couching, brick couching and crepeline attached by running stitches (video)

The English version https://www.youtube.com/watch?v=eoogh96C7eI Den svenska versionen https://www.youtube.com/watch?v=iQSX4u3PqNA













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Papers

Paper I

Johanna Nilsson. "A survey of the most common support methods used on historical costumes and a preliminary investigation of tests assessing the quality of conserved fabrics." Post print at the conference Scientific Analysis of Ancient and Historic Textiles, 2005, University of Southampton, eds. R. Janaway, & P. Wyeth, p. 79-85.

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Error list

 $P.~80, the questionnaire was sent to ~56 conservation studios. \\P.~82 the heading at ~5 Tensile strength testing, "SS-EN ISO 1394 2:1999", should be "EN ISO 13934-2:1999".$

A survey of the most common support methods used on historical costumes and a preliminary investigation of tests assessing the quality of conserved fabrics

Iohanna M. F. Nilsson

ABSTRACT The aim of this study was to find ways of evaluating support methods used in textile conservation. A questionnaire completed by workshops in Sweden and abroad served to identify the most commonly used method of costume support and the main criteria used in determining the success of a conservation treatment. The most common method is for a support to be inserted between the outer fabric and lining and sewn on by laid thread couching. The most important conservation requirements are for the results to be aesthetically attractive and to provide good support.

Standard weave silk and woollen fabrics were aged and then conserved using the above method. Five different mechanical testing procedures were then performed corresponding to the different stresses a costume might suffer: abrasion resistance, flexometer abrasion, flexometer bending, shear stiffness and tensile strength. The methods for abrasion resistance and tensile strength testing proved to be suitable to allow a comparison between different methods of support. Firmer significant conclusions, however, require a larger corpus of material.

Keywords: support, costume, aged, evaluation, silk, wool

Introduction

Knowledge of the advantages and disadvantages of different methods of conservation is vital if we are to preserve our textile treasures and enable them to be exhibited to the general public. The following pilot studies to develop techniques for evaluating different conservation methods were therefore undertaken by the Royal Armoury, Stockholm, in the spring of 2004.

The Royal Armoury is located at Stockholm Royal Palace. In addition to armour, weapons and carriages, its collections also include a unique array of costumes and its conservation workshop has been active since the 1890s. The Royal Armoury has more than 7,000 costumes in its collections, the oldest of them being the coronation cloak made for Erik XIV in 1561. This has been complemented over the centuries by costumes commemorating historic events, people and changing fashions among Swedish kings and queens and their courtiers. Extensive conservation of historical costumes is carried out at the Royal Armoury, enabling wide-ranging research on conservation procedures.

What is textile conservation?

Basically, textile conservation can be divided into two kinds: preventive and interventive. Interventive conservation denotes

the measures taken when damage has already occurred, e.g. cleaning, stabilisation and mounting. Stabilisation, which is the prime concern of this project, means giving new support and/or protection to brittle fabrics.

Costumes differ from many other textile objects because they are three-dimensional and most often have several layers of fabric. The conservator's task includes deciding on the most appropriate choice of conservation method. There are many different types of support but the following are just some examples of those used in the collections of the Royal Armoury:

- Gustavus Adolphus's two 1627 costumes: these exemplify two conservation methods: in one instance the fragmentary traces of silk satin in the sleeves were sewn onto silk crepeline with a running stitch; in the other instance the entire surface was covered with a new silk fabric, resembling that of the original.
- The Persian coat presented to Queen Christina at the beginning of the 17th century: this has been repeatedly conserved. Silk crepeline, for example, has been sewn on to protect the ends of the sleeves and the edging round the neck
- A pair of breeches from mid-1600, formerly belonging to Karl X Gustaf: these were completely unpicked after which a support fabric was inserted beneath the entire surface and sewn down with laid thread couching.

Gustav III's wedding robes from 1766: yet another support method was used for these robes. Each silver thread was sewn down with running stitch onto a new fabric, work that took more than a thousand hours to complete (Nilsson and Enhörning 2003).

Scientific studies of support methods for costumes using stitched conservation are difficult to find. A literature search in the bibliographic database of the Information Network (BCIN) and the Union Catalogue of Swedish Libraries (Libris) using terms such as 'costumes', 'evaluation', 'conservation', 'textiles' and 'support' did not reveal a single study or evaluation of costume conservation support methods. The latest book published in Sweden on the subject of conservation is a survey of all the materials used by the Pietas Society, now the Textile Conservation Department of the National Heritage Board in Stockholm, between 1908 and 1999; conservation methods, on the other hand, are not inventoried (Lundwall 2003). Particular methods, such as experimentally ageing materials used in conservation, have been described (Feller 1994) and have a bearing on the construction of tests where artificial ageing forms a part. Brooks et al. (1995) describe, among other things, various support methods and the process of choosing between them. They also point to the importance of describing methods at greater length and explaining why particular methods have been employed. Landi's Textile Conservators' Manual contains detailed descriptions of different types of stitch that can be used in textile conservation, suggests various methods and describes their advantages and disadvantages (Landi 1992), while case studies of treated costumes are found easily (Finch and Putnam 1985) but without any scientific analysis.

Anyone proposing to research costume conservation methods must begin by answering a number of basic questions:

- What method or methods of conservation are commonly used today and is there any new method which shows promise? The relevance of this research depends on whether it concentrates on a method or methods which are common or especially promising.
- What do experts require of costume conservation?
 Without such knowledge, research findings cannot be evaluated with certainty.
- What factors determine whether a costume is to be conserved? As a first step a questionnaire was constructed and sent to conservators presumed to have had experience of costume conservation at institutions in Sweden, Europe and North America.

A good methodology for evaluating costume conservation treatments probably demands a broad approach whereby examples of treated costumes are studied and experimental methods are tested. Among the former, for example, systematic assessments of previous costume conservations can offer detailed information on the advantages and disadvantages of different methods. Experiments can furnish direct proof that one conservation method can produce better results than others or even no conservation at all.

Also lacking at present is basic knowledge concerning the experimental methodology, which may be relevant in this context. The second part of this study therefore addresses

experimental ageing of materials and methods for testing the properties of the aged textiles with or without conservation. An initial comparison is also made between an aged material using support conservation and one without support conservation. Thus the exploratory part of the study is concerned primarily with basic problems encountered in an experimental approach.

The questionnaire

Aim

The aim of the questionnaire study was to investigate which support methods have been (and are now being) used internationally, which methods are the most common, what requirements are defined for costume conservation and what decides whether or not a costume is to be conserved in the first place.

Method

A questionnaire was sent to 51 conservation workshops in several countries; of these, 32 workshops (16 in Sweden, 15 in other European countries and 1 in North America) contributed replies. A further eight workshops replied that they did not have any textile conservation activities or that the conservator was newly appointed, while 11 did not reply at all. The conservators approached in Sweden received the questionnaire by mail and were informed that a follow-up telephone interview would take place a fortnight later. The telephone interviews were designed to maximise the response rate and to elicit as extensive replies as possible. The response rate was high and several of the workshops interviewed returned with additional information. To avoid language problems, the foreign conservators were asked to reply by letter.

An exact number of costumes conserved was not expected but instead the conservators were asked to estimate the figure to the best of their ability. A study was made of all conservation reports in the Royal Armoury, however, resulting in exact figures.

Findings

On the strength of the replies to the questionnaire, the methods of support can be divided into six groups (later referred to by number):

- A support fabric is inserted between an outer fabric and lining and sewn down.
- 2 A new fabric is used to cover a brittle outer fabric and is sewn down at the edges.
- 3 A support fabric is inserted behind an outer fabric and joined with an adhesive.
- 4 A combination of the above methods is used.
- 5 The sandwich method is used, i.e. brittle material is sandwiched between two layers of fabric.

6 A paper or fabric is loosely inserted between outer fabric and lining.

There are numerous combinations of these groups of support methods, various materials and sewing techniques. Most responses fell into groups 1, 2 and 4. If all variants of conservation methods and support methods are included, a total of 51 were reported.

The most common result is the insertion of a support fabric between the outer fabric and lining which is sewn on by couching over the outer fabric and through the support fabric but not through the lining. This method belongs to group 1. No promising new method was reported by any of the respondents. The most common criteria of a successful conservation are shown in Table 1.

Table 1 Important criteria of a successful conservation.

	No. workshops
Criterion	stating the
	criterion
Aesthetically appealing	18
Good, durable support, preventing further damage	16
Conservation with a minimum of interference	9
Ethical, reversible and invisible	9
Easily executed	6

The most frequent reason given for conservation treatment in the first place was an approaching exhibition, followed by the costume being in need of conservation. In answer to the question as to how a conservation decision is taken, in the main conservators decide for themselves how the costume is to be conserved; the second most common answer was a joint decision taken by the conservator and senior curator. There was full consensus here between the Swedish and foreign replies.

The number of costumes conserved by the workshops ranges from 1 to 13,000. The lower figure refers to a private workshop in Sweden, the highest to the Hermitage in St Petersburg. Several workshops found themselves unable to estimate the number of costumes they had conserved, but the most common figure was between 100 and 200.

The experimental study

Aim

The aim of this part of the study was to find one or more mechanical testing methods that could simulate 'museum handling' with satisfactory sensitivity. The expression 'museum handling' refers here to the wear and strain that a costume in a museum has to endure, for example, being taken in and out of storage or a showcase, packing for transit, during transport and while dressing and undressing mannequins for photographing or exhibitions. A second aim was tentatively to compare aged materials with and without conservation treatment. A method was considered as having a satisfactory sensitivity when it could differentiate between aged and unaged materials.

Method

General procedure

This study is a preliminary investigation. As economical resources for the project were limited, the decision was taken to test only one support method but to examine several test methods, evaluating two different materials: silk and wool. On the basis of the questionnaire replies, the most common support method (group 1 above) was selected for use in the tests

Together with personnel from IFP Research,¹ the author discussed the handling of museum costume items and various experimental methods that are designed to match the wear of fabrics and assess their qualities. Five methods were identified which had experimental protocol with some similarities to the handling to which museum costumes are subjected.

Before these could be tested, materials had to be artificially aged in order to better represent historic museum objects. It was decided to age the materials to about 20% residual strength which, according to IFP Research, is the minimum that will still allow processing by the five methods without failure. The ageing and subsequent testing were carried out by IFP Research.

Materials tested

Silk and wool were chosen for testing as they are the most common materials found in the Royal Armoury's costumes. The test and reference materials used were standard weaves, fabric with an even quality (for silk ISO 105-F06:2000 was used, and ISO 105-F01:2001 for wool).

Accelerated ageing of the test material

Conditioning and testing climate: 20 ± 2 °C and $65 \pm 3\%$ RH.² The accelerated ageing was carried out by means of UV exposure (as per ASTM G 53-96) in 8 hours light/4 hours dark cycles at a temperature of 50 ± 2 °C. The equipment used was a Q-UV Accelerated Weather Tester with a UVB 313 lamp. To determine a suitable ageing time, the material was aged for 1, 2, 3, 4, 7 and 14 days. The residual strength of each sample was then measured with the aid of tear resistance (as per EN-ISO 13937-2:2000).

It was decided to age the samples as much as the experimental handling would permit, i.e. to about 20% residual strength. The silk fabric was aged for 4 days, resulting in 22%, and the woollen fabric for 7 days, resulting in 23% residual strength.

Conservation measures

After the samples had been aged, some of them were conserved by inserting an unaged standard weave of silk or wool beneath the aged fabric, and attaching it by couching with double single-ply silk. The long stitches were sewn at 5 mm intervals and cross-stitches placed over them at approximately 4 mm intervals, the cross-stitches being about 2 mm wide.³

Testing methods and their evaluation

The methods had to model the wear and stress caused by handling of museum items. The results of five test methods imitating aspects of museum handling were analyzed with respect to their sensitivity. A test was accepted as suitable only if it differentiated between aged and unaged material.

1 Abrasion by the Martindale method (as per SS-EN ISO 12947-2:1999)

The abrasion resistance of the material is measured by abrading the test surface against a rather coarse woollen fabric. The method was chosen as imitating the abrasive wear of the costume surface that occurs while handling. Twelve samples were tested: three unaged and three aged samples of both silk and wool. Abrasion pressure: 9 kPa. Testing interval: silk 100 turns and wool 500 turns. End point assessment method: two thread ruptures. Diameter of test piece: 40 mm. Diameter of abrasion area: 28 mm.⁴

Findings

There is a great difference between unaged and aged samples: the aged samples are much weaker, wool showing the biggest difference. The test has satisfactory sensitivity (see Table 2).

2 Flexometer abrasion (as per ASTM D 6182, DIN draft 53351)

This method involves folding, bending and securing the sample and repeatedly flexing it. The method imitates the folding and bending that cannot be avoided in museum handling. Six samples were tested: one unaged sample of both silk and wool and two aged samples of both silk and wool. Exposed/conserved material was tested with the aged side folded either inwards or outwards. The tests were performed in the warp direction of the material. Inspection interval: 50,000 cycles. No. of cycles: 400,000. A test round of 250,000 cycles was run first, with the aged side of the test pieces folded in different directions. This was followed by a new round of 400,000 cycles with the aged side folded outwards. Size of test area: $70 \times 45 \text{ mm}.^5$

Findings

There were no ruptures at all in unaged samples, nor were there any in aged wool after 400,000 cycles. One rupture occurred in the aged silk after 250,000 cycles but this was regarded as accidental because no further ruptures occurred when continuing to 400,000 cycles. The results were not analyzed further. The sensitivity of the test is deemed unsatisfactory.

3 Flexometer bending (as per DIN 53 359:1957, 100,000 folds)

The ends of the samples are mounted and then moved towards each other in order to fold up the sample. This test is a variant of the preceding test. Three samples were tested on both

silk and wool but only on aged material. Size of test area: 20 \times 40 mm. 6

Findings

Aged silk and wool were undamaged after 100,000 folds. The results were not analyzed further. The sensitivity of the test is unsatisfactory.

4 Shear stiffness (as per Kawabata SS-EN 13780:2003)

With this method, the sample is fastened at both ends and while one end remains static the other end moves forward by 8° . This kind of motion can, for instance, occur when dressing a mannequin. Since this test was considered doubtful, only the more susceptible silk was tested. Three samples were tested at a shear angle of 8° . Size of test area: 40×50 mm.⁷

Findings

No differences emerged between unaged and aged silk. Increased load did not produce any additional effect. The sensitivity of the test is unsatisfactory.

5 Tensile strength testing (as per SS-EN ISO 1394-2:1999, grab test)

Rupture strength indicates the strength of a fabric by determining the force required to break it. Rupture due to incautious handling might occur in museum objects, but the method also has the character of a general strength test. Three unaged samples of both silk and wool were tested. Of the aged samples, two were silk and three were wool. Gauge length: 50 mm. Size of test piece: 100×200 mm approx.⁸

Findings

The tests showed clear differences between unaged and aged samples. The test is considered to be suitable (see Table 3).

Only two test methods had satisfactory sensitivity and were suitable for comparisons between conserved and unconserved aged material: no. 1, abrasion by the Martindale method and no. 5, tensile strength. In test no. 1 (abrasion), the total number of samples was 24 of which 12 were conserved. Of these, three silk and three wool samples had couching, diameter 20 mm, over the middle area of the sample, and the other three silk and three wool samples had couching over the entire sample. (Diameter of the sample: 40 mm. Diameter of abrasion area: 28 mm.)

In test no. 5 (testing tensile strength), the total number of samples was 19, of which two silk and two wool samples were conserved with the long stitch of the couching following the warp direction, and two silk and two wool samples were conserved with the long stitch of the couching following the weft direction. (Conserved area: 80×110 mm. Gauge length: 50 mm. Size of sample: 100×200 mm.)

Results

1 Abrasion by the Martindale method

For silk there are only small differences between samples not conserved and those conserved, and between the samples conserved with the long stitches in different directions. For wool, the conserved samples are weaker than the samples not conserved, while the sample with couching over the entire area achieves better results than couching only over part of the area (see Table 2).

5 Tensile strength testing

The readings showed the conserved silk and wool samples to be more than three times stronger than aged material without conservation, and roughly equal in strength to new material. The direction in which the couching is sewn makes a difference for silk but not for wool. When the long stitch follows

 ${\bf Table~2}~{\bf Abrasion~resistance:~number~of~turns~before~two~thread~ruptures.}$

	Abrasion resistance				
Sample	Control	Aged without	Aged and	Aged and	
	Control	conservation	conserved A	conserved B	
Silk					
1	2,900	300	200*	200*	
2	3,100	300	300*	300*	
3	2,900	300	200*	300*	
Mean	2,967	300	233	267	
Wool					
1	26,000	1,500	500*	500*	
2	21,000	1,000	500*	1,000*	
3	22,500	1,500	500*	1,000*	
Mean	23,167	1,333	500	833	

Control = unaged material

A = couching over the middle 20 mm diameter of the sample

Table 3 Tensile strength results

	Tensile strength, N				
Sample	Control	Aged without	Conserved	Conserved	
		conservation	lengthwise ***	transversely ****	
Silk 1	348.6	109.8	366.8*	327.6**	
Silk 2	371.8	106.5	376.1*	344.6**	
Silk 3	360.7	_	-	-	
Mean	360	108	371*	336**	
Wool 1	163.8	58.3	217.5*	199.0**	
Wool 2	169.8	53.8	189.5*	193.4**	
Wool 3	169.8	62.9	-	_	
Mean	168	58	204*	196**	

Control = unaged material

the warp direction and the short one the weft direction, the conservation is somewhat stronger than when the long stitch follows the weft direction (see Table 3).

Discussion

According to the answers to the questionnaire, the most common support method, used both today and in the past, is group 1. The most important requirements are that the conservation should be aesthetically attractive and provide good support in order to prevent damage.

The considerations determining the choice of support method have not been investigated in this study. The choice of support method can, for example, hinge on the type of damage to be repaired. Possibly it may also depend on conservators' particular criteria for successful conservation and on their differing opinions as to which support methods fulfil those criteria.

The aim of our experiments was to find one or more methods capable of imitating the wear to which a textile object is subjected during museum handling. The experiments were also intended to provide some indication as to whether or not the most common support method achieved its purpose.

The materials chosen for testing were silk and wool. A residual strength of 20% was considered to be the minimum value that permitted the aged material to be mounted and handled for the different tests without failing (although the fabric of the costumes conserved may presumably be stronger or weaker than this). The results of this study suggest positive effects of conservation in objects with 20% residual strength, however, conservation support may not add to the longevity of less worn textiles. In the author's experience, many costumes in the Royal Armoury's collections in Stockholm have far weaker fabrics than the aged models.

The five test methods concerned abrasion resistance, flexometer abrasion, flexometer bending, shear stiffness and tensile strength. Only the first and the last methods were sensitive enough, differentiating between aged and unaged material. These two methods concern the abrading of the test surface against another fabric and the general strength of the sample, respectively. Effects of folding and twisting could not be tested adequately.

The abrasion findings, which suggest the conserved wool samples to be slightly weaker than those not conserved, may possibly be due to the former having been subjected to a greater degree of handling. There is also the possibility that couching contributes to the weakening of the fabric by augmenting friction against the abrading fabric. The measurements obtained suggest that the wool samples conserved over a large area are somewhat stronger than those where a smaller area has been conserved. The cause may be that the larger area permits sewing into an area that has been protected during ageing. It could be worthwhile investigating whether the size of the conserved area is of any consequence. Conservators are often faced with deciding whether to insert a support fabric beneath a complete area (full support) or only part (patched support). It is generally recommended that stitching should continue into stable areas.

B = couching over the entire sample

^{*} Burls appear on the 'reinforcing threads' (IFP Research 2004a)

^{*} Weave and reinforcing thread rupture more or less simultaneously

^{**} Weave ruptures before the reinforcing thread

^{***} The long stitch of the couching follows the warp direction

 $[\]ensuremath{^{\circ\circ\circ\circ}}$ The long stitch of the couching follows the weft direction (IFP Research 2004a)

Abrasion is judged to be a viable way of testing different support methods including those whereby the outermost fabric of a costume is covered with different materials (the second most common method used, group 2). This method could be used to assess whether or not the underlying area is protected.

Flexural abrasion, flexural folding and shearing were tried as testing methods because they include movements which bear a close resemblance to museum handling. The flexural abrasion trial was not sensitive, however. The samples in these tests were very small due to the limitations of the ageing acceleration equipment. Ageing of bigger samples or samples with lower residual strength, or ageing of the test pieces on both sides, may possibly yield more successful findings. The same applies for the flexural folding and shearing methods. These methods should be further investigated since they may be important supplements to abrasion and rupture as simulations of museum handling.

Tensile strength testing indicated that conserved samples of both silk and wool are more than three times stronger than the aged materials without conservation. The combined strength of the original and support fabrics, however, will make the aged-and-conserved composite stronger than the new material, which consists of one fabric layer only. Depending on the way the costume is handled and the stress it experiences, it may be more appropriate to clamp the aged fabric alone during further testing. In this tensile strength test, the direction of the long couching stitch in silk had significance, although normally when treating an object there is no choice in this – the long stitches are always laid over the loose threads on a fabric, mostly the weft because it is weaker. In other words, the long stitches are most commonly laid in the warp direction.

The preliminary results of the comparison between unconserved and conserved material are interesting. They suggest that both positive and negative effects are obtained by the support conservation method, that the execution of the sewing can make a difference and that the size of the conserved area is significant. The exploratory nature of this study must be emphasised, however. The comparisons ought to be tested statistically which was not possible in the present study because the number of test pieces was limited by financial constraints.

A good methodology for evaluating costume conservation treatments probably demands a broad approach whereby examples of treated costumes are studied and experimental methods are tested. Among the former, for example, systematic assessments of previous costume conservations can provide detailed information on the advantages and disadvantages of different methods. Experiments can furnish direct proof of whether one conservation method produces better results than others or no conservation at all.

Conclusions

The questions concerning the most commonly used support methods and what constitutes successful conservation were answered with the help of a questionnaire. The preferred method is to insert a new fabric between the outer fabric and lining and sew on by couching over the outer fabric and through the support fabric but not through the lining. When assessing the successful outcome of the treatment, an enhanced support secondary to the finished look was among the factors listed. The criteria dictating the decision on treatment options are still to be elucidated, and it remains to be determined when a particular approach should be taken.

Of the five methods tested, two tests on abrasion resistance and textile strength were deemed suitable for evaluating support methods when used in the conservation of fabrics with about 20% residual strength. This was based on tests for just one support method, however, and the suitability for others needs to be confirmed. Effects of folding, twisting and vibrations are presumably important in museum handling, and methods for the study of such effects should be developed. Methods permitting larger samples to be tested might be worth considering. All these tests must also be complemented with investigations of conserved historical costumes in order to broaden the perspective. Once the range of support methods has been assessed by an appropriate variety of test methods it will then be possible to make considered recommendations.

Acknowledgements

Thanks are due to my employer, the Gunvor and Josef Anér Foundation, the Agnes Geijer Fund Foundation and the Royal Patriotic Society for enabling this study. I would also like to thank all colleagues from the various institutions for taking the trouble to reply so helpfully to the questionnaire. I am grateful to Margit Forsberg (student) for the conservation of test samples, to Professor Carl-Otto Jonsson for vetting my text, to IFP Research and to conservator Emma Hocker for checking the English.

Notes

- IFP Research (Mölndal, Sweden) is an independent agency serving the international industry in the areas of fibrous, composite and polymer materials.
- 2. IFP Research 2004a.
- 3. A no. 10 Modiste needle was used for the sewing.
- 4. IFP Research 2004a
- 5. Ibid.
- 6. Ibid.
- 7. Ibid.
- 8. Ibid.

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Suppliers

Standards ASTM G 53-96, EN-ISO 13937-2:2000, SS-EN ISO 12947-2:1999, ASTM D 6182 (DIN draft 53351), DIN 53 359: 1957, SS-EN ISO 1394-2:1999, SS-EN 13780:2003 and standard weaves ISO 105-

F06:2000, ISO 105-F01:2001: available from SIS, Swedish Standards Institute, St Paulsgatan 6, 118 80 Stockholm, Sweden (www.sis.se).

The author

Johanna Nilsson has been a textile conservator with the Royal Armoury, Skokloster Castle and the Hallwyl Museum since 1990, and since 2000 has been principally concerned with the care of the Royal Armoury textile collections. She is currently involved in the Royal Armoury's Christina project, the aim of which is to devise a nitrogen-filled showcase for the storage of Queen Christina's funeral robes. Johanna teaches cleaning methods and costume conservation to prospective textile conservators at Göteborg University.

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Paper II

Johanna Nilsson, Francisco Vilaplana, Sigbritt Karlsson, Jonny Bjurman and Tommy Iversen. "The validation of artificial ageing methods for silk textiles using markers for chemical and physical properties of seventeenth century silk." *Studies in Conservation*, 2010, 55 (1) p. 55-65.

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Error list

P. 56-57, correct RH values for the samples aged in 60°C with: Mg(NO₃)2 is uncertain and was approximately 42.5 instead of 53%; NaCl was 74.5±0.30% instead of 75%; and, KCl was 80.25±0.41% instead of 86%.

The Validation of Artificial Ageing Methods for Silk Textiles Using Markers for Chemical and Physical Properties of Seventeenth-Century Silk

Johanna Nilsson, Francisco Vilaplana, Sigbritt Karlsson, Jonny Bjurman and Tommy Iversen

The Royal Armoury in Stockholm has conducted a project to experimentally evaluate conservation methods used for historic costumes. As the historic value of authentic artefacts precludes their use in experimental work, artificially aged standard silk needs to be used as a substitute. This study aimed to find a suitable artificial ageing method for standard silk resulting in a degradation state that simulated that of silk from seventeenth-century costumes. Four artificial ageing methods were studied: (1) thermal oxidation in dry air, (2) exposure to different relative humidity (RH), (3) immersion in solutions of varied pH, (4) accelerated ultraviolet (UV) exposure at 50 ± 2 °C, 95% RH. Different chemical and physical properties for silk were evaluated using Fourier transform infrared spectroscopy, size exclusion chromatography and tensile tests, which were employed as analytical indicators for comparison between the artificially aged silk and samples from seventeenth-century costumes. Of the ageing methods tested in this study, thermo-oxidation at 125°C in dry air for 28–56 days produced silk with properties most like those of historic silk samples.

INTRODUCTION

The Royal Armoury in Stockholm has a large collection of costumes dating back to the start of the seventeenth century that is unique in quantity and quality. A common material is silk and although some of the costumes have been conserved, most of them are still in need of conservation. Understanding the advantages and disadvantages of different conservation methods is vitally important to preserve historic textiles and to allow their public exhibition. For this reason the Royal Armoury initiated a project to evaluate support methods used in textile conservation, i.e. when a new fabric is inserted under or laid on top of a fragile fabric and attached by stitches or glue [1]. The historical value of the authentic artefacts precluded their use in experimental work, so artificially aged standard silk needed to be used as a substitute in order to evaluate the various conservation methods. Degradation of silk textiles has been the subject of several recent studies [2-5]. A wide range of environmental factors can cause damage to silk fabrics: light, heat, oxygen, chemical agents, humidity, low and high pH, and mechanical stress and wear. The main chemical mechanisms involved in silk degradation are hydrolysis and oxidation, which are responsible for changes in the physical and chemical properties of silk.

AIM OF THE STUDY

The aim of this study was to lay a scientific foundation for proposing an optimal accelerated ageing procedure, accurately simulating the degradation of naturally aged silk from the seventeenth century. The development of such a method was intended to allow the preparation of suitable substitute sample specimens, to be used in the place of historic costumes in tests to evaluate conservation methods. Chemical and physical analytical indicators for silk degradation were identified which allowed comparison of the type and extent of degradation in artificially and naturally aged silk.

Received March 2009

EXPERIMENTAL PROCEDURES

Standard silk samples were artificially aged by exposure to different environmental conditions: thermo-oxidation (temperature and oxygen), relative humidity (RH), accelerated ultraviolet (UV) exposure, and immersion in solutions of varied pH. To validate these methods against the properties of seventeenth-century silk fabric, attenuated total reflection—Fourier transform infrared spectroscopy (ATR-FTIR), size exclusion chromatography (SEC) and tensile tests were used to compare the chemical and physical analytical markers of historic silk samples with those of artificially aged samples.

Samples for accelerated ageing

Standard silk fabric ISO 105-F06:2000 *Bombyx mori* was used for artificial ageing. A full description is provided in the Appendix. For thermo-oxidation, varied pH, and relative humidity ageing, sample pieces consisting of 50 mm warp × 40 mm weft threads were used. These were conditioned in desiccators containing silica gel [6] for 24 hours and then mounted in sealed vials prepared to represent different environmental conditions. A silk thread was attached to the short end of each sample, and inserted between the lid and vial (Figure 1).

For ageing with the accelerated UV exposure, a 75 mm warp \times 150 mm weft sample was used. A 65 mm \times 95 mm section was exposed on one side of each sample.

Historic costume sample

To avoid dyes interfering with the chemical analyses, and to facilitate the comparison of historic samples with the white standard silk, only white historic silk costumes were selected for the analyses, even though the preparation of the white historic silk differs in several ways from modern standard silk. The white silk costumes in the Royal Armoury's collections have mostly been used for coronations and weddings, which means that they probably have been used only once, but they have, of course, been exhibited on several occasions as being important and precious objects. Cutting samples from original items should be avoided, but in this case it was necessary for the validation: sampling was therefore restricted to three objects. For the tensile strength tests, threads were taken only from the weft of the weave. Eight 40 mm threads were taken from the doublet and the breeches of the coronation costume (inv 25605-6) of King Gustav II Adolf (GIIA) dating from 1617 (Figure 2), and from the coronation cloak (inv 25599) of King

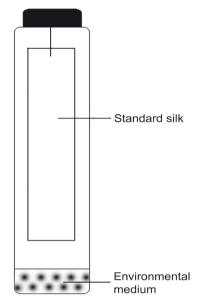


Figure 1 Vial with a silk sample.

Karl X Gustav (KXG) dating from 1654. The doublet's lining, which is tabby weave, has 70 warp threads per cm and 40 weft threads per cm. The outer fabric of the breeches, which is a satin weave, has approximately 190 warp threads per cm and 40–45 weft threads per cm. The cloak, also satin, has approximately 140 warp threads per cm and 66 weft threads per cm. All extracted threads had almost no twist and were taken from areas with open edges. For the ATR-FTIR and SEC analyses, specimens of approximately 5 mm² were cut from the seam allowance.

Design of accelerated ageing experiments

Standard silk samples were exposed to four sets of conditions:

- 1 Thermal oxidation in dry air at 125°C for 14, 21, 28, 35, 42, 49 and 56 days; in air with 53% RH controlled by saturated salt solutions at 25°C and 60°C for 21 and 28 days. The exposure was carried out in a forced ventilation oven, Memmert D06062 (Memmert GmbH, Germany) with temperature control (± 1°C).
- 2 Relative humidity of 0, 53, 75, 86 and 100% controlled by saturated salt solutions (except for



Figure 2 Coronation costume of King Gustav II Adolf from 1617. Photo: Göran Schmidt, The Royal Armoury, Stockholm.

0% RH where dry salt was used and for 100% RH where distilled water was used) at 25°C and 60°C, for 21 and 28 days. To obtain specific RH environments in the vials, a mixture of different salts $(0.5 \pm 0.1 \text{ g})$ and distilled water (1 mL) was added to the vials before mounting the silk (see Table 1 in the Appendix). The samples were mounted in the vials so that they could hang inside freely and avoid touching the humidifying salt in the bottom.

- pH values of 1, 3, 7, 11 and 13 at 25°C and 60°C, for 21 or 28 days, obtained by immersing the samples in different analytical buffers (see Table 2 in the
- 4 Accelerated UV exposure equipment, peak irradiance at 313 nm and total irradiance approximately 0.5 W.m⁻² at 50 ± 2 °C and 95% RH for 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10 days. Five samples were exposed at each exposure time. The cycle employed was of 8 hours of

radiation followed by 4 hours of condensation. The exposure was carried out in a Q-U-V accelerated weather tester from O-Panel Lab. The exposures were carried out in the laboratories of IFP Research Ltd, Mölndal, Sweden [7].

After exposure, the samples were rinsed in deionized water in five baths to get rid of possible remains of the substances used to create the specific conditions, i.e. salts and buffers that otherwise could lead to a continuation of the degradation process. Finally the samples were dried at room temperature in a covered glass box (80 dm³) for 24 hours and put in a desiccator with silica gel for 24 hours before ATR-FTIR and SEC analyses.

METHODS FOR DAMAGE ASSESSMENT

ATR-FTIR

The infrared spectra of the silk textile samples were recorded using an ATR-FTIR spectrometer equipped with a single-reflection accessory for attenuated total reflection measurements. The ATR-FTIR spectrometer used was a Spectrum 2000 from Perkin Elmer (Wellesley, MA) equipped with a Golden Gate single-reflection accessory. Each spectrum was obtained by performing 24 scans between 4000 and 600 cm⁻¹ at 1 cm⁻¹ intervals with a resolution of 4 cm⁻¹ under a consistent pressure. Each sample was analysed in triplicate. The silk sample size and the fact that the threads in the weave have two directions consisting of many fibres gives a randomized signal and the polarization effect can be ignored.

Size exclusion chromatography

Relative measurements of the molecular weight distributions of artificially aged and historic silk samples were carried out using size exclusion chromatography with N,N-dimethylacetamide (DMAc) and 0.5% lithium chloride (LiCl) as the mobile phase and further calibration with pullulan standards. The size exclusion chromatography equipment consisted of a DGU-20A3 degasser, a LC 20AD liquid chromatograph, a CT-20A column oven, a Rheodyne 7725i fixed-loop 100 μL injector system, and a RID-10A refractive index detector, all provided by Shimadzu. Approximately 8 mg of silk was washed with deionized water for 2 hours under agitation. The silk was filtered and washed three times with methanol (3 × 30 minutes) under agitation and filtered after each wash. The solvent was changed to N,N-dimethylacetamide (DMAc) and the silk was

washed again three times (3 × 30 minutes) under agitation, and filtered after each wash. After the washing process, 1 mL DMAc (8% w/w LiCl) was added to the silk and left under agitation at room temperature until the silk was completely dissolved. The silk solution was diluted with 15 mL DMAc to achieve sample injection conditions: 0.5 mg silk per mL DMAc (0.5% w/w LiCl).

For historic silk samples, a similar procedure was employed but the sample size was reduced to 3 mg and the volumes used to achieve injection conditions were adapted. The solutions were filtered with HPLC Teflon® filters (pore size 0.45 μ m) and stored until further analysis. The dissolved sample was injected, and the separation was performed through a PL gel 20 mm Mixed-A column equilibrated at 40°C, using DMAc (0.5% w/w LiCl) as a mobile phase with a flow of 0.5 mL per minute. Molecular weight distributions were calculated by comparison with pullulan standards with molecular weights of 0.32, 1.3, 6, 12, 22, 50, 110, 200, 400 and 800 kDa.

Tensile tests

The tensile testing apparatus performed an automatic test procedure, set with a gauge length of 20 mm, a tension weight of 2000 mg, and an extension rate of 20 mm per minute. The tensile testing apparatus used was Vibrodyn® CRE-type according to DIN EN ISO 5079, ASTM D 3822, BISFA, AFNOR G 07-008. The linear density of thread is a measurement of its thickness and is expressed in tex units, which is the weight in grams of 1000 m threads. The tex value was calculated by removing five to eight threads from the weft of each sample and measuring the length and weight of each thread and calculating the average

Five threads from each ageing method were tested by measuring tenacity, extension at break, and initial modulus, using yarns of historic textiles and standard silk, both artificially aged and unaged. The modulus, a measurement of thread stiffness [8], was measured as the initial stiffness calculated from the stress–strain curve. This number of threads was enough for a statistically significant result.

RESULTS AND DISCUSSION

ATR-FTIR

The surface of artificially aged and historic silk samples was characterized using ATR-FTIR in order to monitor chemical and structural changes due to ageing. Figure

3 shows the general spectra of historical samples and artificially aged silk samples subjected to different environmental conditions. The ATR-FTIR spectra show a similar pattern for all analysed samples, with peaks attributed to amide vibration groups: the main Amide I band between 1610 and 1700 cm⁻¹; the Amide II peak between 1490 and 1560 cm⁻¹; and the Amide III region between 1180 and 1280 cm⁻¹ [9]. Other major bands in the FTIR spectra of silk are attributed to C-H bending (between 1300 and 1460 cm⁻¹), and to the skeletal stretching region (between 900 and 1100 cm⁻¹) [10]. The Amide I band is sensitive to the secondary structure of proteins and is usually used for protein conformational investigations [9]. For the silk samples studied here, the Amide I band exhibits a clear peak centred at 1615 cm⁻¹, with a small shoulder at 1690 cm⁻¹: both are related to the \beta-sheet secondary structure of silk fibroin, which confers silk fibres with their crystalline structure.

An interesting result from this degradation study is that accelerated ageing and natural ageing do not alter the typical β-sheet conformation of silk, since the main Amide I peak remains centred at around 1615 cm⁻¹ for both artificially aged and historic silk samples. Although the β-sheet conformation remained unchanged, interesting changes in regions in the FTIR spectra were observed in artificially aged samples and in historic silk. Figure 4 shows the magnification of two IR regions: the 1700-1775 cm⁻¹ region, associated with the vibration of carbonyl (C=O) groups [11] and the two bands centred at 828 and 850 cm⁻¹, related to tyrosine residues in silk [12]. Formation of carbonyl groups is a common signature of oxidation processes in natural and synthetic macromolecules. These results show that exposure to accelerated UV exposure markedly increases the absorption in the carbonyl region, an effect enhanced by prolonged exposure. A less dramatic effect was observed in silk samples subjected to thermooxidation at 125°C for different time periods. However, thermo-oxidative exposure at 25°C and 60°C do not exhibit bands in the carbonyl region, at least not for the time span used in the accelerated ageing experiments. Exposure to high relative humidity and high pH (pH 11 and pH 13) did not result in a modification of the carbonyl region. Conversely, samples immersed in low pH buffer environments (pH 1 and pH 3) showed a slight increase in the carbonyl absorbance. These changes in the carbonyl region were also observed in silk samples from the seventeenth century, resulting in the increase in broad absorption shoulders in that region.

For the tyrosine doublet bands, a slight decrease in their intensity was observed for historical samples as

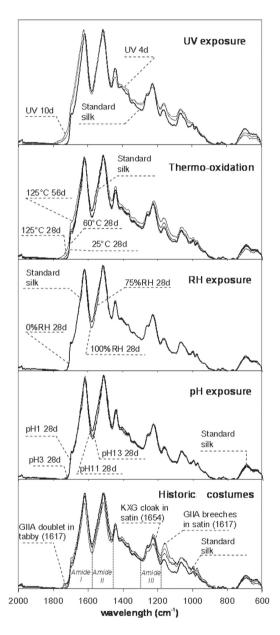


Figure 3 ATR-FTIR spectra of artificially aged silk and historic samples. Ageing with accelerated UV exposure is abbreviated UV.

compared to unaged standard silk. A similar decrease in intensity was detected for artificially aged silk subjected to high alkaline condition (pH 13) and to thermo-

oxidation in air at 125°C at increased exposure times. No alterations in these bands were observed for silk samples exposed to different RH conditions or low pHs. However, prolonged exposure with accelerated UV exposure resulted in a significant decrease in the tyrosine groups; this process could be related to the UV-induced oxidation of tyrosine units present in the amorphous regions of silk [13].

Size exclusion chromatography

Figure 5 shows the molecular weight distributions calculated against the pullulan standards for selected artificially aged silk samples and historic silk samples. A bimodal distribution was observed for standard silk, and was seen more clearly for the artificially aged and for the historic silk samples. This bimodal structure may be linked to the two chain size populations existing in the silk fibroin macromolecule (a heavy chain and a lighter chain) [14]. In general, the historic samples showed lower molecular weights than the standard material. This may be associated with the breakage of peptide bonds, probably induced by hydrolysis reactions. Size exclusion chromatography has been reported to reveal similar reductions in the molecular weight of silk in historic tapestries [15]. Exposure to different relative humidity environments did not substantially alter the molecular weight distribution of silk. Immersion in acidic (pH 1) and alkaline (pH 13) environments caused a reduction in the molecular weight. This effect was more noticeable in the alkaline environment, which is as expected for proteins [16]. UV radiation from the accelerated UV exposure may induce complex alterations in the chain structure of silk macromolecules. When dissolving the samples in DMAc (8% w/w LiCl), a slight suspension of silk was observed (in contrast to the clear solution obtained for the rest of the samples), together with an increased difficulty in completely filtering the solution through the HPLC Teflon® 0.45 µm pore size filter. These observations might indicate the presence of cross-linked silk chains induced by UV radiation, but this effect could not be proven with the experimental techniques employed. Finally, thermo-oxidation at high temperatures (125°C) also caused a decrease in molecular weight distribution. However, the differences in thermo-oxidative exposure time seemed to influence the shape of the bimodal distribution of the two chain constituents in silk fibroin. Broader molecular weight distributions were observed for samples exposed to harsher environments.

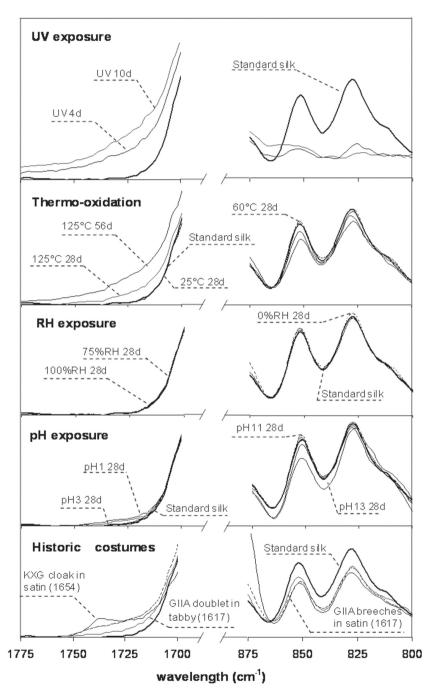


Figure 4 Magnification of two different IR regions.



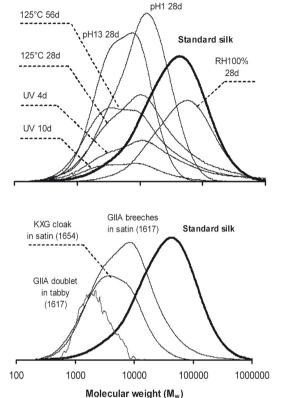


Figure 5 Results of SEC analysis of the molecular weight distribution of artificially aged and historic silk samples.

Tensile tests

Mechanical properties constitute the ultimate indicator of the degree of degradation in textiles and reflect the sum of the overall physical and chemical changes that the fibres have undergone during their lifetime. Tensile testing was performed to elucidate silk mechanical properties and Figure 6 shows the results of modulus (in cN.tex⁻¹), tenacity at break (in cN.tex⁻¹), and extension at break (in %) for standard silk, seventeenth-century silk and a selection of artificially aged silk samples subjected to the different environments studied.

The historic samples were shown to have lower modulus, tenacity and extension compared to unaged standard silk but the values of initial modulus are still quite high. The low values of tenacity and extension at break denote the increased fragility of the historic samples caused by natural ageing.

Artificial ageing resulted in different effects on the mechanical properties of standard silk. Relative humidity had little effect on tenacity and extension at break: modulus decreased slightly, because moisture absorption induces non-reversible dimensional changes in the silk fibres and can also modify their structure. With respect to acidity and basicity, low pH caused a slight decrease in the modulus, tenacity and extension, whereas exposure to alkaline conditions (pH 13) caused a total disruption of silk fibrillar structure, probably due to alkaline hydrolysis of peptide bonds in the protein chain and a total deterioration of its mechanical properties, precluding tensile testing of the exposed material.

The two other types of ageing conditions, thermal oxidation at 125°C in 0% RH and accelerated UV exposure, gave satisfactory results. In the tensile tests, these methods result in reductions in extension and tenacity at break that are comparable to the effects of

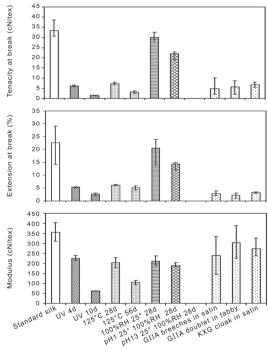


Figure 6 The results of modulus (in cN.tex-1), tenacity at break (in cN-tex-1) and extension at break (in %) for standard silk; seventeenthcentury silk; and a selection of artificially aged silk that has been subjected to extreme conditions of pH. RH and accelerated UV exposure.

the natural ageing factors. These ageing methods are also fairly easy to perform. For these reasons, statistical analyses of tensile tests were performed on these ageing methods.

Modulus, tenacity and extension are significantly reduced in standard silk subjected to both thermal oxidation and accelerated UV exposure at sufficient exposure times. Similarly, all the three historic samples show increased fragility in modulus, tenacity and extension. In comparison with historic samples, however, there are differences between, on one hand, tenacity and extension and, on the other hand, modulus, due to the relatively high modulus of the historic samples. Short exposure time results in insufficient reduction in tenacity and extension, but results in similar reduction in modulus as in the historic samples. Samples exposed to thermal oxidation or accelerated UV exposure at short exposure time have significantly higher tenacity and extension than historic samples, but are similar to the historic samples at longer exposure time. For modulus analysis quite another picture is given. At short exposure time, both thermal oxidation and accelerated UV exposure produce modulus values similar to those of the historic samples, whereas at longer exposure time modulus after experimental exposure is generally significantly lower. Difference between means were tested statistically using one-way ANOVA and Tukey honestly significant difference test (HSD), level of significance being set at 0.05.

Critical evaluation of artificially accelerated ageing procedures in comparison with seventeenth-century silk

This study has proposed and evaluated different environmental conditions for silk ageing, including thermooxidation (temperature and oxygen), relative humidity, accelerated UV exposure and pH. It has been shown that relative humidity alone does not induce chemical or physical changes in the standard silk with respect to the formation of carbonyl groups, the reduction of the molecular weight distribution, or the deterioration of the mechanical properties. On the other hand, it is well known that both low and high pH levels cause a total disruption of the macromolecular structure, thus resulting in deterioration of the mechanical properties. These effects, however, do not fully correspond to the analytical markers for historic costumes. Ageing with accelerated UV exposure and thermo-oxidation have been traditionally used for accelerated ageing purposes in synthetic and natural macromolecules, and especially in textile ageing studies. These methods have

previously been reported to induce different degradation mechanisms on silk chemical structure [17].

In the results presented here, the accelerated UV exposure was responsible for the appearance of carbonyl groups and for the total loss of the double tyrosine bands in the FTIR spectra, together with a considerable modification in the molecular weight distribution and a dramatic deterioration of mechanical properties (fragility and loss of stiffness). These striking changes were not observed in historic samples to the same extent, and may lead to the conclusion that accelerated UV exposure represents an excessively harsh accelerated ageing procedure for silk, in comparison with the natural ageing of historic samples. Finally, thermo-oxidation at moderate temperatures (60°C) during the studied exposure time barely affected the chemical and physical properties of standard silk fabrics. However, thermo-oxidation at higher temperatures (125°C in dry air) induced marked changes in both the chemical structure (e.g. formation of carbonyl groups, reduction in the tyrosine FTIR bands, and a production of shorter molecular weight fragments) and the mechanical properties of the artificially aged samples, similar to those of historic costumes.

CONCLUSIONS

Different accelerated ageing procedures under varying environmental conditions have been critically evaluated to mimic the degradation state of seventeenth-century historical silk costumes. Thermo-oxidation of standard modern silk samples at 125°C in dry air results in chemical and mechanical properties similar to those of the historic silk samples. Exposure to different relative humidity environments does not alone alter the physical and chemical properties of standard silk to the same extent as thermo-oxidation. Exposure to extreme acidic and alkaline pH conditions, however, causes substantial changes in the silk fibre properties, though dissimilar to the changes seen in historic samples. Accelerated UV exposure is a common method for artificial ageing in the textile industry, where UV radiation plays an important role, but its influence on the chemical and physical properties studied in this project does not completely correspond to the natural degradation observed in historic samples.

Therefore, controlling the exposure time to the thermo-oxidative environment will allow better reproduction of the type and extent of degradation seen in historic samples. This artificial ageing procedure may be used in the future at the Swedish Royal Armoury to test and develop conservation methods for historic textiles.

APPENDIX

Sampling

The standard material had an average weight of 60 g.m⁻² with 50 warp threads per cm and 37 weft threads per cm and a defined tone and grade of whiteness. To sample the standard material, at least 1 m of full fabric width was cut out from a roll, at least 3 m from the end. The samples were taken at random over the fabric surface area; no sample was taken closer than 150 mm from any edge, and folds and damaged areas were excluded. None of the samples was allowed to contain exactly the same number of warp or weft threads per cm.

Regimes for the fixed RH and pH

Details are given in Tables 1 and 2.

Table 1 Salts used for the preparation of the environments with different RH

Salt	RH (%)
Calcium chloride, CaCl ₂ , 99% purity	0
Magnesium nitrate, Mg(NO ₃) ₂ , 99% purity	53
Sodium chloride, NaCl, 99% purity	75
Potassium chloride, KCI, 99% purity	86

Table 2 Buffers used to achieve different pH

Buffer	рН
Hydrochloric acid/potassium chloride, HCI/KCI	1
Sodium citrate, Na ₃ C ₆ H ₅ O ₇	3
Sodium phosphate, Na, HPO,	7
Glycine/sodium hydroxide, NH,CH,COOH/NaOH	11
Glycine/sodium hydroxide/sodium chloride,	13
NH ₂ CH ₂ COOH/NaOH/NaCl	

SUPPLIERS

Salt and buffers: Sigma-Aldrich, USA.

Standard silk: Cromocol, Åsboholmsgatan 16, SE-504 51 Borås, Sweden.

Pullulan standards: Sigma-Aldrich, Germany.

ACKNOWLEDGEMENTS

The authors are grateful for the support from the following: The Barbro Osher Pro Suecia Foundation, The Royal Swedish Academy of Letters, History and

Antiquities-Agnes Geijers Foundation, The Sibling Bothéns Foundation, Gunvor and Josef Anérs Foundation and The Royal Patriotic Society, Thanks go to Daniel Limones Herrero (student) for his work with the samples and to Professor Roshan Shishoo for supervising. The authors are also very grateful to The Royal Armoury in Stockholm for making it possible to run the project and to use the collection of the museum in the study. Thanks also go to Steven Muir for checking the English.

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Résumé — L'Armurerie royale de Stockholm a mené à bien un projet d'évaluation expérimentale des méthodes de conservation utilisées pour les costumes historiques. Comme la valeur historique des objets authentiques exclut leur utilisation à des fins expérimentales, des soies standard vieillies artificiellement doivent être utilisées à la place. La présente étude visait à trouver une

méthode de vieillissement fiable pour des échantillons de soie standard qui résulterait en un état de dégradation proche de celui de la soie des costumes du XVII^e siècle. Quatre méthodes de vieillissement ont été testées : (1) l'oxydation thermique dans l'air sec; (2) l'exposition à différentes humidités relatives (HR); (3) l'immersion dans des solutions de pH variés; (4) l'exposition accélérée aux UV à 50 ± 2 °C et 95 % HR. Différentes propriétés physiques et chimiques de la soie ont été évaluées par spectroscopie infrarouge à transformée de Fourier, chromatographie d'exclusion stérique ainsi que par des essais de traction employés comme indicateurs analytiques pour comparer les soies vieillies artificiellement à des échantillons provenant de costumes du XVII siècle. Parmi les méthodes de vieillissement testées dans cette étude, l'oxydation thermique à 125 °C dans l'air sec pendant 28-56 jours a donné des échantillons de soie dont les propriétés étaient presque les mêmes que celles des tissus historiques.

Zusammenfassung — An der Königlichen Waffenkammer in Stockholm wurde ein Projekt zur Evaluierung verschiedener Konservierungsmethoden für historische Kostüme durchgeführt. Da der historische Wert der Artefakte ihre direkte Verwendung in den Experimenten verbietet, wurden künstlich gealterte Seiden als Ersatz genommen. Es wurde versucht, eine Alterungsmethode zu finden, die die künstlichen Standardseidenproben in einen den Proben des 17. Jahrhunderts entsprechenden Alterungszustand versetzt. Es wurden vier Alterungsmethoden untersucht: (1) thermische Oxidation in trockener Luft, (2) Alterung bei verschiedenen relativen Luftfeuchtigkeiten (rH), (3) Eintrag der Proben in Lösungen von verschiedenem pH, (4) beschleunigte Lichtalterung (UV) bei 50 ± 2°C, 95% rH. Die chemischen und physikalischen Eigenschaften wurden mit Hilfe der Fouriertransform – Infrarotspektroskopie (FTIR) und der Gel-Permeations-Chromatographie (GPC) sowie mit Dehnungsversuchen bestimmt. Sie dienten als Vergleichsindikatoren zischen der künstlich gealterten Seide und den Proben des 17. Jahrhunderts. Bei den getesteten Alterungsmethoden erbrachte die Thermooxidation in trockener Luft bei 125°C für 28–56 Tage gealterte Seiden, die denen der historischen Textilen am ehesten entsprachen.

Resumen — La Armería Real de Estocolmo ha llevado a cabo un proyecto para evaluar experimentalmente métodos de conservación usados en vestimentas históricas. Como el valor histórico de los objetos auténticos excluye su uso en los trabajos experimentales, las sedas han de ser sustituidas por sedas estándar envejecidas experimentalmente. Este estudio tiene la finalidad de encontrar un método apropiado de envejecimiento artificial para sedas estándar que produzca un nivel de deterioro similar al existente en las sedas de las vestimentas del siglo XVII. Se estudiaron cuatro métodos diferentes de envejecimiento artificial: 1- oxidación termal en aire seco, 2- exposición a diferentes niveles de humedad relativa (RH), 3- inmersión en disoluciones de diferente pH, 4-exposición acelerada a radiación ultravioleta (UV) a 50±2°C, 95% RH. Se evaluaron diferentes propiedades físicas y químicas de la seda usando espectroscopía infrarroja por transformada de Fourier, cromatografía por exclusión de tamaño y pruebas tensiles, las cuales fueron utilizadas como indicadores analíticos para la comparación entre las sedas envejecidas artificialmente y muestras procedentes de tejidos del siglo XVII. De los métodos de envejecimiento investigados en este estudio, la termo-oxidación a 125°C en aire seco durante 28-56 días produjo sedas con las propiedades más parecidas a las muestras de sedas antiquas.

Paper III

Francisco Vilaplana, Johanna Nilsson, Dorte Vestergaard Poulsen Sommer, and Sigbritt Karlsson, "Analytical markers for the degradation of historic and artificially aged silk in different environments.", *Analytical and Bioanalytical Chemistry*, 2015, 407, (5) p. 1433-1449. DOI 10.1007/s00216-014-8361-z

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Error list

P. 1434-5, correct RH values for the samples aged in 60°C with: Mg(NO3)2 is uncertain and was approximately 42.5% instead of 53%; NaCl was 74.5±0.30% instead of 75%; and, KCl was 80.25±0.41% instead of 86%.

P. 1437 under 3.2 Brightness measurements, "extreme acid pH", should be "extreme pH".

RESEARCH PAPER

Analytical markers for silk degradation: comparing historic silk and silk artificially aged in different environments

Francisco Vilaplana · Johanna Nilsson · Dorte V. P. Sommer · Sigbritt Karlsson

Received: 6 November 2014 / Accepted: 19 November 2014 / Published online: 10 December 2014 © The Author(s) 2014. This article is published with open access at Springerlink.com

Abstract Suitable analytical markers to assess the degree of degradation of historic silk textiles at molecular and macroscopic levels have been identified and compared with silk textiles aged artificially in different environments, namely (i) ultraviolet (UV) exposure, (ii) thermo-oxidation, (iii) controlled humidity and (iv) pH. The changes at the molecular level in the amino acid composition, the formation of oxidative moieties, crystallinity and molecular weight correlate well with the changes in the macroscopic properties such as brightness, pH and mechanical properties. These analytical markers are useful to understand the degradation mechanisms that silk textiles undergo under different degradation environments, involving oxidation processes, hydrolysis, chain scission and physical arrangements. Thermo-oxidation at high temperatures proves

Electronic supplementary material The online version of this article (doi:10.1007/s00216-014-8361-z) contains supplementary material, which is available to authorized users.

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to be the accelerated ageing procedure producing silk samples that most resembled the degree of degradation of early seventeenth-century silk. These analytical markers will be valuable to support the textile conservation tasks currently being performed in museums to preserve our heritage.

Keywords Silk \cdot Conservation \cdot Multivariate analysis \cdot Amino acid composition \cdot Infrared spectroscopy \cdot Mechanical properties

Introduction

Silk from the cultivated Bombyx mori larva is an appreciated material in historic costumes due to its valuable properties such as high lustre, smoothness, strength and lightness. However, silk is one of the natural fibres most sensitive to environmental degradation factors that cause deterioration of its intrinsic properties, becoming fragile and therefore difficult to preserve. The Royal Armoury in Stockholm possesses extraordinary costumes in silk from the seventeenth century, both in quantity and quality, which are in need of conservation. Conservators concerned with the preservation of historic silk must assess the degradation level in the material when deciding whether an item can be exhibited and how it should be treated for future preservation. It is therefore necessary to understand the environmental degradation mechanisms that affect silk through their ageing and to find reliable analytical markers for monitoring the degree of degradation of historic silk textiles.

Silk is a highly oriented and crystalline proteinaceous fibre containing mainly fibroin and sericin proteins. It consists of a double filament of fibroin with sericin acting as glue around the two filaments. The filament is 7–12 µm in width and composed of fibrillar elements of 1 µm width, in turn made up of microfibrils that are 10 nm in diameter [1]. Silk fibroin consists of two main components, H-fibroin with molecular



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weight of about 350,000 Da and a smaller L-fibroin with about 25,000 Da, which are linked by a single intermolecular disulphide bridge [2]. A third component is the glycoprotein P25, which is present in a much smaller proportion than the two others, and associated with the H- and L-fibroins by noncovalent forces [3, 4]. The molar ratio between H-fibroin, Lfibroin and P25 is 6:6:1 [5]. The H-fibroin primary structure consists of 20 amino acids, mainly glycine (Gly) 45.9 %, alanine (Ala) 30.3 %, serine (Ser) 12.1 % and tyrosine (Tyr) 5.3 %. The amino acid sequence consists of alternating crystalline and amorphous subdomains. The crystalline domains are made of a repeated Gly-X dipeptide motif, where X is Ala in 65 %, Ser in 23 % and Tvr in 9 % of the cases [6], Glv-X dipeptide units are present mainly as part of the two hexapeptides ~Gly-Ala-Gly-Ala-Gly-Ser~ (433 copies) and ~Gly-Ala-Gly-Ala-Gly-Tyr~ (120 copies) which together count for 70 % of the crystalline domains. The Gly-X repeats are distributed in 12 "crystalline" domains with varying length between 39 and 612 amino residues, separated by almost identical copies of boundary "amorphous" sequences. These amorphous spacers in B. mori silk are tyrosine rich and also contain most of the other amino residues that are absent in the Gly-X domains, basically amino acids with bulky and polar side chains. These amorphous domains break the Gly-X alternate and terminate the crystalline regions. The Gly-X alternation is strict within the crystalline subdomains; this strongly supports the classic-pleated β-sheet model of secondary structure, in which β-sheets pack on each other in alternating layers of Gly/Gly and X/X contacts. In the amorphous regions, however, distorted β -sheets are present [7].

Research on environmental factors influencing silk objects in historic houses is usually performed by accelerated ageing test methods [8-10]. Exposure to light is usually thought to play an important role in changing historic silk's chemical and physical properties as well as its aesthetics. However, previous studies strongly suggest that the role of light on the deterioration of historic silk textiles may be exaggerated [11]. Ultraviolet (UV) irradiation is a common procedure to artificially mimic the effect of daylight on historic silk textiles; however, accelerated UV irradiation affects the structure and the properties of silk textiles in a different way than natural daylight exposure, resulting in crosslinking of the material and altered mechanical properties [12]. Extreme humidity conditions, both at high and low levels, are known to have negative effects on silk by aiding deterioration. Moreover, temperature affects and accelerates deterioration, reduces molecular weight and has a negative effect on tensile strength [8-14]. In general, these previous studies use different artificial ageing procedures to analyze the chemical structure and properties of silk fibroin in comparison with historic samples, but no comprehensive correlation between the mechanical properties and the molecular structural details is proposed. The aim of the present research is to identify suitable analytical markers at the molecular and macroscopic

level and establish correlations amongst them, in order to assess the degree of degradation of historic silk. The changes at the molecular level (amino acid composition, formation of oxidative moieties, crystallinity and molecular weight) and on the macroscopic properties (pH. brightness, mechanical properties) have been investigated and integrated using statistical approaches. Analytical results of three historic samples from the seventeenth century have been correlated with artificially aged silk in four different environments, namely (i) UV exposure, (ii) thermo-oxidation, (iii) controlled humidity and (iv) pH. Significant and extensive data has been obtained for the first time about the tensile properties (elongation at break, tenacity, modulus) of both artificially aged and historic silk textiles. These analytical markers are used to better understand the degradation mechanisms that silk textiles undergo during prolonged exposure and to support the heritage preservation tasks currently performed in our museums.

Experimental

Materials

White reference silk fabric, ISO 105-F06:2000 B. mori (Cromocol, Boras, Sweden) was used for artificial ageing. The silk has an average weight of 60 g m⁻² and a defined tone and grade of whiteness and pH according to ISO 3071. The manufacturing process of the historic silk differs from the one used today for modern standard silk, but for this study, it was not possible to make exact copies produced in the same way as historic silk. The historic silk samples were taken from the costume collection at the Royal Armoury (Stockholm), specifically from the doublet (inventory number Lrk 25605) and from the breeches (inventory number Lrk 25606) of the coronation costume of King Gustav II Adolf (GIIA) from 1617 and from the coronation cloak (inventory number Lrk 25599) of King Karl X Gustav (KXG) from 1654. Samples were taken from the weave in the seam allowance taking special care to avoid external contamination (Fig. 1). The chemicals and buffers were supplied by Sigma-Aldrich, USA.

Accelerated ageing of silk

Samples of reference silk were artificially aged in groups of five replicates by exposure to four different environmental conditions: (a) accelerated UV exposure for 1–10 days; (b) thermo-oxidation in dry air at 60 and 125 °C for 14, 21, 28, 35, 42, 49 and 56 days; (c) exposure to 0, 53, 75, 86 and 100 % relative humidity (RH) at 25 and 60 °C for 28 days; and (d) immersion in and exposure to solutions of pH 1, 3, 7, 11 and 13 at 25 °C and at 60 °C for 28 days. Exposure to a controlled temperature in methods b–d was carried out using a forced ventilation oven, Memmert D06062 (Memmert Gmbh,



Fig. 1 Historic silk costumes and sampling procedure. a King Gustav II Adolf (GIIA) doublet showing where sample was taken from the lining; b detail of GIIA doublet lining; c GIIA breeches showing where sample was taken; d detail of the seam allowance. e King Karl X Gustav (KXG) cloak showing where samples were taken; f detail of seam allowance. Photo: a–d, f courtesy of Erik Lernestål; e courtesy of Göran Schmidt



Germany). The samples subjected to method b–d; sample size 5×8 cm) were mounted in sealed vials. The relative humidity was controlled by saturated salt solutions: dry calcium chloride (CaCl₂) was used for 0 % RH, saturated solutions of magnesium nitrate (Mg(NO₃)₂) for 53 % RH, sodium chloride (NaCl) for 75 % RH, potassium chloride (KCl) for 86 % RH

and distilled water for 100 % RH. Different buffers from Sigma-Aldrich were used to control pH exposure: Hydrogen chloride/potassium chloride (HCl/KCl) was used for pH 1, citric acid/sodium hydroxide/sodium chloride buffer (C_6H_8O -/NaOH/NaCl) for pH 3, potassium phosphate/ disodium hydrogen phosphate buffer (KH₂PO₄/Na₂ HPO₄)



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for pH 7, boric acid/sodium hydroxide/potassium chloride buffer (B(OH)₃/NaOH/KCl) for pH 11, sodium chloride/glycine/sodium hydroxide buffer (NaCl/glycine NaOH) for pH 13. Samples were either immersed or exposed over the pH buffer to control the effect of the pH degradation. Accelerated UV exposure was performed in a Q-U-V Accelerated Weather Tester with a UVB 313 lamp according to ASTM G 53–96, with a peak irradiance at 313 nm and total irradiance of 0.5 W.m⁻² at 50±2 °C at 95 % RH for 1–10 days in a cycle of 8 hours exposure and 4 hours condensation at a temperature of 50±2 °C. The samples for UV exposure were cut with dimensions 7.5×15 cm. After exposure to the different degradation environments, the samples were rinsed five times in deionised water and dried at room temperature at ambient humidity in a covered glass box (80 dm³) for 24 h. Samples were kept in desiccators prior to analysis.

Analysis of silk pH

The method to determine pH was miniaturised following the guidelines of previous studies [10, 15, 16]; 12 mg silk was soaked in 1.0 ml degassed 100 mM NaCl solution at 20 ± 2 °C for 30 min until equilibration and the pH of the extract was measured with a BDH Glass+combination microelectrode, following two point calibration (pH 4.00 and 7.00). Depending on sample availability, the pH was determined for selected samples including the unaged reference standard silk, UV exposed for 4 and 10 days, dry thermal-oxidation at 125 °C in 28 and 56 days, immersed in solutions of pH 1 or pH 13 at 25 °C in 28 days, 100 % humidity at 25 °C in 28 days and samples from the three historic costumes.

Brightness measurements

The sample brightness was measured in triplicate on the artificially aged silk textile samples using a UV–vis spectro-photometer equipped with an integrating sphere (Varian) and reported as the transmittance values (%) at 457 nm. The brightness could unfortunately not be measured for the historic silk textiles due to the large sample size required for such measurements.

Fourier-transform infrared spectroscopy

The chemical changes on the surface of the silk samples were monitored by FTIR using a Spectrum 2000 FTIR spectrometer (Perkin Elmer, Wellesley, MA) equipped with attenuated total reflection (ATR). Spectra were collected from the average of 24 scans between 2000 and 600 cm⁻¹ at intervals of 1 cm⁻¹ with a resolution of 4 cm⁻¹. The spectral data for each sample was subjected to baseline correction and to normalisation towards the absorption at 1800 cm⁻¹, where no absorbance band was observed in any of the samples, using the

Spectrum FTIR software. Each measurement was performed in triplicate, and the quantitative results for the carbonyl and amide II crystalline indexes were calculated as the averages from these measurements.

Amino acid composition

Between 80 and 145 µg of silk sample were hydrolyzed for 24 h in an evacuated and sealed glass ampoule at 110 °C in a solution of 100 µl 6 M redistilled HCl, 5 µl 1 % 3,3'dithiodipropionic acid (DTDPA) in 0.2 M NaOH and 5 µl 1 % phenol in water. The hydrolyzed amino acids were separated by ion exchange HPLC (Waters, USA) with two Waters high pressure pumps, equipped with high sensitivity pulse dampers and microflow modules, Waters M 717, refrigerated auto sampler, two Reagent Manager pumps and a column oven. Separation was carried out in a 15×0.40 cm steel column packed with MCI CK 10 U resin (Mitsubishi Chemical Industries) using a pH gradient system with two buffers: (A) pH 3.10:0.20 sodium citrate containing 0.65 % nitric acid and 5 % isopropanol; (B) pH 10.20:0.210 M sodium borate, 5 % isopropanol and 0.17 M sodium hydroxide. The eluted amino acids were quantified by post-derivatisation with ortho-phthalaldialdehyde (OPA) using a Waters M 474 fluorescence detector with 338 nm band-pass excitation filter and 450 nm long-pass emission filter. The amino acids were identified and quantified on the basis of an external standard mixture of amino acids Beckman no 33 1018, with additional Hydroxylysine and the degradation products α -aminoadipic acid (α -Ada), α -aminobuteric acid (α -Abu), β -alanine (β -Ala), γ -aminobuteric acid (γ -Abu), 6-aminohexanoic acid (6-Aha) and ornithine (Orn). The results are compared with a model silk sequence (MSS) calculated from the amino acid sequences of the B. mori fibroin heavy chain (sequence number P05790, UniProtKB/Swiss-Prot), B. mori fibroin light chain (sequence number P21828, UniProtKB/Swiss-Prot) and the sequence of fibroin P25 (sequence number P04148, UniProtKB/Swiss-Prot). The reported values are the sum of the three in the ratio 6:6:1 [11]. The amino acid composition was determined for selected silk samples (same as for the pH measurements).

Size-exclusion chromatography

Size-exclusion chromatography (SEC) was performed in a LC-20AD liquid chromatography instrument with refractive index detection (RID; Shimadzu, Japan). Between 3 and 8 mg of sample were initially washed at room temperature in water $(3\times30 \text{ min})$ and methanol $(3\times30 \text{ min})$ to swell and open up the structure. After filtering, 1 ml N,N-dimethylacetamide (DMAc) with 8 % (w/w) lithium chloride (LiCl) was added to the silk and left under agitation at room temperature until the silk was completely dissolved. The silk solution was

further diluted to achieve injection conditions (DMAc/0.5 % (w/w) LiCl). Separation was performed using a PL gel 20 mm Mixed-A column (Agilent, USA) at 40 °C using DMAc/0.5 % (w/w) LiCl as a mobile phase with a flow of 0.5 ml min⁻¹. Relative calibration was performed using Pullulan standards (Agilent, USA).

Tensile tests

The tensile tests were conducted in a Vibrodyn ® CRE-type with a gauge length of 20 mm, a tension weight of 2000 mg and an extension rate of 20 mm/min, following DIN EN ISO 5079 standard*. Samples were conditioned at 20±2 °C and 65 ± 3 % RH prior to analysis. Eight 40-mm threads taken from the weft of each historic costume were tested; they were taken in the seam allowance of the breeches and the cloak and in a damaged area of the doublet. All extracted threads had almost no twist. Analyses of the historic silk showed that it was degummed but had small residues of sericin. A minimum of five threads from each artificially aged sample were removed from the weft, their tex value was calculated by measuring the length and weight of each thread and were finally subjected to tensile testing. The tensile tenacity (cN/tex), break extension (%) and Young's modulus were obtained as the average from the tensile testing. Tests of significance, one-way ANOVA, were performed to ensure that reliable effects were obtained. The focus was first on general different effects of the exposures to the ageing methods, followed by tests of pair-wise differences where unaged reference silk was one part if the general difference was found to be significant. When appropriate, Welch's method and Tamhane's T were applied. In the studies of the effect of RH that had two sources of variance, univariate ANOVA was used to estimate the individual effect on variance caused by different sources as well as their interaction (SPSS Statistics, version 19).

Results and discussion

pH measurements

Acidity is a traditional indicator of silk degradation in textile conservation science. Low pH values during textile washing are usually correlated with specimen deterioration and this needs to be taken into account during their conservation to avoid further catalytic degradation action. Therefore, a neutral pH is strived for during textile preservation. In general, accelerated aged silk shows lower pH values than the reference silk and the acidity increased progressively with exposure time for the UV and thermo-oxidised samples (Fig. 2). The historic samples show similar pH values to the samples exposed to accelerated UV for 10 days and

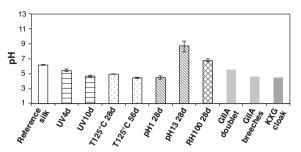


Fig. 2 Extract acidity of the historic and artificially aged silk textiles

the samples subjected to thermal-oxidation at 125 °C for 28 days. This increased acidity indicates changes in the chemical structure of the silk fibres with natural and artificial ageing that will be studied in detail using integrated physico-chemical analyses.

Brightness measurements

Brightness is a commonly used visual degradation marker that is related to colour changes (yellowing) in non-dyed silk textiles. The brightness results from the artificially aged silk exposed to different environments are presented in Fig. 3 and in the Electronic supplementary material (ESM Fig. S1). UV irradiation causes a marked exponential initial decrease in brightness, which stabilises after 2-4 days. Contrarily, thermo-oxidation at high temperatures (125 °C) causes a progressive linear decrease in brightness with exposure time, whereas lower exposure temperatures do not cause such dramatic reduction in brightness. Indeed, after 28 days of exposure to thermo-oxidation at 60 °C, no remarkable changes in brightness could be observed. Exposure to relative humidity at 25 and 60 °C for 28 days does not affect the brightness of the silk samples. Finally, only immersion into extreme acid pH solutions (both acidic at pH 1 and alkaline at pH 13) causes a dramatic decrease in brightness, similar to that of UV exposure. Milder alkaline and acidic pH conditions do not cause a marked alteration in the brightness after immersion or exposure for 28 days at neither 25 nor 60 °C. The sample immersed at 60 °C at pH 13 could not be measured since it completely disintegrated during exposure. The decrease in brightness is thought to be caused by the formation of chromophoric groups (e.g. oxidative moieties) during the degradation environments to which silk textiles are exposed; this will be correlated with the chemical changes by FTIR and with the amino acid content.

Fourier-transform infrared spectroscopy

Chemical and structural changes were monitored using FTIR for the accelerated aged and historic silk samples. Figure 4a shows selected spectra in the region between 600 and



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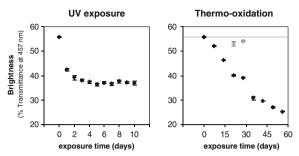
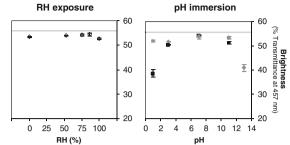


Fig. 3 Effect of the different artificial degradation environments on the brightness (expressed as % of transmittance at 457 nm) of silk textiles. a UV exposure; b thermo-oxidation (black, 125 °C; grey 60 °C); c RH



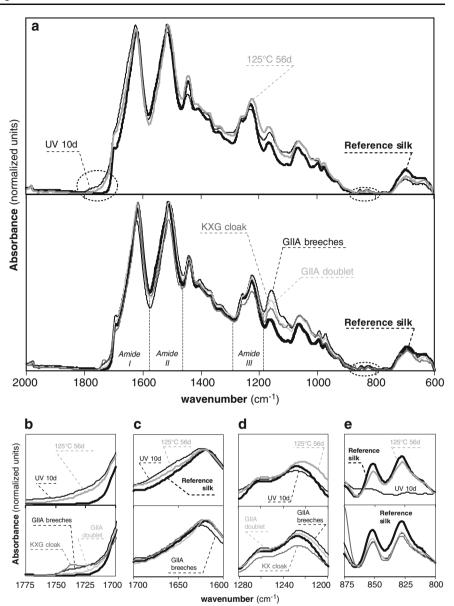
exposure (black, 60 °C); **d** pH immersion (black square 60 °C; grey rhombus, 25 °C). The area in grey corresponds with the reference silk

2000 cm⁻¹, where the main bands corresponding to the amide I (1580–1700 cm⁻¹), amide II (1480–1580 cm⁻¹), amide III (1200–1300 cm⁻¹), C–H bending (1175 cm⁻¹) and skeletal stretching (900–1100 cm⁻¹) regions can be observed [17, 18]. The FTIR spectra for the rest of the accelerated aged silk samples were also obtained but are not presented here for the sake of clarity. These major bands correspond mainly to the associated backbone vibrations of the peptide chains in the Gly-Ala-Gly-Ala-Gly-X sequence (X accounts for Ser or Tyr), which accounts for the vast majority of the crystalline domains in silk fibroin and also correspond with above 90 % of the amino acid content in our studied silk textiles (see "Amino acid composition"). Two small bands can be observed at wavenumbers of 835 and 850 cm⁻¹, which are assigned to the phenyl groups of Tyr.

The most important differences in the spectra are the appearance of absorbing bands in the free carbonyl region (1700-1775 cm⁻¹) for both the accelerated aged and the historic costumes when compared with the standard silk (Fig. 4b). The formation of free carbonyl moieties is a common indicator of oxidation processes in natural and synthetic macromolecules, and it can be used to evaluate the extent of degradation processes. At a first glance, UV and thermooxidative exposure at 125 °C cause the presence of such carbonyl bands, whereas pH exposure and RH do not cause such effect. Historic costumes do exhibit absorption in the carbonyl region, which evidences the extent of oxidation in such textiles. The formation of carbonyl moieties caused by natural and accelerated degradation can be quantified using the carbonyl index. The carbonyl index was calculated from the height intensities at a wavenumber of 1732 cm⁻¹ corresponding to the C=O vibration, normalised to the maximum height of the amide I band at 1620 cm⁻¹. We verified the sensitivity of the amide I band towards degradation by evaluating the height of the maximum of the band at 1620 cm⁻¹ for all the artificially aged and historical samples, and no marked changes were observed for any of the degradation environments or the historic samples (ESM Fig. S2), even though peptide hydrolysis occurred in certain conditions. Other studies on degradation in archaeological scrolls have observed an increase in the absorbance at 1650 cm⁻¹ relative to OHbending in the amide I region due to major hydrolysis of the collagen protein present in the parchments [19]. Collagen is also a fibrous protein, but unlike silk, collagen forms triple α helix structures that may be more susceptible to hydrolytic degradation than the β-sheet secondary structures in silk fibroin. The carbonyl index for the artificially aged samples (UV radiation, thermo-oxidation, RH and pH) and the historic costumes are presented in Fig. 5 and ESM Fig. S3. UV exposure causes a drastic increase in the carbonyl index. especially for the shorter exposure times, tending to stabilise for longer times (Fig. 5). Thermo-oxidative exposure at lower temperatures (60 °C) does not cause an increase in the carbonyl index after 28 days. Higher temperatures (125 °C), on the other hand, cause a progressive increase in the carbonyl groups, reaching at 60 days similar values to those exhibited after UV radiation (Fig. 5). RH exposure does not induce changes in the carbonyl region after 28 days, neither at ambient temperature (25 °C) (ESM Fig. S3) nor at higher exposure temperatures (60 °C) (Fig. 5). Immersion into aqueous solutions with extreme acidic pH conditions (pH 1 and pH 3) for 28 days causes a marked increase in the carbonyl index, especially at higher temperatures of 60 °C but also at ambient temperatures (Fig. 5). Exposure to environments with extreme acidic pH conditions does not cause such marked effect as direct immersion (ESM Fig. S3). The effect of pH immersion on the carbonyl region is however lower than the changes caused by UV and thermo-oxidative exposure. Extreme acidic conditions may catalyse oxidative reactions undergone by the exposed or immersed silk that arise to the formation of carbonyl groups. On the other hand, extreme alkaline conditions do not induce any alteration of the carbonyl region. The results for historic silk samples indicate that oxidative reactions have indeed occurred during the natural ageing of the textiles. The carbonyl index of the samples from the seventeenth century is at the same level as the thermo-oxidised silk at 125 °C (Fig. 5). These results evidence the importance of the carbonyl index as a fundamental analytical marker to monitor the degradation



Fig. 4 FTIR spectra of selected artificially aged and historic silk textiles: **a** overview of the midinfrared region (600–2000 cm⁻¹); **b** free carbonyl region (1700–1775 cm⁻¹); **c** amide I region (1600–1700 cm⁻¹); **d** amide III region (1200–1280 cm⁻¹); tyrosine doublet region (800–875 cm⁻¹)



state of historic silk samples. This could lead to the mapping of the degree of degradation of historic objects as a tool in collection management.

An interesting feature of this study is that neither artificial nor natural ageing affect to a great extent the features of the main band corresponding to amide I (Fig. 4c). The amide I band encodes information about protein conformation through the overlapping of the signals corresponding to α -helix, β -sheet and amorphous peptide chains [17]. The amide I region for standard and aged silk samples exhibits a main band at 1615 cm⁻¹ corresponding to hydrogen bonding between intermolecular β -sheet strands, a broad band at around 1650–

1660 cm⁻¹ for random coil chains and an elbow at 1690 cm⁻¹ assigned to β-turns of the antiparallel β-sheet structure [20–22]. The comparison of the FTIR signals from the amide I region indicates that the silk fibres do not undergo any major conformational transition induced by the natural or accelerated degradation processes. This observation reinforces the use of the amide I band as a reference for the calculation of the carbonyl index.

For a clearer observation of the secondary conformation of the aged silk samples from *B. mori*, the region corresponding to the amide III was analysed in detail (Fig. 4d). Two distinct bands can be observed at 1227 and 1265 cm⁻¹, which can be



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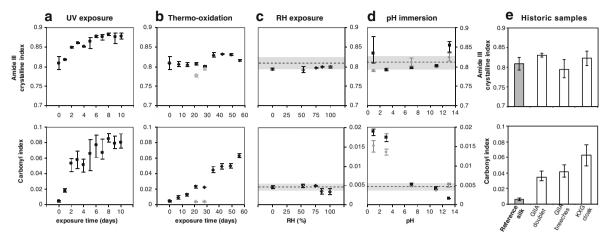


Fig. 5 FTIR crystalline and carbonyl indexes: a UV exposure; b thermo-oxidation (black, 125 °C; grey, 60 °C); c RH exposure (black, 60 °C); d pH immersion (black squares: 60 °C; grey rhombus, 25 °C); e historic silk samples. The area in grey corresponds with the reference silk

assigned to the random coil and β-sheet configurations, respectively [18]. In principle, no clear changes in the conformation can be observed after accelerated or natural ageing, in agreement with the previously reported results from amide I. However, a more detailed analysis by calculating the amide III crystalline index using the relative height intensities from the bands at wavenumbers of 1260 and 1227 cm⁻¹ reveals subtle changes in the fibre conformation (Fig. 5; ESM Fig. S3). UV radiation causes a marked increase in crystallinity, which tends to stabilise after prolonged exposure. Thermooxidation at 125 °C also seems to cause a subtle increase in the crystallinity index, but far less obvious than UV radiation. Finally, pH exposure at extremely acidic and alkaline conditions (pH 1 and pH 13, respectively) also seems to increase the crystallinity index. Exposure to different RH conditions does not seem to alter the conformation at the amide III region. In the case of the historic samples, the crystalline index for the GIIA doublet shows a slight increase in the crystalline index, whereas the remaining samples show similar values as the reference silk. This increase in crystallinity may be caused by chain scission reactions in the amorphous (random coil) regions of the silk fibres leading to fragments of lower molecular weight that can be more easily arranged into crystalline segments. These results point out that the amide III crystalline index constitutes a sensitive marker for monitoring the structural changes in historic silk textiles.

Finally, UV radiation causes a drastic reduction in the intensity of the tyrosine doublet band at wavenumbers of 835 and 850 cm⁻¹ (Fig. 4e); this is a consequence of the photo-oxidation of the chromophoric tyrosine amino acids that are present in the amorphous silk regions [23]. The historic samples exhibit a decrease in the tyrosine doublet as well, but not as drastic as the one caused by UV exposure. A similar decrease in the tyrosine bands can be observed for the

sample thermo-oxidised at 125 °C for 56 days. These changes in the Tyr intensity are confirmed by the amino acid results (see "Amino acid composition"). This indicates that accelerated UV exposure causes more severe degradation effects than the ones undergone by silk during its natural ageing, indicating that this artificial ageing procedure should be used with caution for mimicking the degradation state of historic textiles.

Amino acid composition

The results of the amino acid analyses for the selected historic and artificially aged silk samples can be found in Table 1, expressed as the percentage of the total amount of amino acids. In general, there is a fine conformity between the calculated model silk sequence (MSS) and the results, especially with the results of the unaged reference silk. The deviating values of Ser and Glu may be due to the poor separation of the two peaks during chromatography. Gly and Ala constitute the core of the amino acid sequence in the crystalline domains of silk and are usually regarded as stable amino acids. According to the model reference, a small amount of thiopropionyl-cystine (TPCys; which is a marker for cystine and cysteine) should be expected, but it has only been detected in the UV-aged samples. TPCys may be present in all samples but in such small amounts that the TPCys peak is hidden under the delayed Ala peak.

Tyrosine content (Tyr) is known as a marker for oxidation in silk fibroin [9]. The unaged reference silk shows a Tyr value very similar to the model reference, which again indicates the robustness of the amino acid quantification. Accelerated UV exposure causes a marked decrease of the Tyr value, which almost decreases by half after 10 days of exposure. Thermooxidation also causes a progressive decrease in the amount of



Amino acid composition (%) of historic and selected artificially aged silk textile samples Table 1

Sample	Amino acid	Amino acid composition (%)	(%															FC	Tyr/ Ty	Tyr/ Pl	Protein
	Asp ^b	Thr	Try° Ser	Glu ^b		Pro (Gly	Ala	TPCys	Val	Met	Ile	Leu	Tyr	Phe	His I	Lys Arg				
MSSa	1.51) 86:0	0.24 11.97	11.11		0.43	44.14	29.44	0.14	2.05	0.08	0.62	0.46	5.19	0.68	0.21 0	0.29 0.46		0.12 0.	0.18 –	
Reference silk	1.51 (0.01)	Reference 1.51 (0.01) 1.05 (0.21) - silk		10.28 (0.15) 1.29 (0.00) 0.44 (0.01)	0 (00.00) 6	.44 (0.01)	44.31 (0.06)	30.96 (0.05)	1	2.05 (0.02)	0.06 (0.00) ().62 (0.01)	0.45 (0.01)	5.18 (0.00)	0.73 (0.01)	$0.06\ (0.00) 0.62\ (0.01) 0.45\ (0.01) 5.18\ (0.00) 0.73\ (0.01) 0.28\ (0.04) -$		0.37 (0.03) 0	0.12 0.	0.17 90	2.96
UV4d	1.59 (0.01)	- (70.0) 0.97 (0.07) -		7 (0.10) 1.32	2 (0.02) 0	1.44 (0.02) 4	13.66 (0.64)	10.37 (0.10) 1.32 (0.02) 0.44 (0.02) 43.66 (0.64) 32.41 (0.23) 0.45 (0.23) 2.17 (0.06) 0.06 (0.01) 0.59 (0.00) 0.49 (0.01) 3.80 (0.00) 0.71 (0.01) 0.21 (0.07) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01) 0.71 (0.01)	0.45 (0.23)	2.17 (0.06)	0.06 (0.01) ().59 (0.00)	0.49 (0.01)	3.80 (0.00) (0.71 (0.01)	0.21 (0.07) -		0.31 (0.05) 0.09		0.12 8	85.0
P0IAO	1.49 (0.05)	1.49 (0.05) 1.07 (0.12) -		8 (0.06) 1.25	3 (0.03) 0	1.33 (0.06) 4	14.15 (0.13)	$10.18 \ (0.06) \ \ 1.23 \ (0.03) \ \ 0.33 \ (0.06) \ \ 44.15 \ (0.13) \ \ 33.06 \ (0.17) \ \ 0.68 \ (0.23) \ \ 2.13 \ (0.01) \ \ 0.05 \ (0.00) \ \ 0.65 \ (0.00) \ \ 0.47 \ (0.00) \ \ 3.08 \ (0.03) \ \ 0.66 \ (0.01) \ \ 0.18 \ (0.02)$	0.68 (0.23)	2.13 (0.01)	0.05 (0.00) ((0.00) 55.	0.47 (0.00)	3.08 (0.03)	0.66 (0.01)	0.18 (0.02) -		0.29 (0.03) 0	0.07 0.	0.09 8	80.9
T125C 28d	1.52 (0.02)	F125C 28d 1.52 (0.02) 0.91 (0.08) -		2 (0.12) 1.30	0 (10.0) 0	1.41 (0.03) 4	$10.12\ (0.12) 1.30\ (0.01) 0.41\ (0.03) 44.67\ (0.03) 31.52\ (0.03)$		1	2.10 (0.04)	2.10 (0.04) 0.04 (0.00) 0.59 (0.01) 0.49 (0.01) 4.70 (0.04) 0.73 (0.02) 0.23 (0.00)).59 (0.01)	. (10.0) 4	4.70 (0.04)	0.73 (0.02)	0.23 (0.00) -		0.26 (0.03) 0	0.11 0.	0.15 87	87.7
T125C 56d	1.68 (0.01)	T125C 56d 1.68 (0.01) 0.67 (0.02) -		(0.03) 1.35	8 (0.02) 0	1.42 (0.02) 4	9.25 (0.03) 1.38 (0.02) 0.42 (0.02) 43.87 (0.05) 33.09 (0.12)		1	2.18 (0.01)	$2.18\ (0.01) 0.02\ (0.01) 0.62\ (0.01) 0.54\ (0.02) 4.32\ (0.04) 0.72\ (0.05) 0.41\ (0.05)$).62 (0.01) ().54 (0.02)	4.32 (0.04)	0.72 (0.05)	0.41 (0.05) -		0.37 (0.02) 0	0.10 0.	0.13 90	90.0
RH100 28d	1.59 (0.00)	RH100 28d 1.59 (0.00) 0.71 (0.01) -		(0.04) 1.35	3 (0.01) 0	1.45 (0.02) 4	$9.81 \ (0.04) 1.33 \ (0.01) 0.45 \ (0.02) 43.94 \ (0.10) 31.28 \ (0.13)$		1	2.23 (0.03)	$2.23\ (0.03) 0.06\ (0.00) 0.63\ (0.02) 0.52\ (0.01) 5.24\ (0.02) 0.75\ (0.00) 0.46\ (0.01)$).63 (0.02) (3.52 (0.01)	5.24 (0.02)	0.75 (0.00)	0.46 (0.01) -		0.50 (0.01) 0.12		0.17 9	95.0
pH1 28d	1.51 (0.06)	pH1 28d 1.51 (0.06) 0.72 (0.01) -		7 (0.14) 1.37	7 (0.03) 0	1.45 (0.06) 4	10.07 (0.14) 1.37 (0.03) 0.45 (0.06) 42.23 (0.74) 3	32.46 (0.36)	1	2.19 (0.05)	$0.07 \; (0.00) \;\; 0.64 \; (0.01) \;\; 0.50 \; (0.02) \;\; 5.55 \; (0.11) \;\; 0.80 \; (0.02) \;\; 0.46 \; (0.06)$.64 (0.01) ().50 (0.02)	5.55 (0.11) (0.80 (0.02)	0.46 (0.06) -		0.51 (0.06) 0	0.13 0.	0.17 93	93.4
pH13 28d	0.86 (0.01)	pH13 28d 0.86 (0.01) 0.47 (0.04) -		9.84 (0.52) 1.01 (0.17) 0.37 (0.08) 43.75 (1.04)	1 (0.17) 0	1.37 (0.08) 4	13.75 (1.04)	33.96 (0.45)	1	1.95 (0.01)	$1.95 \; (0.01) 0.03 \; (0.00) 0.38 \; (0.00) 0.27 \; (0.01) 5.38 \; (0.01) 0.55 \; (0.01) 0.47 \; (0.07)$.38 (0.00) (3.27 (0.01)	5.38 (0.01)	0.55 (0.01)	0.47 (0.07) -		0.37 (0.04) 0	0.12 0.	0.16 9	97.3
GIIA	1.63 (0.09)	1.63 (0.09) 0.97 (0.07) -		10.61 (0.05) 1.40 (0.04) 0.47 (0.06) 43.86 (1.15)	0 (0.04) 0	47 (0.06) 4	43.86 (1.15)	31.00 (0.76)	1	2.21 (0.04)	$0.06\;(0.02) 0.62\;(0.02) 0.52\;(0.00) 4.81\;(0.46)$).62 (0.02) ().52 (0.00)		0.77 (0.02) 0.28 (0.02)	0.28 (0.02) -		0.33 (0.04) 0	0.11 0.	0.16 76	76.2
GIIA	1.61 (0.02)	1.61 (0.02) 1.13 (0.08) -		4 (0.12) 1.36	5 (0.01) 0	45 (0.00) 4	10.34 (0.12) 1.36 (0.01) 0.45 (0.00) 43.90 (0.07) 31.66 (0.02)		1	2.10 (0.03)	$2.10\ (0.03) 0.05\ (0.00) 0.61\ (0.00) 0.47\ (0.01) 4.62\ (0.01) 0.72\ (0.01) 0.26\ (0.01)$.61 (0.00) ().47 (0.01)	1.62 (0.01)	0.72 (0.01)	0.26(0.01) -		0.29 (0.01) 0.11		0.15 86	0.98
breeches KXG cloak	1.62 (0.01)	breeches KXG cloak 1.62 (0.01) 1.11 (0.13) –		8 (0.16) 1.41	1 (0.01) 0	1.53 (0.02) 4	10.28 (0.16) 1.41 (0.01) 0.53 (0.02) 43.74 (0.87) 31.50 (0.73)		1	2.14 (0.01)	0.06 (0.00) ().61 (0.01)).47 (0.01)	4.78 (0.06)	0.75 (0.03)	2.14 (0.01) 0.06 (0.00) 0.61 (0.01) 0.47 (0.01) 4.78 (0.06) 0.75 (0.03) 0.29 (0.01) -		0.30 (0.05) 0.11 0.15	.11 0.		83.2

The standard deviation (SD) is presented between brackets

^a The model silk sequence (MSS) is calculated from the amino acid sequences of the *Bombyx mori* Fibroin heavy chain (sequence number P05790), *B. mori* Fibroin light chain (sequence number P04148), where the reported values are an addition between the three in a ratio 6:6:1, respectively ^b The value of Glu is representing both glutamin and glutamic acid, and Asp is representing both asparagin and aspartic acid

^c Tryptophan is only present in the model reference as it is destroyed during the acid hydrolysis of the samples

^d Tyr/Gly ratio according to Zhang et al. [23]

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Tyr, but this is moderate in comparison with UV exposure. On the other hand, no decrease of Tyr can be observed in the samples treated neither at pH 1 and pH 13 nor with 100 % RH exposure. The historic samples also exhibit lower Tyr values, which again indicate that they have been subjected to some degree of oxidation during their natural ageing. The low Tyr values in the historic and the artificially aged (UV and thermooxidised) silk textiles may indicate the occurrence of oxidative reactions in the amorphous spacers within the silk structure, in agreement with the FTIR results. Indeed, it is known that the amorphous spacers are rich in Tyr that will be more susceptible to oxidative reactions than the crystalline regions. In the case of the UV and thermo-oxidised samples, the results point towards to the transformation of Tyr into Ala by loss of the phenol group [9, 24] and the formation of oxidative derivatives such as hydroxyphenylalanine and quinones [25, 26]. Zhang et al. [24] use the molar ratio Tyr/Gly to indicate presence of sericin, but due to tyrosine's sensitivity towards oxidation the ratio may also be used as an indicator of changes within the silk fibroin structure. The Tyr/Gly ratios are presented in Table 1, which show a decrease for the samples exposed to UV compared with the reference and historic silk textiles. An even better marker for increasing oxidative changes may be found in the decreasing Tyr/Ala ratio (Table 1), where Tyr in many cases is transformed into Ala. Again, it can be observed that the UV exposure surpasses the effect caused by thermo-oxidation in comparison with the historic costumes. No appreciable decrease in the Tyr content is observed for the samples exposed to RH and immersed into extreme acidic and alkaline pH conditions. These results point out that Tyr oxidation is negligible in these exposure conditions.

Immersion into extreme acidic and alkaline pH environments (pH 1 and pH 13, respectively) is expected to cause hydrolysis and chain scission of the peptide bonds in silk fibroin. This hydrolysis is expected to occur predominantly in the amorphous spacers within the fibroin chains, which are in principle more sensitive to degradative effects compared with the crystalline regions. Indeed, a meaningful reduction in the amino acids with polar side chains (Asp, Thr, Ser) and with hydrophobic side chains (Ile, Leu) is observed, especially for the sample subjected to pH 13. These amino acids are particularly found in the amorphous regions connected to the Gly-X crystalline domains, which indicates the special sensitivity of these amorphous spacers to alkaline hydrolysis. A progressive decrease in amino acids with polar chains (especially Ser and Thr, but not Asp) is similarly observed for the samples exposed to thermo-oxidation.

Traces of the breakdown product γ -Abu are found in some historic samples (KXG cloak and GIIA doublet) and in the samples exposed to UV and thermo-oxidation at 125 °C (data not shown). γ -Abu is most likely originating from Pro and Lys [27]. All samples show a small peak right before Lys. This peak is identical with Ornithin, a breakdown product

originating from Arg. None of the samples showed traces of the degradation products Ada, Abu, β-Ala and 6-Aha.

From the amino acid analysis, it can be concluded that environmental degradation causes important changes in the molecular structure of silk fibroin, affecting predominantly the amino acids contained in the amorphous regions. UV and thermo-oxidation seem to cause the oxidation of the Tyr group and an increase in the formation of Ala, which may lead to an increase of the crystallinity and changes in the mechanical performance. A progressive loss of Ser is also observed in these samples, which may affect the crystallinity of these samples. On the other hand, severe acid and alkaline conditions seem not to affect Tyr but cause hydrolysis in the amino acids containing polar and bulky side chains in the amorphous regions, which seem to be the weakest points for hydrolysis in the silk structure.

Size-exclusion chromatography

The relative molecular weight distributions of the artificially aged and historic silk samples were evaluated with SEC in N,N-dimethylacetamide (DMAc)/0.5 % (w/w) LiCl as the solvent system. Reference silk presents a monomodal SEC distribution (Fig. 6) with large (poly)dispersity, which is expected to cover both populations of H- and L-fibroins. Exposure to UV irradiation causes a shift in the retention time of the SEC chromatogram to higher elution volumes and the formation of a bimodal distribution, evidencing the reduction in chain length caused by UV degradation. It is worth mentioning that the presence of insoluble particles was observed in the DMAc/LiCl solutions of UV-irradiated silk; these particles were filtered prior to SEC analysis to avoid column deterioration and were therefore not analysed. The formation of insoluble particles was not reported neither for other degraded samples in this study nor for the historic silk samples. The formation of these particles may indicate the occurrence of crosslinking caused by UV irradiation in addition to oxidation and chain scission, as it has been reported for other macromolecules [28].

Exposure to thermo-oxidation at moderate temperatures (up to 60 °C) does not seem to affect silk chain length after 28 days. However, exposure to high temperatures (125 °C) causes a progressive shift of the chromatograms to higher elution volumes. The formation of bimodal distributions is observed as well for the thermo-oxidised samples at 125 °C, indicating the formation of two macromolecular populations of different chain lengths by chain scission. The relative abundance of these macromolecular population changes progressively with exposure times as it can be observed by the relative peak heights in the SEC chromatograms, with increasing relative abundance of the population of lower chain lengths at higher elution volumes with increasing exposure.



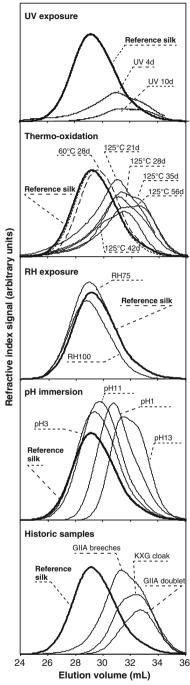


Fig. 6 SEC chromatograms for the artificially aged and historic silk samples: **a** UV exposure; **b** thermo-oxidation; **c** RH exposure at 25 $^{\circ}$ C for 28 days; **d** immersion into pH solutions at 25 $^{\circ}$ C for 28 days; and **e** historic samples

Exposure to relative humidity does not affect the shapes of the SEC distributions, which indicates that high RH values at moderate temperatures do not induce chain scission reactions in the time frames of the present study. Immersion into extreme pH solutions at 25 °C, on the other hand, causes a shift of the SEC distributions to higher elution volumes, evidencing the effect of hydrolysis of the peptide chains. This effect is more marked for extreme alkaline conditions (pH 13), which effectively causes extensive hydrolysis and the formation of the degraded bimodal distribution already observed for the UV and thermo-oxidised samples at 125 °C.

The historic samples all exhibit similar SEC bimodal distributions with lower chain length as the reference silk, evidencing the occurrence of chain scission of the peptide silk chains. This reduction of molar mass is indeed a common feature in organic archaeological materials and artefacts. The degree of degradation can therefore be monitored for the historic samples by the relative abundance of such macromolecular populations with reduced chain length. From the results of the SEC analyses, it seems that thermo-oxidation at 125 °C using different degradation times can be effectively used to model the chain length distribution of historic samples subjected to different degrees of degradation.

Tensile properties

To validate the four methods for artificial ageing against the properties of seventeenth-century silk fabric, tensile tests were used to compare the mechanical markers of historic silk samples with those of artificially aged samples (Table 2). The tenacity indicates the resistance to steady forces and will be the suitable magnitude to consider when a specimen is subjected to a steady pull. Elongation at break and tenacity should be regarded as the most important mechanical properties in conservation science when comparing artificially aged samples with historic silk. These properties are relevant when a specimen is subjected to stretching, as when the neck of a garment is being pulled over the head and when costumes are being mounted on mannequins during museum handling [29].

Accelerated ageing by UV exposure results in a sudden increase of the modulus after short exposure times (1–3 days) compared with the reference silk and a progressive decrease thereafter. On the other hand, the values of tenacity and elongation at break decrease progressively when compared with the reference silk. This sudden increase of the elastic modulus after only one day of UV irradiation may be explained by the physical reorganisation of the silk chains in the amorphous regions at moderate temperatures above room temperature, a typical phenomenon known as physical ageing in polymer science [30]. After this initial exposure, chemical changes caused by UV radiation become irreversible as shown by FTIR, SEC and amino acid composition, which results in a progressive deterioration of the mechanical properties. This indicates that degradation by UV irradiation does not



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 Table 2
 Mechanical properties of historic and artificially aged silk textile samples.

			Modulus (cN/tex)	Elongation at break (%)	Tenacity at break (cN/tex)
Unaged reference silk					
Unaged silk			191.5 (44.4)	21.0 (4.4)	31.8 (4.7)
Historic silk					
GIIA doublet			304.0 (66.8)	2.2 (0.8)	5.8 (2.2)
GIIA breeches			240.0 (61.8)	2.7 (0.6)	4.8 (2.7)
KXG cloak			274.0 (29.3)	3.3 (0.3)	5.9 (0.9)
UV exposure					
Temperature (°C)	Time (days)	RH (%)	Modulus (cN/tex)	Elongation at break (%)	Tenacity at break (cN/tex)
50±2 °C	1	95	337.0 (33.9)**	11.2 (1.8)**	17.6 (3.6)
	2	95	267.0 (14.4)***	8.9 (0.4)***	13.7 (0.8)***
	3	95	266.4 (6.4)***	7.2 (0.3)***	10.6 (0.4)***
	4	95	226.0 (14.8)	5.4 (0.2)***	6.2 (0.5)***
	5	95	189.8 (27.2)	5.3 (0.4)***	5.8 (0.5)***
	6	95	126.0 (8.9)***	4.3 (0.6)***	4.0 (0.7)***
	7	95	109.2 (15.8)***	4.5 (0.4)***	3.7 (0.7)***
	8	95	103.0 (7.5)***	3.8 (0.2)***	2.5 (0.4)***
	9	95	70.8 (8.3)***	3.4 (0.2)***	2.1 (0.2)***
	10	95	62.0 (1.7)***	2.6 (0.5)***	1.5 (0.1)***
Thermo-oxidative expos					
Temperature (°C)	Time (days)	RH (%)	Modulus (cN/tex)	Elongation at break (%)	Tenacity at break (cN/tex)
25	28	53	233.0 (41.1)	25.4 (5.6)	30.1 (5.2)
60	28	53	230.0 (47.8)	18.4 (3.9)	28.8 (6.6)
125	14	0	296.0 (8.1)***	9.0 (0.1)***	15.0 (0.2)***
	21	0	252.0 (15.8)	9.7 (0.3)***	14.7 (1.1)***
	28	0	208.0 (11.4)***	5.6 (0.3)***	7.2 (0.2)***
	35	0	132.0 (16.7)***	5.5 (0.2)***	4.3 (0.3)***
	42	0	119.0 (13.3)*	4.6 (0.1)***	3.1 (0.5)***
	49	0	143.0 (44.4)*	5.1 (0.3)***	4.3 (0.4)***
TT : 124	56	0	106.0 (8.6)**	5.1 (0.5)***	3.3 (0.5)***
Humidity exposure	Time (days)	DII (0/)	Madulus (aN/tan)	Elongation at break (%)	Tanasitas et busels (eN/tess)
Temperature (°C)	Time (days)	RH (%)	Modulus (cN/tex)		Tenacity at break (cN/tex)
25	28	0	194.0 (26.1)	20.5 (3.6)	23.0 (3.7)
		53 75	233.0 (41.1)	23.6 (3.9) 20.7 (4.6)	30.1 (5.2)
		86	231.0 (18.0)	` '	34.1 (7.0)
		100	229.0 (25.4)	23.7 (1.2)	32.1 (7.6)
60	28	0	211.0 (20.0)	22.3 (4.0)	30.2 (2.0)
60	28		224.0 (12.0)	17.7 (4.9) 18.4 (3.9)	27.5 (3.8)
		53 75	230.0 (47.8) 219.0 (5.7)	` /	28.8 (6.6) 29.9 (5.9)
		86		20.8 (8.2)	31.0 (4.8)
		100	232.0 (12.4) 175.0 (11.4)	21.4 (1.1)	` ′
125	20		` ′	20.6 (2.1) 9.0 (0.1)***	23.4 (3.0)* 15.0 (0.2)***
pH immersion	28	0	208.0 (11.4)	J.U (U.1). · · ·	13.0 (0.2)
Temperature (°C)	Time (days)	рН	Modulus (cN/tex)	Elongation at break (%)	Tenacity at break (cN/tex)
25	28	рн 1	191.0 (11.2)	16.8 (1.3)	24.1 (6.6)***
43	20	3	209.0 (6.9)*	22.8 (0.8)*	25.0 (4.0)
		3 7	198.0 (30.8)		30.0 (2.9)
		11	202.0 (6.9)	19.5 (3.1) 19.8 (2.8)	29.8 (2.3)
		13	202.0 (0.9)	` ′	` ′
		13	_	_	_



Table 2 (continued)

)					
60	28	1	_	_	_	
		3	253.0 (12.3)	16.6 (1.6)	28.6 (3.8)	
		7	215.0 (14.8)	23.7 (1.0)	32.6 (3.0)	
		11	224.0 (6.1)	15.7 (1.7)	25.1 (2.4)	
		13	_	_	_	

The figures between brackets are standard deviation

*p<0.05; **p<0.01; ***p=0.000. Asterisks denote significant differences from unaged reference silk

correspond appropriately with the natural degradation undergone by the historic silk.

Thermo-oxidative exposure at higher temperatures (125 °C) reduces break extension and tenacity already from the shortest time exposure with some levelling effects after 28 days. The results for modulus were however different. The modulus after thermo-oxidative exposures at short times was significantly higher than the modulus of unaged reference silk and was not significantly reduced until 42 days exposure. This could be related to physical chain relaxation effects of the silk chains during initial thermo-oxidative exposure, as it is also seen for the samples exposed at lower temperatures (60 °C).

Relative humidity (RH) seems to have very little effect on the mechanical properties of accelerated aged silk. When the two sources of variance, humidity (0, 53, 75, 86, 100 %) and temperature (25, 60, 125 °C), were analysed separately in univariate analysis of variance, only one difference in break extension was significant: 0 % RH in 125 °C (p=0.016). It is therefore the exposure to heat that causes the differences. The three temperatures explain more variance in break extension than the five humidity levels (partial eta squared equals 0.88 and 0.55, respectively). However, as the values demonstrate in Table 2, it is the exposure at 125 °C that has the decisive effect. In modulus, there are no significant differences between exposed and unexposed silk. In tenacity, significant reductions were seen at RH 100 % at 60 °C and at RH 0 % at 125 °C, and in break extension, a significant reduction was only obtained at RH 0 % at 125 °C.

Immersion of the silk samples in extreme pH conditions caused severe effects on their mechanical properties. The exposure at pH 13 at 25 and 60 °C and at pH 1 at 60 °C had such a deleterious effect that the silk could not be tested mechanically. In the remaining exposure conditions, pH 1, 3, 7 and 11 in 25 °C and pH 3, 7 and 11 in 60 °C (Table 2), Young's modulus was not at all significantly affected, and tenacity decreased significantly only after exposure to pH 1 at 25 °C. Break extension was significantly reduced at pH 1 at 25 °C and at 60 °C at pH 1 and pH 13. However, the levels of extension and tenacity even after prolonged exposures were much higher than the historic silk.

Historic silk samples show slightly higher modulus values and much-reduced elongation at break and tenacity in comparison with the reference silk. Indeed, the seventeenth-century silk shows on average a tenacity that is 17 % of the

reference silk. This is a clear indication of the brittleness of the historic silk fibres caused by combined physical (e.g. chain reorganisation) and chemical (e.g. chain scission and oxidation reactions) structural effects during their natural ageing.

The mechanical results when using thermo-oxidative exposure are fairly similar to the fragile historic silk, especially when balancing the high elastic modulus and the reduced elongation at break and tenacity. The reductions in elongation at break and tenacity reach the low level of historic silk when aged for 4 days or more. It seems that competitive or combined physical and chemical degradation occurred in historic silk, leading to such mechanical properties that are difficult to model using accelerated ageing tests. However, thermooxidative ageing at 125 °C allows a certain control of the divergent effects on the elastic modulus and the elongation at break by selecting the exposure times, which may be related to the predominance of physical and irreversible chemical changes. We have already emphasised that break extension and tenacity are the most important properties to achieve for conservation purposes and must have precedence over the elastic modulus. The discrepancy in the modulus values between the historic samples and the artificial aged samples might depend on the sericin remains and the resinous traces found. Therefore, according to the tensile tests thermooxidation is the artificial ageing method that gives silk properties closest to the historic silk.

Integration of the analytical results using exploratory principal component analysis

The analytical markers monitoring the structure and properties of silk were statistically integrated using exploratory principal component analysis (PCA), to obtain correlations between the effect of the different artificial ageing environments and the properties of the historic silk samples. Only 11 artificially aged and historical silk samples were available for the statistical analyses; in them, all the qualitative markers were accessible. These observations include the reference silk, UV irradiation for 4 days, UV irradiation for 10 days, thermo-oxidation at 125 °C for 28 days, thermo-oxidation at 125 °C for 28 days, immersion at pH 1 at 25 °C for 28 days, immersion at pH 13 at 25 °C for 28 days, exposure at 100 % RH at 25 °C for 28 days, King Gustav II Adolf's doublet from 1617, King Gustav II Adolf's breeches from 1617 and King Karl X

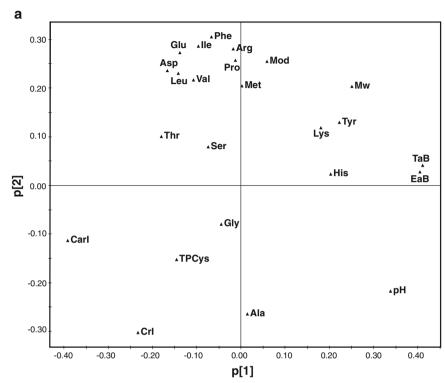


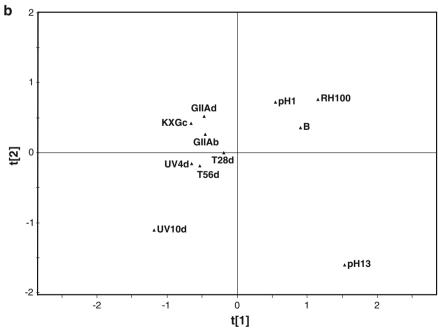
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Gustav's cloak from 1654. The variables (analytical markers) comprised the amino acid composition, carbonyl index, crystalline index, molar mass from SEC, acidity, Young's modulus, elongation at break and tenacity at break (see Electronic

Supplementary Material). Taking into account these results, we decided to implement the exploratory PCA model using a total of 11 samples (observations) and 24 variables (analytical markers), as described in Fig. 7. We are aware of the

Fig. 7 a Exploratory principal component analysis (PCA) of the analytical markers for the degradation of historic and artificially aged silk textiles. Loading plot for the analytical markers (variables) under the first two principal components. These two principal components explain 66.8 % of the total variance. Nomenclature: amino acid composition (as in Table 1), carbonyl index (CarbI), crystalline index (CrI), molar mass from SEC (Mw), acidity (pH), Young's modulus from tensile tests (Mod), elongation at break (EaB), tenacity at break (TaB); b PCA score plot for the silk textile samples (observations) under the first two principal components. Nomenclature: B reference silk, UV4d UV irradiation (4 days), UV10d UV irradiation (10 days), T28d thermo-oxidation (125 °C, 28 days); King Gustav II Adolf doublet from 1617, King Gustav II Adolf breeches from 1617 and King Karl X Gustav cloak from 1654







limitations imposed by the few number of observations related to the large number of variables, which restrict the significance of the multivariate analysis. However, we believe that the exploratory PCA plots may be very useful to integrate the information from the analytical markers of silk degradation and to offer indicative correlations amongst the historic silk textiles and silk samples artificially aged in different environments. The PCA model was created employing SIMCA-P package from Umetrics (Sweden) using the correlation matrix. Four principal components with Eigenvalues larger than unity were derived from the analysis; principal components PC1 to PC4 explained 38.9, 27.9, 11.9 and 9.6 % of the variance in the data set, respectively, and 88.3 % of the total variance altogether. The loading matrix for the different principal components is presented in the ESM Table S1.

Figure 7a shows the principal component loading plot for the different analytical markers distributed in the surface according to their contribution to the two main principal components (PC1 and PC2). Together, these two principal components explain 66.8 % of the total variance. The different amino acid components appear clustered according to their sensitivity towards degradation; the placement of Ser and Thr clustered together is noteworthy, compared with Tyr, Lys and His on another side, and finally, Ala on opposite sides. This distribution of the amino acids in the loading plot correlates well with the effect of the different degradation mechanisms (oxidation and hydrolysis) on the amino acid composition. Similarly, the carbonyl index (CarbI) from FTIR and the tyrosine content (Tyr) are inversely placed on the loading plot, evidencing the direct connection between the decrease of the Tyr content by oxidative reduction and the increase of the carbonyl groups, which may be related to the formation of quinones [25, 26] as discussed in "Amino acid composition." The reciprocal placement of the molecular weight (from SEC) and the crystalline index (from FTIR) may be related to the occurrence of hydrolysis during silk degradation. Indeed, a decrease in the molar mass (from SEC) caused by chain scission of the amorphous regions leads to shorter fibroin chains that may reorganise in crystalline structures, therefore causing an increase in the crystallinity index (from FTIR). This phenomenon is commonly observed in the degradation of synthetic and natural semi crystalline polymers. Finally, the decrease in the mechanical properties (mainly elongation and tenacity at break) correlates well with structural markers such as the decrease of the Tyr content and the molar mass, and the increase of the carbonyl and the crystalline indexes. The alteration of the organisation of silk fibroin at the primary (amino acid composition) and secondary (crystallinity) structural levels and the formation of oxidative moieties (e.g. carbonyl groups) may induce an overall weakening of the structural integrity of the silk fibres. However, these structural changes induced by competitive or combined physical and chemical degradation mechanisms, lead to interconnected

effects on the modulus, elongation and tenacity at break that are difficult to control by accelerated ageing procedures, as stated in "Tensile properties."

Exploratory PCA provides as well interesting information of the effects of the different degradation environments on the properties of silk compared with historic samples in the principal component scores plot of the observations projected on a model hyperplane (Fig. 7b). The samples subjected to 100 % relative humidity (RH100) and extreme pH environments (pH 1 and pH 13) appear dispersed and far away from the historic samples. It is apparent that exposure to RH and immersion into extreme acidic and alkaline pH environments do not induce similar degradation effects that historic silk textiles have undergone. Exposure to RH and immersion in acidic pH are consistently grouped together with the reference silk sample based on the analytical markers; this indicates that these degradation environments do not alter to a great extent the structure and properties of silk textiles. Immersion into extreme alkaline pH solutions, however, seems to cause severe hydrolysis of the peptide chains to an extent that the fibrilar configuration is disrupted and the mechanical properties are lost. On the other hand, thermo-oxidation at high temperatures (125 °C) and UV exposure seem to reproduce in a closer way the analytical markers of historic silk, especially at shorter exposure times. This seems to be caused by a combination of oxidation, chain scission (hydrolysis) and physical processes that occur synergistically to different extents in the artificially aged samples exposed to UV, thermooxidation and in the historic samples. However, prolonged UV exposure seems to induce harsher and divergent degradation effects compared with the historic samples, as indicated by the complete reduction of the Tyr groups (see "Fouriertransform infrared spectroscopy" and "Amino acid composition") and the occurrence of crosslinking (as mentioned in "Size-exclusion chromatography"). From these results, it can be concluded that thermo-oxidative exposure at 125 °C provides artificially aged silk samples with the properties closest to those of historic textiles, and that it is possible to mimic the degree of degradation of the historic silk samples by tailoring the exposure time to thermo-oxidation.

Conclusions

The preservation of our historic heritage requires the multidisciplinary cooperation of experts in art history and conservation science, materials science and analytical chemistry. In this work, suitable analytical markers have been identified to monitor the degree of degradation of historic silk textiles based on the changes in the chemical structure and in the macroscopic properties. Integration of these analytical results is crucial to establish correlations amongst properties but also to obtain fundamental knowledge of the mechanisms of



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deterioration caused by different environments. Accelerated ageing methods are useful to understand the degradation mechanisms to which historic samples are subjected, but their influence must be considered with precaution. This study proved that thermo-oxidation at elevated temperatures (here 125 °C) is the accelerated ageing procedure that best mimics the degradation state of historic silk textiles, and that the degree of degradation can be controlled by the exposure time. Oxidation, hydrolysis, chain scission and chain rearrangements (physical ageing) are shown to be the main degradation mechanisms affecting the structure and properties of silk textiles. The integration of accelerated ageing procedures with the identification of analytical markers proves to be a valuable procedure to support the conservation tasks currently performed in our museums and as a starting point for largescale assessment of the degree of degradation of silk textiles from different historic periods. In addition to this, it provides the basic scientific knowledge for studying the in vitro and in vivo degradation of silk-based biomaterials, with significance in biomedical applications.

Acknowledgments Daniel Limones is gratefully acknowledged for his experimental assistance. The authors are grateful to Dr. Östen Axelsson, Professor Carl-Otto Jonsson and Professor Rolf Sandell for valuable supervision in the multivariate data analysis and to Anna Stow for carefully proof reading the manuscript. JN is grateful to The Royal Armoury for giving access to the museum collection in the study. DVPS would like to thank Anne Blicher at Department of Systems Biology, Technical University of Denmark for running the amino acid analysis as well as René Larsen Royal Danish Academy of Fine Arts Schools of Architecture, Design and Conservation-School of Conservation for support with the data analysis. FV acknowledges the Knut and Alice Wallenberg Foundation for the support to his research position. This research was sponsored by The Gyllenstienska Krapperup Foundation, The Barbro Osher Pro Suecia Foundation, The Royal Society of Arts and Sciences in Gothenburg, and The Sibling Bothéns Foundation.

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Paper IV

Johanna Nilsson. "Evaluation of stitched support methods for the remedial conservation of historic silk costumes." *e-conservation Journal*, 2015, 3. DOI 10.18236/econs3.201506

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Evaluation of Stitched Support Methods for the Remedial Conservation of Historical Silk Costumes

Johanna Nilsson

ABSTRACT

Different textile conservators tend to favour different techniques, mostly according to personal preferences or habit, since there are few empirical studies of the methods' properties. This study investigated how three methods, using different stitching techniques and supporting layers, uphold the maximum force at break of silk samples. The samples were artificially aged, damaged either by cutting a slit to mimic a tear or by abrasion to simulate wear and then subjected to conservation. The studied methods were: securing the damage to a support fabric with laid couching stitches; securing the damage to a support fabric with spaced brick couching; and covering the damage with silk crepeline attached with running stitches. The tensile properties of the surrogates were evaluated after cumulative treatments: conservation, accelerated wear and finally conservation removal. The results show that after conservation, laid couching made both surrogates with tear and with abrasion the strongest though brick couching made surrogates with tear almost as strong. After accelerated wear, the surrogates with tear and laid couching as well as with brick couching were still stronger than the ones with crepeline. After the conservation was removed, however, crepeline in objects with abrasion gave the strongest surrogates of the methods. Laid couching took the longest time to execute and brick couching the shortest. Knowing the properties of different techniques should help in choosing the method that best suits the unique circumstances of each object; for instance, crepeline could be the method of choice when treating much degraded, fragile silk.

1. Introduction

Historical costumes play an important role in the collections of many museums worldwide. They are exhibited to reflect a country's history, a society, traditions and ceremonies or a single person's story. It is important that we can preserve these costumes for the future. When preserving historical textiles, remedial measures are taken to support a fragile and damaged fabric in order to make it last longer by making it stronger. Remedial conservation methods for the preservation of textiles that use stitches in combination with fabrics have not previously been the object of an experimental investigation. Different materials and stitching

techniques employed are described in literature, often as case studies, although the long-term success of such treatments has not been systematically examined [1-2]. However, there are some retrospective studies [1, 3-5], evaluating how the conservation treatments and the objects have withstood time and both Reeves [3] and Himmelstein [4] specifically discuss stitching used in textile conservation. Sahin *et al.* [6] tested a common engineering approach, tensile testing of mechanical properties in textiles, to study how this method can be used in order to understand how the affect of preservation on tapestries. Asai *et al.* [7] analysed how different supports influence the extension and deformation of tapestry

surrogates in order to see how the support methods affect large hanging textiles. Several surveys have studied the use of adhesive treatments in textile conservation [8-10] and some report specialised methods such as fibroin-EGDE to consolidate fragile silk [11]. The explanation of the lack of systematic studies of stitching technique may be that stitching is a traditional method that has been used for many years and is regarded as generally harmless and reversible even though this has been questioned by a few [3-4], whereas the non-traditional use of adhesives on textiles has raised concerns in what has been a more conservative conservation specialisation [2].

The most common types of damage occurring in silk costumes, making it necessary to conserve them, are frayed surfaces due to abrasion caused by repeated frictional contact over time and rips or tears resulting from sudden movement of a degraded, fragile fabric or because the silk is weighted. Although fragile, many historical costumes are in any case displayed in exhibitions and are lent to museums around the world. This leads to unavoidable handling before and during transport, during mounting on mannequins for display and photography. This handling further weakens an already degraded material even if it is conserved.

When deciding whether and how a costume should be conserved, it is important to have an informed idea of both the short- and long-term outcome. Various stitching methods in combination with a fabric continue to be the most common physical stabilisation techniques in textile conservation. Therefore, it is important to examine and evaluate their preserving effect, their durability and the potential damage they may cause to museum textiles. Moreover, in an era of cost cutting it is also important to document and compare the time needed to execute the treatments.

The main goal of this study is to assess how three selected conservation methods, commonly used in the conservation of historical costumes, affect the strength of surrogates for historic silk by determining maximum force at break. The surrogates are artificially aged silk samples that have similar fragility to the silk fabric in authentic 17th century silk costumes and are intentionally damaged by tear or abrasion [12]. How the strength is affected in the damaged and conserved areas when subjected to accelerated wear, corresponding to many years of handling is also studied. The study also investigates how the conservation methods affect the surrogates by comparing them with each other after the conservation has been removed following accelerated wear. Included in the study is also a comparison of how long each method takes to execute.

2. Selection of Textile Conservation Methods

To establish which conservation methods are most commonly used in the preservation of costumes, and therefore important to evaluate, the popularity of various conservation methods was assessed in a questionnaire-based survey. The questionnaires were sent to textile conservators in Europe and North America [13]. Furthermore, conservation reports describing the conservation at the Abegg Foundation (Switzerland) of 23 predominantly silk costumes from the early 17th century were studied. All conservation reports (a total of 819) concerning the conservation of costumes at the Royal Armoury in Stockholm from the 1890s and onwards were also studied.

In all instances, the most common method used is to lay the fragile textile on top of a support fabric placed aligned with the fabric grain and to then secure the damage with laid couching onto the support fabric. Laid couching is a long straight

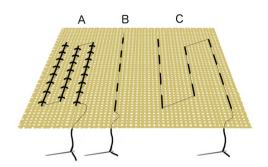


Figure 1. Examples of common stitches used in textile conservation: laid couching (A), running stitch (B) and brick couching (C). Illustration by Isak Nilsson.

stitch, laid in line with either the warp or weft of a fabric and then fastened in place with short perpendicular holding stitches, inserted at regular intervals (Figure 1A) [1, 14]. Another common method is to protect a damaged and frayed area by a covering layer of silk crepeline that is attached to the fabric with running stitches along the edges of the crepeline (Figure 1B). Usually, when used on costumes, the shape of the crepeline is adapted to be able to fix it along its edges at existing seams or lines in the fabric. When a piece of crepeline covers a larger area it is common to further attach it with spaced rows of running stitches to keep it close to the underlying fabric to avoid bagging or wrinkling in the crepeline. A third method was used on several major conservation interventions at the workshop of the Royal Armoury in Stockholm on costumes with fabrics of plain silk or silk in combination with metal wires, flat strips or wool. The fragile textile is laid on top of a support fabric that is placed aligned with the fabric grain and the damage is secured with brick couching of various spacing onto the support fabric. Brick couching is a short stitch laid perpendicularly over one or several warp or weft threads sewn to form a regular pattern like brick-work (Figure 1C). Brick stitch is described by several authors [15-17] as a technique commonly used in tapestry

conservation; warp couching and couching are also mentioned as stitches used in tapestry conservation and they are related to brick couching. Based on the survey and the consulted treatment reports it was decided to evaluate and compare three methods: method 1 (M1) - a damage is secured onto a support fabric with laid couching; method 2 (M2) - a damage is secured onto a support fabric with spaced brick couching; and method 3 (M3) - a damage is covered with silk crepeline which is attached with running stitches. A video explaining in detail how the methods were carried out for this study was produced [18].

3. Experimental

Artificially aged silk specimens to be used as surrogates for 17th century silk were exposed to cumulative experimental treatments in four steps: mechanical damaging, conservation with three methods (M1 laid couching, M2 brick couching and M3 crepeline), accelerated wear, and removal of the conservation. After each step, the maximum force at break of the surrogates was determined.

Preparation of the specimens and surrogates and the conservation interventions were carried out by the author and her colleagues at the Royal Armoury's workshop in Stockholm. The abrasion, accelerated wear and tensile testing were carried out at an accredited research institute, Swerea IVF AB and the work was executed in cooperation between the author and one of the company's textile engineers.

3.1. Materials

Standard silk fabric ISO 105-F06:2000 *Bombyx* mori was selected for the silk fabric to be investigated in this study, both for the specimens and

for the support fabric in M1 and M2. Silk crepeline was used as cover in M3. The stitching in all three methods was performed with double-ply silk thread (supplied by Silke-Annet) using a Milward beading needle No. 15.

Circular specimens (160 mm in diameter) were stamped out of the standard silk fabric (size and shape of specimens chosen to conform to testing requirements of the Martindale abrasion system). Rectangular specimens (120 x 200 mm) were cut with the longer length in the warp direction. A total of 60 specimens were stamped for each shape.

3.2. Methods

3.2.1. Accelerated Ageing

Prior to ageing, 55 silk fabric specimens of each shape were dried in a desiccator over silica gel until the relative humidity was stabilised at 4% [19]. They were then suspended in 1000-mL borosilicate glass bottles using the screw-top polypropylene caps to secure the suspending thread that was attached to the circular specimens so that they hang vertically in the warp direction. The rectangular specimens had a similar thread attached at one of their short ends. To the bottom of each bottle 42 g calcium chloride (CaCl₂) was added to maintain a low relative humidity. Ageing was undertaken in two different cycles due to the limited capacity of the available oven, a Termaks TS 7472. The specimens were aged for 42 days at a mean temperature of 125°C. The ageing method, thermo-oxidative ageing, was developed earlier by Nilsson et al. [12].

3.2.2. Damages

Two types of damage, a tear or an abrasion were created on the aged surrogates to make them similar to historical textile objects in need of conservation. Physical harm caused naturally is normally more complex than this with a combination of different damages but simplification was necessary. In the centre of each rectangular surrogate a tear was made by cutting a 50 mm long slit with a scalpel in the weft direction (Figure 2A). On the circular surrogates a frayed surface was created by abrasion according to SS-EN ISO 12947-2:19991 using the Nu-Martindale abrasion apparatus (James H Heal & Co Ltd) at a pressure of 12 kPa (Figure 2B). The resulting damaged area was 80 mm². The abrading process was discontinued when the surrogate was clearly frayed and holes had developed. This occurred between 141 to 1004 rotations with an average of 418 turns per surrogate. The aged surrogates were degraded by either tear or abrasion, 50 of each kind.

3.2.3. Securing the Damage in M1

A support fabric 95 x 95 mm was centred and aligned under the damaged area of the surrogate, the 50 mm long slit and the 80 mm² abraded area respectively. The support fabric was secured with c. 1 mm long running stitches on the face side and c. 5 mm long running stitches on the reverse close to its edges giving a total of c. 150 needle insertions.

The damaged area of the surrogate was secured with laid couching. On average 11 vertical long stitches with c. 7 mm spacing were sewn over the damage, the length of the stitches adjusted to the damage. Perpendicular stitches c. 1 mm long with a spacing of c. 4 mm were sewn over the long stitches. The vertical long stitches on the surrogates

 $^{^1}$ The standard ENISO 12947-2:1999 has the high uncertainty of \pm 21% which clearly illustrates the complication of trying to produce uniformly damaged surrogates by abrasion with this method.

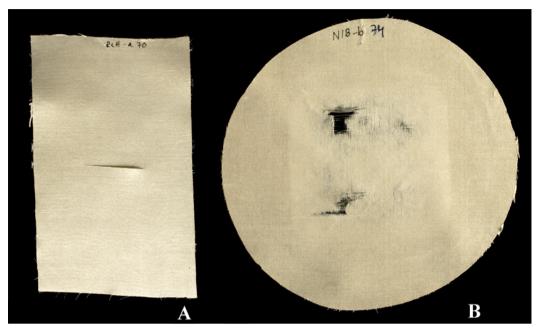


Figure 2. Aged and damaged surrogates before conservation: surrogate with tear (A) and surrogate with abrasion (B). Photo by Östen Axelsson.

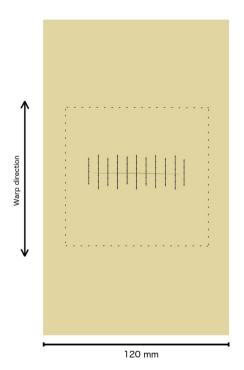


Figure 3. Surrogate with tear, conserved with M1. A support fabric is centred under the tear and attached with running stitches, represented by the dotted lines, and the tear is secured with laid couching. Illustration by Isak Nilsson.

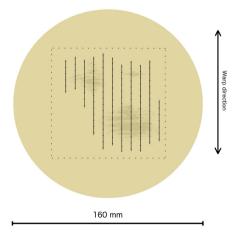


Figure 4. Surrogate with abrasion, conserved with M1. A support fabric is centred under the abraded area and attached with running stitches, represented by the dotted lines, and the damage is secured with laid couching. Illustration by Isak Nilsson.

with tear were by turns c. 25 mm and c. 20 mm long (Figure 3). The length of the vertical stitch on the surrogates with abrasion varied depending on the damage shape (Figure 4). The amount of

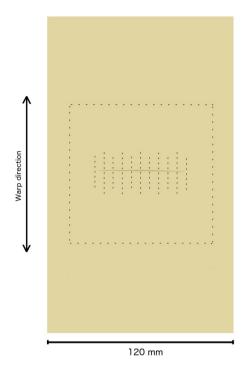


Figure 5. Surrogate with tear conserved with M2. A support fabric is centred under the tear and attached with running stitches, represented by the dotted lines, and the tear is secured with spaced brick couching. Illustration by Isak Nilsson.

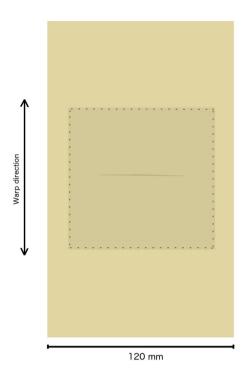


Figure 7. Surrogate with tear conserved with M3. The crepeline, illustrated by the darker square, is attached by running stitches, represented by the dotted lines, onto the surrogate. Illustration by Isak Nilsson.

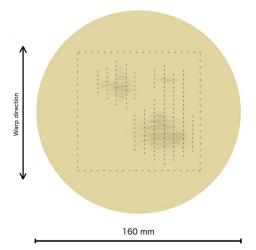


Figure 6. Surrogate with abrasion, conserved with M2. A support fabric is centred under the abraded area and attached with running stitches, the dotted lines, and the damage is secured with spaced brick couching. Illustration by Isak Nilsson.

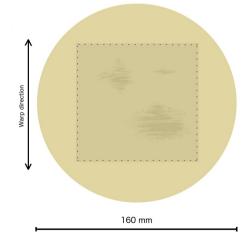


Figure 8. Surrogate with abrasion, conserved with M3. The crepeline, illustrated by the darker square is attached by running stitches, represented by the dotted lines, onto the surrogate. Illustration by Isak Nilsson.

needle insertions for the surrogates with tear is c. 320, and for the surrogates with abrasion c. 600 (including the 150 needle insertions for the attachment of the support fabric). This method was carried out on 15 surrogates of each type of damage.

3.2.4. Securing the Damage in M2

A support fabric was aligned and secured to the surrogate in the same way as in M1 up to securing of the damage.

The damaged area of the surrogate was secured with spaced brick couching. The stitches formed on average 11 rows in the warp direction on the surrogates with c. 5 mm spacing. The stitches on the face side were c. 1 mm long and c. 4 mm long on the reverse. The length of the stitching rows on the surrogates with tear was by turns c. 25 mm and c. 20 mm (Figure 5). For surrogates with abrasion, the length of the stitching rows varied depending on the form of the damage (Figure 6). The amount of needle insertions for the surrogates with tear is c. 300 and for the surrogates with abrasion c. 550 (including the 150 needle insertions for the attachment of the support fabric). This method was carried out on 15 surrogates of each type of damage.

3.2.5. Securing the Damage in M3

The damaged area was covered by crepeline. The edges of a 120 x 120 mm square of silk crepeline were folded to create a 95 x 95 mm square piece of fabric that was centred and aligned over the damaged area. The fabric was attached, close to the edge of the crepeline with c. 1 mm long running stitches on the face side with c. 5 mm spacing giving a total of c. 155 needle insertions (Figures 7 and 8). This method was carried out on 15 surrogates of each type of damage.

3.2.6. Accelerated Wear

Many historical costumes have a lining, which acts as a protection and support to a costume's outer fabric. To make the accelerated wear more natural, the surrogates were given a temporary lining of standard silk, ISO 105-F06:2000 Bombyx mori, attached by running stitches close to their edges. The lining protected the reverse of the surrogates and the accelerated wear was concentrated to the face side. The accelerated wear was accomplished by washing the surrogates at 60° C according to EN ISO 6330:2012 for 30 minutes, thereafter they were tumbled in a tumble dryer, Electrolux T2130, according to EN ISO 6330:2012 (20°C) with a 2 kg polyester makeweight² for a period of 660 minutes plus additional 60 min in 50°C; the makeweight was re-wetted after every 180 minutes. The lining was removed after the accelerated wear was completed.

Normal museum handling is much less severe than this accelerated wear. This treatment was necessary in order to speed up the effect, trying to mimic natural handling over a long period of time. Of the 110 aged surrogates, accelerated wear was carried out on 30 surrogates with tear and 30 with abrasion. Ten of each were conserved with either M1, M2 or M3.

Figure 9 shows surrogates with abrasion before and after accelerated wear: the damage area is larger, the crepeline has become distorted and there is a loss of material which might be considered too great compared to naturally worn textiles. For comparison, however, Figure 10 and 11 show an antependium and a chasuble in silk that have undergone a great deal of handling after the

² Makeweight is used to keep the material in an even process and to give the material a certain weight according to the standard.

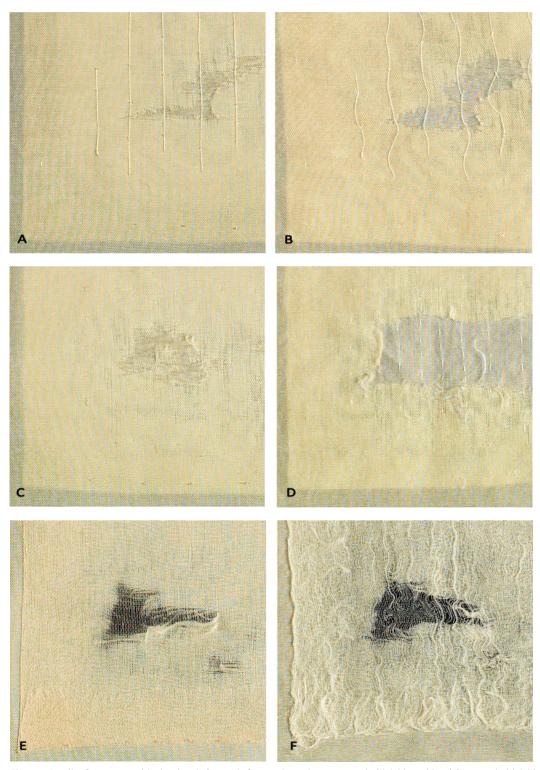


Figure 9. Details of surrogates with abrasion, before and after accelerated wear: secured with laid couching (A); secured with laid couching after accelerated wear (B); secured with spaced brick couching (C); secured with spaced brick couching after accelerated wear (D); covered with silk crepeline (E); and covered with crepeline after accelerated wear (F). Photo: Erik Lernestål.



Figure 10. Detail of an antependium by Agda Österberg 1931, Christiane Church. The conservation with laid couching has become worn by time and the antependium is in need of re-conservation. Photo by Ingeborg Skaar.



Figure 11. Detail of a chasuble by Sten Kauppi 1970, Sunne Church. The stitches from the conservation with brick couching are left and parts of the fragile silk are gone. Photo by Ingeborg Skaar.

conservation with laid couching (ante-pendium) or brick couching (chasuble). These naturally worn objects, just like the surrogates exposed to accelerated wear, show a severe loss of material supporting the validity of the achieved level of accelerated wear. The antependium's laid couching is partly damaged while the brick couching on the chasuble is intact.

3.2.7. Tensile Tests

To determine the ageing effect and to ensure that the tenacity was similar to that of 17^{th} century silk, found by Nilsson [12], weft yarn was tested on a Vibroscope/Vibrodyne device (Lenzing, Germany). Ten weft yarns of a circular surrogate and ten from a rectangular one were tensile tested. The samples were conditioned at $20 \pm 2^{\circ}$ C and 65 \pm 3% RH 24 h prior to testing. The Tex³ value was calculated by taking out ten yarns from the weft of each sample, measuring the average length and

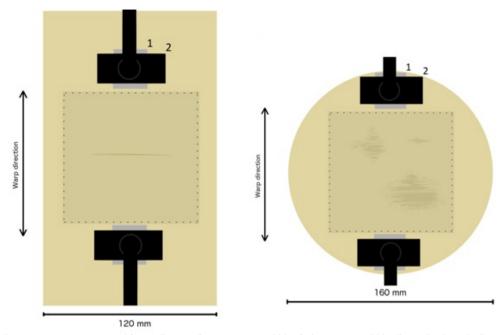


Figure 12. Surrogates mounted for tensile test: 1) Insert, 25 mm width, 2) Clamp 50 mm width. Illustration by Isak Nilsson.

weight of the yarns. For the yarn tensile testing the gauge length was 20 mm, the test speed 20 mm/min and the tension weight 2000 mg following DIN FN ISO 5079.

Tensile tests of the fabric surrogates were performed according to EN ISO 13934-2:1999, the grab method. Equipment was an Instron 5966 (Instron, USA). The surrogates' size follows the testing standard and they were conditioned for 24 h prior to testing in an atmosphere of 20±2 °C and 65±3% RH. Each surrogate was fastened between clamps, 5 mm from the support fabric and the crepeline (Figure 12), the gauge length being 100 mm and the test speed 50 mm/min. The force was applied in the warp direction of the surrogates, the same direction as the rows of the laid couching

Every combination of the two types of damage, the three conservation methods and the four steps of treatment forms a set of conditions. Five unaged specimens and five surrogates of each set of conditions were tensile tested and submitted to statistical tests. For the significance tests, One-way ANOVA was performed to ensure that reliable effects were obtained. When the general test was significant, pair-wise differences were analysed using LSD or Tamhane's T when the two variances were similar or dissimilar, respectively (SPSS Statistics v22).

4. Time Required to Execute the Interventions

In order to measure the time it took to carry out the conservation interventions, another 24 surrogates (12 of each type of damage) were conserved

and brick couching stitches. The surrogate was extended until it started to rupture, defining the maximum force at break.

 $^{^3}$ The linear density of thread is a measurement of its fineness and is expressed in Tex units, which is the weight in grams of 1000 m thread.

with Methods 1, 2 or 3 as described above. Four surrogates with the same type of damage were conserved with each method. The conservation was executed by Augusta Persson, employed at the Royal Armoury's workshop at the time, and the author. Each conservator treated two surrogates with each method and damage. Before conservation, the surrogates were lined with the standard silk ISO 105-F06:2000 Bombyx mori. This step was taken as historical costumes usually have a lining and it is more complicated and time consuming to conserve a fabric that is lined as it is preferable not to stitch into the lining. Therefore, the comparison between the times used to execute the different support methods with a lining will be realistic. Laid couching took on average 68 min to execute while brick couching took 40 minutes and crepeline 56 minutes.

5. Results

5.1. Effect of Ageing, Damaging, Conservation and Accelerated Wear

The yarn in the artificially aged surrogates had on average 17.5% of the tenacity of unaged standard silk. This fragility is close to the Nilsson *et al.* finding that silk yarn from 17th century costumes had 17% of the tenacity of unaged standard silk [12].

Before proceeding to the main question, if there are significant differences between the effects of the three conservation methods, the general state of the silk surrogates after each step of the cumulative experimental treatment (conservation, accelerated wear and removal of conservation) will be accounted for in relation to original, aged surrogates (Figures 13 and 14).

The damage by tear or abrasion left only 5% of the relative maximum force at break, and upon

conservation the strength was increased four times to 20%. The subsequent accelerated wear left about 9% and when the conservation was removed. 4% of the relative maximum force remained.

5.2. Maximum Force at Break After Conservation

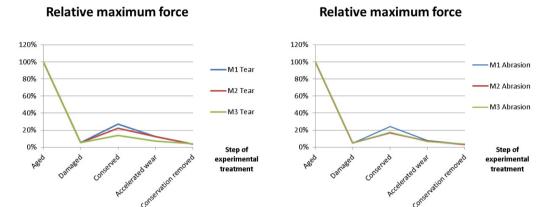
One of the running stitches in the weft direction, which attached the support fabric or the crepeline to the surrogate, was most often first to break during tensile testing of the conserved surrogates. This was followed by breakage of the thread in the laid- or brick couching. Not until a major part of the couching had been disrupted, would the surrogate itself begin to break.

For conserved surrogates with tear, both laid couching and brick couching increased maximum force at break significantly more than crepeline (p=.003 and .026, respectively). The maximum force at break of the conserved surrogates with tear became more than one and a half time stronger with crepeline, three times stronger with brick couching and more than four times stronger with laid couching (Figure 15).

For conserved surrogates with abrasion, laid couching was superior to both brick couching and crepeline (p=.001 and .003, respectively). The maximum force at break for the surrogates with abrasion, conserved with crepeline or brick couching became two and a half time stronger and with laid couching almost four times stronger (Figure 16).

5.3. Maximum Force at Break After Accelerated Wear

After the conserved surrogates had been subjected to accelerated wear, there were significant differences only for surrogates with tear. Both laid couching and brick couching had significantly



Figures 13 and 14. Relative maximum force at break after each step of experimental treatment for M1, M2, M3 and each damage. For comparison reason the maximum force at break for the aged, undamaged silk is set as the relative value of 100% (which is 17.5% of the strength of unaged standard silk). In Figure 14, the green and red line override each other being thus only visible one line.

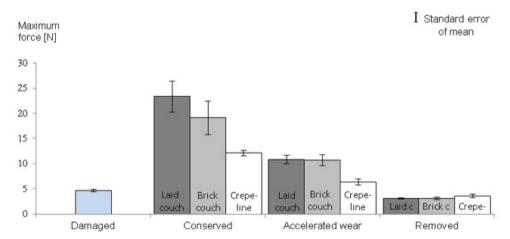


Figure 15. The maximum force at break of surrogates with tear without any conservation and after conservation with three different methods, after being subjected to accelerated wear and after removal of the conservation.

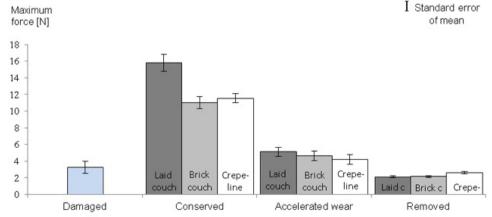


Figure 16. The maximum force at break of surrogates with abrasion without any conservation and after conservation with three different methods, after being subjected to accelerated wear and after removal of the conservation.

higher maximum force at break after accelerated wear than crepeline (p= .003 and .005, respectively). The strength of the surrogates was about halved by the accelerated wear (Figure 15).

When visually inspecting the material loss of the surrogates that have been subjected to accelerated wear, it is impossible to separate the surrogates with abrasion with laid couching and those with brick couching from each other. It is clearly visible though, that the frayed material of the surrogates with abrasion, conserved with crepeline has lost less material than the other two. The crepeline has become ruined by the accelerated wear but seems to have had a function as a protection and a sacrificing layer.

5.4. Maximum Force at Break After Removal of the Conservation

After accelerated wear followed by removal of the conservation, there are significant differences only for surrogates with abrasion. Surrogates treated with laid couching and brick couching were significantly weaker after the conservation had been removed than surrogates conserved with crepeline (p=.015 and .017, respectively). The stitching with laid couching and brick couching decreased more than half the strength of the surrogates while crepeline decreased less (Figure 16).

6. Discussion

The artificial ageing method used in this study creates a fabric with fragility similar to examples of 17th century silk from the costume collections of the Swedish Royal Armoury. The results of this study may therefore be applied to silk costumes from that period and to other silk fabrics of similar fragility.

Laid couching and brick couching were sewn with the same number of rows in the study. This similarity is important for the comparison between the methods. As Asai et al. [7] have demonstrated, the extension of tapestry surrogates may be affected by the number of sewn rows. According to the preliminary data obtained in the present study, extension and maximum force at break show high correlation at supportive conservation. When maximum force at break has a high value. the value for extension is low and vice versa. The importance of sewn rows is also indicated by the observation that the first to break are the running stitches. The reliability of the study can be further supported by the fact that laid couching and brick couching were sewn with almost similar number of stitches, laid couching was sewn with only 7-9% more stitches; the differences are so small that they probably do not explain the top positions of laid couching.

The relatively great standard errors of mean of the conserved laid couching and brick couching samples should be noted. They tend to obscure reliable differences.

It is evident that conservation, especially with laid couching and brick couching, greatly increases the maximum force at break of damaged surrogates. However, the damaged and conserved surrogates had on average only 20% of the strength of aged surrogates that had not been damaged. In comparison, laid couching had a strengthening effect of 27% on surrogates with tear and 24% on surrogates with abrasion, while the corresponding values for brick couching were 22% / 17% and for crepeline 14% / 18%. Thus, the performed conservation interventions are far from restoring the damaged surrogates to their original strength. This result may be disappointing but it might as well be considered an advantage. If a conserved fragile textile is exposed to stress, the conservation will likely break before the undamaged area of the textile is harmed. This perspective is supported by the observation that the first to break are the running stitches and then the couching before the surrogate itself is visibly affected. The amount of rows of stiches probably affects the strength of the conservation intervention. These are important aspect in decision making concerning what conservation method to choose. This is a topic for further investigation.

Both at intact conservation and after accelerated wear, laid couching gives the strongest surrogates and brick couching is second best. However, there seems to be a slight trend that surrogates with tear as well as with abrasion, conserved with laid couching or crepeline are more negatively affected by accelerated wear than brick couching. The thread is probably more exposed and sensitive to wear with laid couching stitches, as is the crepeline placed on top of the damage; this might cause differences in resistance. After the conservation has been removed, the strength is greater for surrogates conserved with crepeline than those with laid couching or brick couching. This may be explained by the lower amount of needle insertions needed by the crepeline method, but also by the fact that crepeline acts as a protecting layer of the aged and damaged silk. After accelerated wear crepeline has become ruined but seems to have had a function as a protection and sacrificing layer.

Under these simplified conditions, brick couching was the conservation method that required the shortest time to execute, followed by crepeline while laid couching was the most time consuming. However, as type of object, damage and material can vary a lot when working with real textiles, the time for executing the conservation might differ considerably from those registered here. There may also be considerable differences between conservators with different experiences and skill.

7. Conclusions

This study assessed how three common textile conservation methods affect maximum force at break of silk surrogates with similar fragility as the silk fabric in authentic 17th century costumes. The surrogates were damaged by tear or abrasion and then submitted to three experimental treatments: conservation with one of three stitching methods, accelerated wear, and removal of the conservation. Strength was tested after each treatment. Included in the study is a comparison of how much time each method took to execute.

The three conservation methods were laid couching, brick couching, and conservation with crepeline. After conservation, laid couching made both surrogates with tear and with abrasion the strongest though brick couching made surrogates with tear almost as strong. After accelerated wear, the surrogates with tear and laid couching as well as with brick couching were still stronger than the ones with crepeline. After the conservation was removed, however, crepeline in objects with abrasion gave the strongest surrogates of the methods. Laid couching took the longest time to execute and brick couching the shortest.

It is important that the silk fabric to be conserved is in such conditions that it withstands extensive stitching if laid couching or brick couching is chosen. If a very fragile silk is to be conserved, crepeline is suggested to be the best method as the amount of stitches is low and the crepeline still has a good protective effect for surrogates with both types of damage. In addition, crepeline affects the strength of fragile silk less than the other two methods, as shown after removal of the conservation interventions. A harmless conservation is especially important when conservation may have to be replaced in due time.

Further tests should follow as there are several more combinations of materials, conservation methods, support materials and conservators with different experiences and skills to be investigated. Also, other effects of the conservation interventions such as on extension and modulus are interesting to study. The aesthetical appearance of the conservation is important and should be evaluated. The tentative interpretation of some data in this study that brick couching is less vulnerable to accelerated wear than laid couching or cover with crepeline must be further investigated.

8. Acknowledgments

The author is very grateful to the Swedish Royal Armoury and Skokloster Castle and the Hallwyl Museum Foundation, the Department of Conservation from the University of Gothenburg and the following funding bodies: The Gyllenstiernska Krapperup Foundation, The Barbro Osher Pro Suecia Foundation, The Royal Society of Arts and Sciences in Gothenburg and The Sibling Bothéns Foundation. Abegg Foundation is thanked for generously sharing their conservation reports. Prof. Carl-Otto Jonsson, from the Stockholm University's Department of Psychology, is thanked for the statistical analysis, and Isak Nilsson, from the School of Engineering Sciences at the Swedish Royal Institute of Technology for the illustrations. Augusta Persson, from the Royal Palaces, is thanked for her conservation work, and Ingeborg Skaar, from Textilvård, is thanked for her photographs. Thanks also go to independent conservator Anna Stow, for checking the language.

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List of suppliers

Standard silk ISO 105-F06:2000 *Bombyx mori* from Cromocol, Åsboholmsgatan 16, SE-504 51 Borås, Sweden.

"Hair fine silk", originally weaving yarn imported from France. Silke-Annet, Dorthesvej 2, Dk-3520 Farum, Denmark.

Silk crepeline, Talas, 330 Morgan Ave, Brooklyn, NY 11211, US.

Glass bottles from se.vwr.com art. no 215-1595. Calcium chloride 99% purity from Sigma Aldrich USA.

Paper V

Johanna Nilsson and Östen Axelsson. "Attributes of aesthetic quality used by textile conservators, evaluating conservation interventions on museum costumes.", *Perceptual and Motor Skills* 2015, 121, (1), p. 199-218.

DOI 10.2466/27.24.PMS.121c10x7

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ATTRIBUTES OF AESTHETIC QUALITY USED BY TEXTILE CONSERVATIONS IN EVALUATING CONSERVATION INTERVENTIONS ON MUSEUM COSTUMES^{1,2}

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Summary.—Aesthetic quality is central to textile conservators when evaluating a conservation method. However, the literature on textile conservation chiefly focuses on physical properties, and little is known about what factors determine aesthetic quality according to textile conservators. The latter was explored through two experiments. Experiment 1 explored the underlying attributes of aesthetic quality of textile conservation interventions. Experiment 2 explored the relationships between these attributes and how well they predicted aesthetic quality. Rank-order correlation analyses revealed two latent factors called Coherence and Completeness. Ordinal regression analysis revealed that Coherence was the most important predictor of aesthetic quality. This means that a successful conservation intervention is visually well-integrated with the textile item in terms of the material and method.

Museums all over the world use costumes to reflect a trend, a society, a country's history, or a single person's story. According to the International Council of Museums' code of ethics [International Council of Museums (ICOM), 2013], museums are responsible for protecting natural, cultural, and scientific heritages by actively acquiring, preserving, and promoting their collections. This idea of conservation emerged during the nineteenth and twentieth centuries as awareness arose that certain skills and

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²This research was sponsored by the Gyllenstienska Krapperup Foundation, the Barbro Osher Pro Suecia Foundation, the Royal Society of Arts and Sciences in Gothenburg, and the Sibling Bothéns Foundation. The first author is very grateful to the Royal Armory, Skokloster Castle, and the Hallwyl Museum, and to the Department of Conservation, University of Gothenburg, for supporting the study. A great thanks goes to all the textile conservators who so generously and with engagement volunteered in the study, and to the photographers as well as the illustrator. During the time this paper was written, the second author was a Newton International Fellow at the School of Architecture, University of Sheffield in the UK. The fellowship was sponsored jointly by the Royal Society and the British Academy. Special thanks go to Professor Ola Wetterberg, Professor Carl-Otto Jonsson, Robert Nilsson, and five anonymous reviewers for valuable comments on the text.

approaches were necessary to preserve historic objects correctly (e.g., Muños Viñjas, 2005). These skills are essential to modern museums.

In the mid-twentieth century, the aestheticist conservation theory became popular. It originated from the art historian Cecare Brandi and is centered on the notion of aesthetic integrity of an object with an artistic value, while at the same time emphasizing that the imprints that history has left on the object must be preserved, and that the conservation intervention must be subordinate to the object (Muños Viñjas, 2005). On a similar note, Appelbaum (2007) argued that items are preserved because they have different kinds of values that must be taken into account when deciding on conservation interventions. Aesthetic value is one of these.

Brajer (2009) remarked that there is a contemporary trend in conservation of art, representing a move away from a science-based approach that prioritizes the preservation of the material toward emphasizing the aesthetic aspects relating to presentation and appearance, to improve the object's meaning to the public. Similarly, Cloonan (1995) highlighted the dilemma of whether the aesthetic or the practical properties of a book should be prioritized in conserving book bindings. This dilemma was accentuated in the twentieth century when artists began binding books—bindings that may be artistic, but not necessarily durable. At the core of the dilemma is the question of whether and when to save an original binding. Along the same lines, Lennard (2006a) argued that the physical structure and the image (i.e., appearance) are equally important in conservation of tapestries (see also Lennard, 2006b; Lennard, Baldursdóttir, & Loosemore, 2008). This notion ought to be valid also to conservation of historic costumes, which is the topic of the present paper.

When conserving historic textiles, remedial measures are taken to support a fragile and damaged fabric in order to make it last longer. Typically, a support fabric is stitched on underneath the damage to secure it and to prevent it from further deterioration (cf. Takami & Roberts, 2011). Remedial measures influence the appearance of the object, and the visual aesthetic quality of the conservation intervention largely depends on the judgment and craftsmanship of the conservator, who, alone or in collaboration, decides what conservation material and method to use. As an illustration, Fig. 1 presents a shoe before and after conservation.

ICOM (2013) stated that conservation interventions should be as reversible as possible. Therefore, they must be documented and possible to distinguish from the original object. In practice, conservation interventions are not necessarily made visible to laypersons. However, it must be possible for a conservator to spot a conservation intervention and to find out from the documentation what measures have been taken to preserve the object. These demands may require a compromise between techniques used and the aesthetics upheld in conservation.





Fig. 1. Shoe in silk and silver fabric, before and after conservation (top and bottom panel, respectively), illustrating the visual effect of a conservation intervention executed 2010 (Inv. no. Lrk. 21538). Courtesy of Ida Areklett Garmann (top panel) and Jens Mohr (bottom panel).

Nilsson (2005) conducted an international survey to investigate (1) what types of conservation interventions have been, and are being used on costumes; (2) which of them are the most common; (3) which criteria are used to determine whether or not an intervention is successful; and (4) what factors determine whether or not an intervention should be performed. A questionnaire was sent to 51 conservation workshops in Europe and North America. Out of these, 32 workshops (16 in Sweden, 15 in other European countries, and 1 in North America) provided useful answers, while 8 replied that they did not have any textile conservation activities or that the conservator was newly appointed, and 11 did not reply at all. For the purpose of the present paper, the most interesting responses concerned the question about which criteria are used to determine whether an intervention is successful. The two most important criteria were that an intervention must be aesthetically appealing (18 workshops affirmed) and that the intervention must be good and provide a durable support that prevents further damage to the textile (16 workshops affirmed).

Because the literature on textile conservation chiefly focuses on the physical properties of the conservation methods, the results of the questionnaire study were somewhat unexpected, because they indicated that textile conservators emphasized aesthetics equally to physical properties. Thus, in agreement with the aestheticist conservation theory, they not only take technical aspects into consideration in their work, but also decide what conservation methods and materials to use based on aesthetic preferences. Consequently, it is important to followup the results of the survey and investigate what textile conservators consider 'aesthetically appealing' and potentially what properties of conservation interventions determine visual aesthetic quality according to textile conservators.

Although the literature on textile conservation chiefly focuses on the physical properties of the interventions, there is a discussion on aesthetics in terms of the match between the conservation material and the original object, in both color and structure, or in terms of how the craftsmanship or the presentation of an item in an exhibition determines its appearance (e.g., Flury-Lemberg, 1988; Landi, 1992).

Kaldany, Berman, and Sigurdardottir (1999) evaluated aesthetic qualities of artistic coloring materials used to create compensation infill for losses in textile items. For this purpose, they first defined six characteristics of the coloring materials as aesthetically important: (1) it must be able to reproduce fine lines on fabric; (2) lines must remain sharp and should not spread out and form a feathered edge; (3) it must result in an even layer; (4) it must not stiffen the fabric; (5) thickness of the film layer must be controllable; and (6) the fabric should remain supple. It is not clear from the text who performed these aesthetic evaluations.

Conti (2011) described a new support and stitching technique developed in her workshop to achieve a more aesthetic conservation of damask brocade. Conti claimed that unlike other conservation methods commonly used on this type of fabric, this new method does not flatten the textile nor increase the shadows (i.e., it does not change the shape of the original textile), which would make the conservation too apparent. The new method is said to have several advantages in how it preserves the original structure and design of the fabric and its pattern.

Gill (2006) conducted a systematic comparison of four stitching patterns with regard to their visual effects, their effectiveness in stabilizing and preserving a damaged textile, and the time taken to implement them. As with Kaldany, *et al.* (1999), it is unclear who performed the aesthetic evaluation.

The three preceding references suggested that according to textile conservators, an aesthetically successful intervention is visually well-integrated with the original textile item. However, this remains to be investigated empirically.

The purpose of the present study is to investigate what textile conservators mean when they say that a conservation intervention is aesthetically appealing and which properties of the interventions determine the visual aesthetic quality, according to textile conservators. This is done in interdisciplinary collaboration, using a psychological, experimental approach, modeled on Axelsson's (2007a, 2007b, 2011) research on aesthetic appreciation. This paper reports two experiments. In the first experiment, the underlying attributes of aesthetic quality of 33 photographs of textile conservation interventions were explored with the support of 24 textile conservators from the Swedish Association for Textile Conservation. These attributes were assumed to represent the determinants of aesthetic quality of the interventions. In the second experiment, the relationships between the attributes identified in Exp. 1 and the aesthetic quality of the 33 interventions were investigated with the support of 10 textile conservators, who had not taken part in Exp. 1.

Experiment 1

SORTING AND EXTRACTING OF ATTRIBUTES

Method

Participants.—The participants were 24 randomly selected members of the Swedish Association for Textile Conservation (1 man, 23 women; M age=51.4 yr., range=30–77). On average, they had been active as textile conservators for 17 yr. (range=0.5–37). In total, the association had 58 registered members at the time of the selection. All the participants volunteered for the study and were not reimbursed.

Material.—For practical reasons and in order not to harm the delicate textile objects through excessive handling, the materials consisted of 33 matte, color prints of digital photographs, each depicting an individual conservation intervention on a textile object from the collections of the Royal Armory or the Hallwyl Museum in Stockholm, Sweden. All interventions were photographed under exactly the same conditions—including identical lighting—with a Canon EOS 5D camera (2,912×4,368 pixels), using a Canon Compact-Macro EF 50 mm f/2.5 lens. All photographs (RGB color; 8 bits/channel) were grey scale balanced (QPcard 101 v2) and printed (300 dpi) at Scale 1:1. In order to calibrate the size of the photographs, a large sheet of paper with a rectangular hole in the size of an A4 sheet (210 × 297 mm) was laid on top of the textile object, framing the intervention while it was photographed. All interventions were photographed from the same distance. Only the area of the textile object that was visible through the rectangular hole in the template was then printed on an A4 sheet of matte Fujicolor professional paper. Before the experiment, some

prints were cut down to a somewhat smaller size to remove edge shadows from the template frame or to separate different interventions where more than one had been included on the same textile object in a photograph.

The 33 conservation interventions in the study represented the three most common textile conservation methods used on costumes, reported in the questionnaire study conducted by Nilsson (2005). They were also the most common conservation methods at Abegg Foundation (Switzerland) according to the conservation reports from the conservation of 23 silk costumes made in 2005-2008, which the first author reviewed in Abegg in 2010. The same was true for all conservation reports (a total of 819) concerning costumes at the Royal Armory in Stockholm (Sweden) reviewed in 2004. Among the 33 interventions included in the present study, the damage was secured with laid couching to an underlying support fabric in 9 cases; in 13 cases the damage was covered with crepeline (i.e., a semitransparent silk fabric) that was attached with running stitches; and in 11 cases the damage was secured with brick couching to an underlying support fabric. Thirty-one interventions were on costumes and two on curtains. Fig. 2 illustrates how (A) laid couching, (B) running stitch, and (C) brick couching are stitched into a fabric. Figs. 3–5 show examples of the three methods applied on textiles, from the present study.

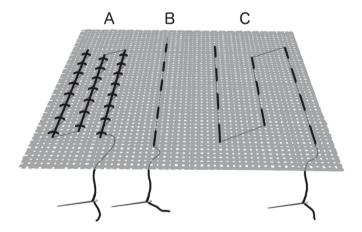


Fig. 2. Illustration of how (A) laid couching, (B) running stitches, and (C) brick couching are stitched into a fabric. Courtesy of Isak Nilsson.

With three exceptions, the interventions were authentic. Three temporary interventions with crepeline were arranged for the purpose of the study, as examples of less aesthetic interventions, to increase the range of aesthetic quality. For example, the crepeline selected had a color that slightly deviated from the textile object, or the crepeline was formed into a

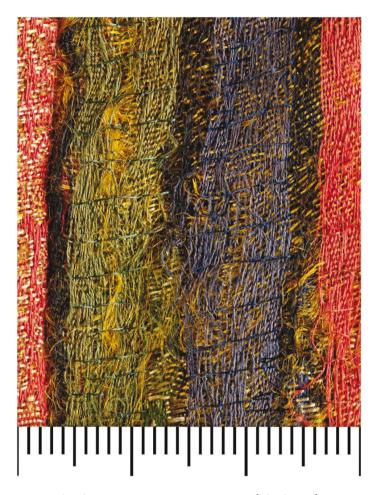


Fig. 3. An example of a conservation intervention with laid couching executed in 2011. Courtesy of Erik Lernestål. The scale of the ruler is in millimeters.

square piece that was not adjusted after the shape of the textile object. The aim was to make these interventions look authentic but as if executed by a less skilled conservator. Creating 'false' interventions on historic costumes might seem unethical from a conservation point of view. However, the crepeline could be lifted off without any harm to the textile. This is also the reason why only crepeline was used for this purpose and not any of the other two conservation methods.

Figs. 6–8 present three examples of the 33 photographs presented to the participants in this study. Please observe that the photographs were presented to the participants in Scale 1:1. However, it is not possible to print the photographs at this size in the journal. Therefore, the photographs in Figs. 6–8 are proportionally scaled down to fit on the page. Fig. 6 illustrates one of the nine cases in which the damage was secured with



Fig. 4. An example of a conservation intervention with silk crepeline attached with running stitches executed in 1996. Courtesy of Erik Lernestål. The scale of the ruler is in millimeters.

laid couching. Fig. 7 illustrates one of the 13 cases in which the damage on the textile was covered with crepeline, attached with running stitches. Fig. 8 illustrates one of the 11 cases in which the damage was secured with brick couching. See also Figs. 3–5 for details of the conservation interventions.

Procedure.—Of the 24 participants, 15 lived outside Stockholm, where the main researcher is based. Because it was not possible to ask all of the participants to come to Stockholm, the researcher traveled to and visited the participants. The participants who lived in Stockholm were tested in their own workshops or at the Royal Armory in Stockholm. Among the 15 participants who did not live in Stockholm, nine were tested in their present or former work environments—if they were retired, two in offices connected to museums that the researcher or the participant had access to, two in semi-public places, like in a conference center at a hotel, and one in her own home. Every participant was tested individually.

First, the researcher presented the 33 photographic prints and asked the participant to sort the interventions into three groups of (1) low, (2) intermediate, and (3) high aesthetic quality. In line with the purpose of the study, no definition of 'aesthetic quality' was provided, and the participant based the sorting on his or her own understanding of the concept. The participant was allowed to change the sorting throughout the session, including the subsequent interview. Second, the researcher interviewed the participant about what criteria he or she had developed and used for sorting the interventions, beginning with the interventions sorted into the



Fig. 5. An example of a conservation intervention with brick couching executed in 2010. Courtesy of Erik Lernestål. The scale of the ruler is in millimeters.

group representing high aesthetic quality. A full session, including sorting of the interventions and the interview, lasted for at most 70 min. During the interview, the researcher took notes. A majority of the interviews were also audio recorded.

Results

The interview data were organized in accordance with the three groups of (1) low, (2) intermediate, and (3) high aesthetic quality. The interventions in Group 1, representing low aesthetic quality, were often described as "unprofessional," "a beginner's work," "too visible," "messy," "wrong material," "ugly stitches," "not safe," and "too few stitches." The interventions in Group 2, representing intermediate aesthetic quality, were described as "little," "fairly," "possibly," and "marginally less good" than the interventions placed in Group 3 representing high aesthetic qual-



Fig. 6. An example of the 33 photographs of conservation interventions used in the present study, as presented to the participants: King Gustav V's morning coat in silk (Inv. no. Lrk 13961). The damage is secured with laid couching. Courtesy of Östen Axelsson.

ity. Sometimes they were described as "good," but never as perfect. The participants also gave advice on how the intermediate interventions could be improved with comments like: "this could have belonged with the best ones if only the stitches are better adjusted to the material," or "a little lighter color on the thread and the stitches will blend in." The participants did also sometimes show sympathy with the conservators who had executed the interventions in the intermediate group by saying, "I know this is difficult but nevertheless the support fabric should have been flat" or "these stitches are too visible, but how could one make them less visible?" Interventions in Group 3 were described as "professional," "invisible," "beautiful," "neat," "discreet," and "fantastic." When the partici-



Fig. 7. An example of the 33 photographs of conservation interventions used in the present study, as presented to the participants: King Gustav III's trousers in silk (Inv. no. Lrk 21447). The damage is covered with crepeline, attached with running stitches. Courtesy of Östen Axelsson.

pants were asked to summarize what characterized the interventions in Group 3, they said: "Not visible," "do not disturb the eye," "correct color on the conservation material," "the costume is sensed as intact," "one should not sense that anything has been done to the object, one should see that it is worn and old," "there must be a system, a regularity," "the interventions are adjusted to the textile and its texture; if so, the intervention does not disturb the eye," and "it is clear what is the intervention and what is the original of the object." In contrast, the impressions of the inter-



Fig. 8. An example of the 33 photographs of conservation interventions used in the present study, as presented to the participants: Waistcoat (provenance unknown) in silk and silver (Inv. no. Lrk 20814). The damage is secured with brick couching. Courtesy of Östen Axelsson.

ventions in Group 1 were summarized with sentences such as: "the intervention catches the eye," "the intervention is what you see instead of the costume," "the conservation material does not adapt to the object," and "the damage is not taken care of properly."

The interview results were summarized into seven sentences that best express the underlying attributes of aesthetic quality found among Groups 1–3, and were to be used as attribute scales in Exp. 2. The sentences were formulated into attribute scales in collaboration with the 24 participants in Exp. 1 through e-mail correspondence. In addition, a pre-test of the potential attribute scales was conducted with a textile conservator

to make sure that the scales were comprehensible and distinct. These resulting attribute scales were:

- 1. From visible and apparent to increasingly invisible interventions (note: disregard the damage, it is the visibility of the intervention that should be scaled).
- 2. From unstructured and messy to increasingly orderly and structured interventions.
- 3. From less to increasingly adapted interventions that visually are ever better integrated with the textile item.
- 4. From worst to best craftsmanship in executed interventions.
- 5. From poor to good execution (i.e., coherent, taut, and tight, most professional impression, intervention executed with a clear methodology).
- 6. From poor to good with regard to how well the conservation material is adapted to the textile item, in terms of color and fineness of the thread and fabric, etc.
- 7. From textile items that look damaged and unrepaired to textile items where the damages are conserved and the object looks complete.

Experiment 2

SCALING AND MODELING

The next objective was to use the seven attribute scales developed in Exp. 1 to identify the most important attributes of aesthetic quality.

Method

Participants.—The participants were 10 randomly selected members of the Swedish Association for Textile Conservation who had not taken part in Exp. 1 (10 women; M age=43.9 yr., range=27–62). On average, they had been active as textile conservators for 15 yr. (range=2–33). All the participants volunteered for the study and were not reimbursed.

Procedure and material.—The main researcher visited four of the 10 participants in their work environments; three were tested at the Royal Armory in Stockholm, two in their own homes, and one in the researcher's home. Thus, every participant was tested individually. On request of the participants, all of them received a PDF by e-mail before the experiment containing digital color photographs of the 33 interventions, in order to study the material in advance.

In the experimental session the participants were asked to rank-order the 33 photographic prints of interventions used in Exp. 1 on each of the seven attribute scales developed in Exp. 1. In order to avoid scalingorder effects (e.g., Gescheider, 1997), each participant used the seven attribute scales in a unique irregular order. A full session lasted for at most 2.5 hours.

Results

Because the seven attribute scales refer to observable properties of the interventions and consequently are physical attributes that textile conservators should be able to agree on, all 10 participants' measurements for each of the seven attribute scales, separately, were subjected to rank-order correlation analyses (Spearman's rho) with a two-tailed test of statistical significance. A correlation coefficient that is not statistically significant was assumed to indicate that a participant used the attribute scale in a different and thus an invalid manner. Consequently, participants whose measurements on an attribute scale did not have statistically significant correlation coefficients with the other participants' measurements on the same attribute scale were removed from the set of data, separately for each of the seven attribute scales. This means that a participant's measurements may have been removed from the set of data for one of the attribute scales, but remained for other attribute scales. Table 1 presents the number of participants whose measurements remained in the set of data for each of the seven attribute scales.

TABLE 1

Number of Participants, Out of 10, Whose

Measurements Remained in the Set of Data For
Each of the 7 Attribute Scales in Exp. 2

Attribute Scale	No. Participants			
1	8			
2	7			
3	8			
4	9			
5	10			
6	6			
7	8			

Rank-order scales of the central tendency of the participants' responses to each of the seven attribute scales were created by calculating the median rank across the participants whose measurements remained in the set of data after the previous step. Where two or more interventions had the same median rank, they were separated with the aid of the arithmetic mean rank. The 33 interventions were thus rank-ordered on seven group attribute scales and assigned the values 1–33.

The seven group attribute scales were subjected to rank-order correlation analysis with a two-tailed test of statistical significance. Table 2 presents the correlation coefficients, and shows that Scales 3, 5, and 6, as well as Scales 2, 4, and 7 were highly associated with each other, as shown by the high correlation coefficients displayed in boldface font. This indicates that the scales represent two underlying latent factors, which were named Coherence and Completeness, respectively.

TABLE 2

RANK-ORDER CORRELATION COEFFICIENTS (SPEARMAN'S RHO) FOR 7 GROUP ATTRIBUTE SCALES (SCALES 1–7) FROM Exp. 2

Attribute Scale	5	3	6	4	7	2
Coherence						
5. From poor to good execution						
3. From less to increasingly adapted interventions better integrated with the textile item	.95†					
6. From poor to good with regard to how well the conservation material is adapted to the textile item	.93†	.90†				
Completeness						
4. From worst to best craftsmanship in executed interventions	.58†	.69†	.46†			
7. From textile items that look damaged and unrepaired to textile items where the damages are conserved and the object looks complete	.46†	.59†	.38*	.89†		
2. From unstructured and messy to increasingly orderly and structured interventions	.51†	.60†	.41*	.84†	.83†	
From visible and apparent to increasingly invisible interventions	.21	.32	.10	.61†	.75†	.79†

Note.—Attribute scales are arranged in order of their relationship with the two latent factors Coherence and Completeness, respectively. Correlation coefficients representing Coherence and Completeness are in boldface. *p<.05. †p<.01 (two-tailed test of statistical significance).

A latent factor is an aggregate of a set of observed variables. In this case, Coherence was an aggregate of Scales 3, 5, and 6, whereas Completeness was an aggregate of Scales 2, 4, and 7. Coherence represented how visually well-integrated an intervention is with the textile item, whereas Completeness represented to what extent the damage of the textile has been conserved. Coherence and Completeness represent continuous variables.

The capacity of Coherence and Completeness to predict the ratings of aesthetic quality of the 33 interventions was investigated. To prepare for the analysis an Aesthetic Quality scale was created based on the data collected in Exp. 1. Because the 24 participants in Exp. 1 had sorted the 33 interventions into three groups of (1) low, (2) intermediate, and (3) high aesthetic quality, it was decided that the Aesthetic Quality scale should represent the central tendency of the participants' responses on an ordinal category scale with three levels.

A contingency table was organized for every intervention, showing how often the 24 participants in Exp. 1 had placed an intervention in Groups 1–3. One χ^2 analysis per intervention was conducted. The interventions were then categorized based on how probable it was that they belonged to one of the three groups (e.g., if 21 participants had placed an intervention in Group 3, representing high aesthetic quality, its probability of belonging to this group was 21/24=87.5%). Interventions for which the χ^2 coefficient was not statistically significant were allocated to the intermediate Group 2.

Ordinal category scales with three levels were created also for Coherence and Completeness. First, rank-order scales of Coherence and Completeness were created by calculating the median rank across all participants whose measurements remained in the set of data and across the relevant attribute scales (i.e., Scales 3, 5, and 6 for Coherence, and Scales 2, 4, and 7 for Completeness). As for the seven group attribute scales, interventions with the same median rank were separated with the aid of the arithmetic mean rank. The 33 interventions were thus rank-ordered on the two factors and assigned the values 1–33. Second, the two rank-order scales of Coherence and Completeness were transformed into ordinal category scales by assigning the value 0 to interventions ranked 1–11 (Low), the value 1 to interventions ranked 12–22 (Intermediate), and the value 2 to interventions ranked 23–33 (High).

An ordinal regression analysis (PLUM in SPSS 19 for Windows) was conducted with Aesthetic Quality (from Exp. 1) as the dependent variable, and the ordinal category scales of Coherence and Completeness (from Exp. 2) as predictors. Table 3 presents the ordinal-regression coefficients (β), the 95% confidence intervals (CI), and the p values of the parameters for this model.

The test of model fit showed that the ordinal regression model was statistically significant (χ_4^2 =31.8, p<.001), the goodness-of-fit test that the model had a good fit with the data (Pearson: χ_{12}^2 =18.7, p=.10; Deviance: χ_{12}^2 =17.3, p=.14), and the test of parallel lines that the underlying assumption of proportional odds was met (χ_4^2 =4.8, p=.30).

Table 3 shows that the ordinal-regression coefficients for Completeness were not statistically significant. Coherence alone explained the rat-

TABLE 3
Ordinal Regression Statistics For Aesthetic Quality of Textile Conservation Interventions in Exp. 2 (N=33)

Predictor	β	95%CI	р
Coherence			
Low	-5.94	-9.17, -2.70	<.001
Intermediate	-2.56	-4.80, -0.31	.03
High	0.00	Reference	
Completeness			
Low	-1.25	-3.46, 0.96	.27
Intermediate	0.84	-1.28, 2.96	.44
High	0.00	Reference	

Note.—β: ordinal-regression coefficient; *CI*: confidence interval.

ings of Aesthetic Quality of the 33 interventions among the 24 participants in Exp. 1.

GENERAL DISCUSSION

Two experiments were conducted to investigate what textile conservators mean when they say that a conservation intervention is aesthetically appealing, and what properties of these interventions affect ratings of visual aesthetic quality among textile conservators. This study identified two factors, named Coherence and Completeness. Coherence represents how visually well-integrated an intervention is with the textile item, whereas Completeness represents to what extent the textile has been conserved. For an intervention to be regarded as complete, it must take care of the entire damage. Ordinal regression analysis showed that Coherence was the most important predictor of ratings of Aesthetic Quality of textile conservation interventions, based on the responses of 34 textile conservators.

This study's review of the discussion on aesthetics in the literature on textile conservation suggests that according to textile conservators an aesthetically successful intervention is visually well-integrated with the original textile item (Flury-Lemberg, 1988; Landi, 1992; Kaldany, *et al.*, 1999; Gill, 2006; Conti, 2011). In the present study, an intervention was regarded as aesthetically successful when the intervention was performed with a clear methodology, in a material that was well adapted to the textile item in terms of color and fineness of the thread and fabric. Thus, the present results are in agreement with and provide empirical support to the discussion in the literature.

With regard to the ability to generalize the results of the present study, it is central that the 24 participants in Exp. 1 were a random sample of the

58 members of the Swedish Association for Textile Conservation. Thus, on average, the results are applicable to Swedish textile conservators. The agreement with the literature indicates that the present results may be extended beyond the Swedish borders.

One limitation in the present set of data is that it does not allow an analysis of individual differences. (For discussions on individual differences with regards to aesthetic appreciation, see Axelsson, 2007a, 2011.) Also, one limitation of the present study is that the range of the stimulus material was restricted, although it was extended for the purpose of the study in terms of professional execution. Another limitation is that the stimulus material was presented in the form of color-calibrated photographs at Scale 1:1 and printed in A4 size. In practice, this was the largest possible print size. As shown in Figs. 6–8, this restricted how much of the original textile objects could be presented to the participants. Besides the intervention, a photograph depicted only a part of an original textile object and never an entire object. It could be argued that the results would have been different if the participants had been able to study the full textile objects in reality, seeing the interventions in their context. However, a conservator normally works very close to the textile object and is used to viewing it both at closeup and from a distance. It may also be argued that the overall impression of the finished result depends on the quality of the intervention at the level of detail. In agreement with the latter, the participants were very meticulous in their approach and wanted to examine the interventions close-up, which the photographs allowed. In addition, several of the participants spontaneously expressed satisfaction with the quality of the photographs. Importantly, the participants' task was to judge the interventions and not the end results of the textile items as a whole.

It may also be argued that the method of measurement of aesthetic quality is a limitation in that the participants in Exp. 1 were allowed to use their own definitions of this multidimensional concept. Alternatively, a predefined set of attribute scales, like 'pleasant,' 'attractive,' and 'interesting' could have been used to capture different aspects of 'aesthetic quality' (cf. Cox & Cox, 2002; Carbon & Leder, 2005; Faerber, Leder, Gerger, & Carbon, 2010; Carbon, 2011; Gattol, Saaksjarvi, & Carbon, 2011). However, in the present study the authors gave priority to simplicity over complexity in the assessments.

After refining the measurement method developed in the present study (i.e., scales and instructions), it would be interesting to follow it up. This could include investigating individual differences in aesthetic appreciation among textile conservators, as well as using a full factorial design of Coherence and Completeness to investigate the relative importance of these two factors in more detail, although this would require a different kind of stimulus material than the primarily authentic material used in the present study.

Concluding Remarks

For textile conservators the aesthetic quality of a conservation method is just as important as the technical aspects. This study has shown that with 'aesthetic quality' textile conservators chiefly mean that an intervention must be coherent, that is, visually well-integrated with the textile item. It represents a first step toward understanding the practical implications of aesthetic quality in textile conservation, and further studies are needed. They may include investigating what conservation methods are the most successful from an aesthetic point of view or other issues that the present study has given rise to.

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Accepted May 20, 2015.