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Viscose Rayon: An Absorbing Problem
An Investigation into the Impact Conservation Wet Cleaning Treatments have on Historic Woven Viscose Rayon Fabrics; with a Supplementary Analysis of Current Techniques for Identifying Man-Made Fibres

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Abstract

Poor wet properties are attributed to viscose. Research has shown how the fibre's manufacture influences this characteristic and how progressive improvements to processing methods mean that viscose rayons pre-1940 have even poorer wet properties than later versions. It was decided to investigate the implications this has for conservation wet cleaning treatments. Tensile strength testing was carried out on specimens of viscose rayon from three eras – c.1940s, c.1960s and c.1980/90s – which had been subjected to a wet cleaning treatment, to contextualise research for conservation. Both Orvus WA® and Dehypon LS45 were used for wet cleaning. Results showed the two later viscose rayons lost significant strength in the wet state, up to 50%. The c.1940s fabric had a greater strength reduction of around 80% - however, degradation from a black colourant present was shown to have affected results. This highlighted the risk that degradation, even if not visible macroscopically, can be significantly exacerbated in viscose rayon when wetted. It was concluded that wet cleaning viscose rayon can be a suitable with either the aforementioned detergents, although it may be less appropriate for older more degraded versions. Problems with the fibre identification of viscose rayon were also explored, showing successes in using a combination of different *basic* analytical techniques.

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Introduction

Viscose rayon is a semi-synthetic regenerated cellulosic fibre. In production in the Western World the longest of any man-made fibre, its inception was just over a century ago.[1] During this time it has been known by different generic and trade names.

The lifespan of viscose rayon makes it a relative infant in the history of textile fibres, compared with natural fibres like cotton and wool which have been in use for millennia.[2] Its shorter existence is reflected in the minimal amount of literature available on its conservation. It is likely that viscose rayon textiles will increasingly appear at the textile conservator's workbench and therefore more research is needed, to ensure well informed treatment decisions.

This research will investigate what impact conservation wet cleaning has for woven viscose rayon textiles, focusing on the low wet strength attributed to viscose rayon. These properties have been well documented and researched within the context of the textile industry and this project now seeks to use this body of knowledge to assess the implications these have within a conservation context.

Progressive changes to manufacturing methods over the years since the fibre's inception have lead to serial improvements in the wet properties of viscose rayon. This means that early examples of viscose rayon fibres have worse wet properties than later versions. The most significant improvements to wet strength are documented as occurring in the 1950s and 1970s.[3] It was decided to contextualise this research by assessing the extent of any loss in strength for wet cleaning past examples of viscose rayon; with the aim of showing whether different treatment choices, based on year of manufacture, may be appropriate.

To assess this, a series of controlled scientific tests has been conducted on specimens of viscose rayon from three eras, c.1940s, c.1960s and c.1980/90s, to investigate strength using a tensile strength tester. These tests compare differences between dry and wet specimens which have been subjected to a controlled wet cleaning treatment. In addition,

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[1] Susannah Handley, 'Chapter 1, 1700s-1930. The Chemist Conquers the Worm', in Nylon: The Manmade Fashion Revolution, (London: Bloomsbury, 1999), p. 21.

[2] Jennifer Harris, 'The Ancient World: Introduction', in *5000 Years of Textiles*, (London: The British Museum Press, 2010), pp.54-55.

[3] Kathryn L. Hatch, *Textile Science*, (St. Paul, USA: West Publishing Company, 1993), p. 186.

specimens were also tested which had been subjected to a controlled wet cleaning treatment and then allowed to air dry, to show whether any changes in wet properties were permanent.

Test specimens were obtained from charity shops and through an online auction website.

During researching, it was determined that there exist certain complexities in the field of man-made fibre identification, including the identification of viscose rayon, with the appearance of singular fibre types having variance, as well as, being near-identical to some other man-made fibres, under a microscope. It was therefore decided to carry out research into viscose rayon fibre identification as part of this project and as a precursor to strength tests.

This research aimed to explore the appropriateness of wet cleaning as a treatment option for conservators working on viscose rayon.

The aims and objectives for research now follow.

Research Aims and Objectives

1. Research current literature available on the conservation of viscose rayon in relation to wet cleaning treatments by looking at secondary conservation sources.

2. Investigate the implications viscose rayon's wet properties have for the context of conservation wet cleaning treatments, looking at wet strength and dimensional change when wet by conducting tensile strength and dimensional change tests on wet and dry examples of viscose rayon. To contextualise results wet specimens to have undergone a wet cleaning treatment prior to testing.

3. Determine the permanence of any change in strength and dimensions, as a result of wet cleaning by including tests on viscose rayon specimens which have been wet cleaned and then allowed to air dry before testing.

4. Investigate changes in viscose rayon manufacture over the years which have improved its wet properties and ascertain whether earlier examples of woven viscose rayon may be significantly weaker, or behave significantly differently, to later examples by researching industry and science secondary sources. Examples of viscose rayon from different times which cover significant improvements in fibre processing, as recorded by industry, to be selected for testing to highlight any specific weaknesses in viscose rayon produced in different eras.

5. Investigate whether detergent choice impacts on the wet properties of viscose rayon fibres by wet cleaning specimens before testing with both an anionic and a non-ionic detergent.

6. Investigate how the chemical properties of viscose rayon cause it to have poor wet properties by researching textile science secondary sources.

7. Carry out fibre identification on textiles purportedly of viscose rayon content to address issues concerning the accurate identification of these fibres and, as a precursor to testing: analysis to be carried out using microscopy, cross-sections, solubility tests and ATR-FTIR spectroscopy[4].

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[4] See Appendix 2 for an explanation of what ATR-FTIR spectroscopy is.

Chapter 1 – Literature Review

Introduction

The focus of this review is to present the literature which served as inspiration and also provided the rationale for this research project.

Upon examining current conservation literature on viscose rayon, it became apparent that relatively little was published on this subject, particularly in relation to treatment choices – indeed this appears to apply to the subject of man-made fibres as a whole. Research into textile science literature provided knowledge on the chemical and mechanical properties of viscose rayon. Of particular interest are descriptions defining viscose rayon as having a comparatively lower wet strength than most textile fibres. This suggested it would be useful to investigate the implications of viscose rayon's low wet strength for wet cleaning treatments.

This chapter will first briefly survey information available in textile science literature pertaining to viscose rayon's low wet strength. It will then investigate available literature on its conservation. Publications identifying the need for more conservation research into viscose rayon will then be presented. Lastly, inferences will be drawn as to the reasons for this lack of research.

1.1 Wet Properties of Viscose Rayon

When regular viscose rayon fibres are wetted, they have been described as losing a significant level of strength and as having a higher elongation, compared to in their dry state. The textile science lecturer Kathryn Hatch explains how: '[Viscose] Rayon loses about 50% of its tenacity when saturated with water. Elongation increases about 20%; it is almost twice as easy to elongate the saturated fiber.'[5]

E. Gohl and L. Vilensky similarly discuss the comparatively low tenacity of viscose rayon between its dry and wet states saying: 'When wet, viscose is only half as strong as when dry.'[6]

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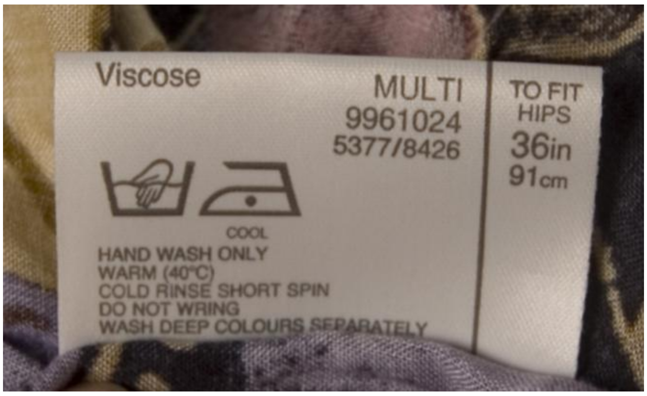
[5] Hatch, (1993), p. 184.

[6] E. P. G. Gohl and L. D. Vilensky, *Textile Science: An Explanation of Fibre Properties*, (Melbourne, Australia: Langman Cheshire Pty Ltd., 1981), p. 31.

It can be gathered that, as viscose rayon loses strength when wet, care needs to be taken when wet cleaning such fabrics. Indeed, this is demonstrated by looking at domestic care labelling of textile goods such as that in Fig. 1 from a pair of c.1980/90s trousers.

Fig. 1 Domestic wash care label from a pair of viscose rayon B*h*S trousers, c.1980/90s



Viscose
MULTI
9961024
5377/8426
TO FIT
HIPS
36in
91cm
COOL
HAND WASH ONLY
WARM (40°C)
COLD RINSE SHORT SPIN
DO NOT WRING
WASH DEEP COLOURS SEPARATELY

In 1976 S. Zeronian, wrote a synopsis of man-made fibres, in which the different forms of viscose rayon were outlined amongst brief discussion of manufacture finishing and methods of fibre identification, followed by reference to its degradation. In terms of treatment however, no reference was made to wet cleaning and its suitability for use on viscose rayon nor are any specific case studies mentioned. There is only a brief assertion that: ‘Rayons have good resistance to dry-cleaning solvents, and both oxidizing and reducing bleaches may be used with suitable precaution.’[7]

J. K. Hutchins, in 1984, described the different chemicals used in the manufacture of viscose rayon prior to 1940 and how these may influence deterioration. Hutchins aim was to provide conservators with information so that they could: ‘[…] consider and treat the sources rather than the symptoms of decay.’[8] Like Zeronian above however, this article did not provide any treatment case studies.

The first publication found offering specific treatment advice for viscose rayon objects is the influential conservation textbook *Chemical Principles of Textile Conservation (CPOTC)*, first

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[7] S. H. Zeronian, ‘Conservation of Textiles Manufactured from Man-Made Fibres’, in Preservation of Paper and Textiles of Historic and Artistic Value, 172nd Meeting of the American Chemical Society, (1977), p. 215.

[8] J. K. Hutchins, ‘Regenerated Cellulosics: Their Fabrication and Finishing Before 1940’, in ICOM Committee for Conservation, 7th Triennial Meeting, (1984), p. 84.9.21.

published in 1998. In here is described how: ‘Wet viscose textiles require support because they readily stretch and lose their shape.’[9] This information corresponds to the information taken from Hatch and, Gohl and Vilensky above. There is however, no mention in *CPOTC* of the different types of regenerated cellulosic fibre, such as modal, which are similar to viscose rayon but which have a higher wet strength.[10] Nor, is there mention of improvements to the manufacture of viscose rayon, which may mean that earlier examples of viscose rayon are weaker than later examples.[11] These two points will be explored further in Chapter 2. These added considerations could mean that not all regenerated cellulosic fibres need to be treated the same, with the implication that wet cleaning treatment methods may alter dependent on the factors outlined above.

1.2 Conservation Literature Relating to Viscose Rayon

An overview of the available conservation literature is now discussed, looking at available case studies of wet cleaning treatments followed by issues of detergent choice in wet cleaning. Finally, the problems with identifying viscose rayon fibres will be discussed; fibre identification having pertinence as a precursor to wet cleaning treatment.

1.2.a Conservation Treatments

Only a handful of available case studies make reference to treatments carried out on viscose rayon and, these mostly provide only minimal information with modest insight into the reasons behind treatment choices. For instance, the textile conservator Elizabeth-Anne Haldane in 2007 published an article relating to the Victoria & Albert’s (V&A) exhibition *Surreal Things*, in which the presence of semi-synthetic fibres was discussed. Haldane begins by describing how regenerated cellulosic fibres have: ‘[…] relatively poor wet strength.’[12] However, as the article focuses on man-made fibre identification, only a passing mention is made stating that treatments were carried out on these semi-synthetic textiles, which included examples of viscose rayon, with no specific details.

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[9] Ágnes Tímár-Balázsy and Dinah Eastop, ‘Part 1 Materials: 1.4 Man-Made Fibres’, in *Chemical Principles of Textile Conservation*, (Oxford: Butterworth-Heinemann, 1998), pp. 57.

[10] Calvin Woodings (ed.), *Regenerated Cellulosic Fibres*, (Cambridge: Woodhead Publishing in association with The Textile Institute, 2001), p. 248.

[11] J. W. S. Hearle, ‘Progress in Rayon’, in *The New Scientist,* 3rd September 1959, Vol. 6, No. 146, (London, 1959), p. 344.

[12] Elizabeth-Anne Haldane, ‘Surreal Semi-Synthetics’, *V&A Conservation Journal,* Number 55, Spring, (2007), p. 16.

Only two case study references were found in the literature surveyed discussing the wet cleaning of a textile containing viscose rayon and neither of these were examples of a textile containing solely viscose rayon. The first example involved the treatment of a coat containing viscose rayon blended with other fibres; the second, a Torah mantle containing viscose rayon in its fringing. (Both illustrate how viscose rayon may be present as a composite amongst other materials in an object.) These case studies are discussed in detail below.

The wet cleaning treatment of the coat was carried out as a student project by the textile conservator Clare Stoughton-Harris.[13] The coat was constructed in the mid-1960s and analysis showed that its outer material was composed of a blend of viscose rayon, wool, cotton and acrylic.[14] However, the focus of this treatment was on issues relating to the rubberised coating present on the coat, and only a brief mention is made of the viscose rayon content. The treatment was further published in *CPOTC*, where the fragility of the viscose rayon fabric was mentioned: 'Viscose rayon […] fibres are weaker when wet. The coat was therefore supported on nylon mesh screens during the wet cleaning process, to reduce the risk of mechanical damage that might arise by the increased weight of the coat in the washing and rinsing solutions.'[15] This description appears to refer to the high absorbency of viscose rayon fibres, suggesting that their saturated weight could make the fibres more susceptible to mechanical damage during wet cleaning.

The wet cleaning of the 1920s Torah mantle was described in an unpublished report, undertaken as part of a student project by textile conservator Karen Thompson. Reference is again made to the weakened state of viscose rayon fibres when wet, and, like Stoughton-Harris' treatment above, the object was supported on a net screen during wet cleaning: 'A large mesh screen was constructed […] to support the object during the wet cleaning process. Due to the fragile nature of […] viscose rayon when wet it was important to keep the handling to a minimum.'[16] Thompson describes how following wet cleaning, the fringing

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[13] Clare Stoughton-Harris, *Treatment of 20th Century Rubberised Multimedia Costume: The Conservation of a c. 1967 Raincoat Designed by Mary Quant,* Final Diploma Report, Textile Conservation Centre, Courtauld Institute of Art, University of London, 1991.

[14] Stoughton-Harris, (1991).

[15] Ágnes Tímár-Balázsy and Dinah Eastop, 'Part 6 Case Histories: 6.1 The Treatment of a Raincoat Designed by Mary Quant, c. 1967', in *Chemical Principles of Textile Conservation*, (Oxford: Butterworth-Heinemann, 1998), p. 142.

[16] Karen Thompson, *The Conservation of a Torah Mantle*, TCC 0858.6, unpublished student conservation report, Textile Conservation Centre, 1992, p. 17.

had suffered some damage which she describes as possibly, '[…] due to handling when the object was padded out during drying as the opening where the fringing was the only point of access.'[17] This demonstrates the need for caution when handling wet viscose rayon textiles.

1.2.b Detergent Choice

Both the above treatment examples involving the wet cleaning of viscose rayon textiles cited the use of the non-ionic detergent Synperonic N. However, neither specifies why the choice of a non-ionic detergent was made.

It is speculated that decisions to use a non-ionic detergent were possibly influenced by the fact that Synperonic N was commonly used as a generic detergent choice for all fibres by some conservators at the time of treatment. John Fields, Andrew Wingham, Frances Hartog and Vincent Daniels describe how Synperonic N in Europe was: '[…] the surfactant of choice, as it was the only surfactant recommended by a scientist (Plenderleith 1956) and has proved highly efficient on cotton and wool.'[18] Harold Plenderleith played a major role in developing the field of conservation science,[19] publishing one of the first books on conservation in 1956.[20]

The conservator Ágnes Tímár-Balázsy provides more information on how despite research into different detergent options for use in conservation in the 1980/90s: '[…] many workshops started to use predominantly one type of surfactant routinely (see the popularity of […] Synperonic N in the UK), despite there being many varied surfactants available and recommended in the conservation literature.'[21]

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[17] Thompson, (1992), p. 18.

[18] John A. Fields, Andrew Wingham, Frances Hartog, Vincent Daniels, 'Finding Substitute Surfactants for Synperonic N', in Journal of the American Institute of Conservation, Vol. 43, No. 1, (USA: The American Institute for Conservation of Historic & Artistic Works, 2004), p. 56.

[19] British Museum, 'History of the Department', Conservation and Science, (http://www.britishmuseum.org/about_us/departments/conservation_and_science/history.aspx), accessed 7th August 2012.

[20] H. J. Plenderleith, The Conservation of Antiquities and Works of Art: Treatment, Repair and Restoration, (London: Oxford University Press, 1971).

[21] Ágnes Tímár-Balázsy, 'Wet Cleaning of Historical Textiles: Surfactants and Other Wash Bath Additivies', in IIC Reviews in Conservation Vol. 1, (2000), p. 59.

This practice of using Synperonic N in conservation however underwent upheaval in 2000 with a ban in Europe following concerns that its biodegradation products were toxic to fish.[22] This event prompted fresh research by conservators into suitable alternative detergents for use in the conservation of textiles. Fields et al. tested both non-ionic and anionic detergents on artificially soiled samples of different natural fibres and showed the suitability of two anionic detergents and three non-ionic detergents for conservation.[23]

Currently, conservators consider that non-ionic detergents are most effective at soil removal from proteinaceous fibres and anionic detergents are most effective at soil removal from cellulosic fibres.[24] As viscose rayon is a cellulosic fibre, it seems logical that an anionic rather than a non-ionic detergent would be the most effective at soil removal from this fibre type. The research described above however focused on natural fibres and no discussion was found on suitable detergents for use with man-made fibres in the literature surveyed.

1.2.c Issues Relating to Fibre Identification

Problems in the identification of viscose rayon fibres have been discussed by textile specialists both in industry and conservation. The 1953 edition of the annual textile industry publication *Cotton Year Book* shows that the use of *basic* identification methods has gone largely unchanged for the past 60 years, alongside concerns over their accuracy: 'With the introduction of new yarns and fibres every year, their accurate identification becomes increasingly difficult and more and more a job for the specialist.'[25] This volume provides methods of identification that include: '*Visual Examination*', '*Burning Tests*' (akin to hot-stage microscopy), '*Stain Tests*', '*Solubility Tests'* and '*Microscopic Appearance*'.[26]

The industry tests mentioned above are still the primary methods used for fibre identification today. Although, some test methods are less suitable for use in conservation, such as stain tests which require un-dyed fibre samples. With these tests, the same concerns over

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[22] Rebecca Tinkham and Nancy Kerr, 'Effectiveness of Soil Removal by Two New Nonionic Surfactants, Orvus WA Paste and Surfactant Blends', in AIC Textile Specialty Group Postprints, (2001), p. 48.

[23] Fields et al., (2004), pp. 69-70.

[24] Centre for Textile Conservation, *Wet Cleaning: Aims, Issues and Techniques,* unpublished course hand-out, University of Glasgow, 2011, p. 5.

[25] Textile Mercury Annuals, 'Rayon', in *Cotton Year Book (Incorporating the Rayon and Synthetic Fibres Annual) 1953*, (Manchester: Textile Mercury Ltd., 1953), p. 700.

[26] Textile Mercury Annuals, (1953), pp. 700-704.

accuracy in identifying man-made fibres remain today – although these techniques are fairly accurate in identifying natural fibres. This issue is all the more challenging for textile conservators who are not predominantly dealing with the new fibres of today but, who are more likely to be challenged over identifying historical man-made fibres. This requires insight into previous production techniques which may have influenced microscopic and cross-sectional appearance.[27] The accuracy of the use of such test methods for the identification of viscose rayon fibres has been analysed by the conservation professionals Clare Lewarne, Irene Karsten and Beverly Lemire. They describe the use of a combination of bright-field microscopy, acetone solubility tests and hot-stage microscopy to differentiate between viscose rayon and other man-made fibres; concluding that such techniques can be effective deciphering tools.[28] The use of a combination of techniques to identify manmade fibres has similarly been expressed in The Textile Institute's book *Identification of Textile Materials:*

> 'The introduction of so many modified fibres has made the task of identifying man-made fibres more difficult in recent years. While microscopical examination […] is a useful method it is still advisable to use some of the wide range of chemical and physical tests to supplement the optical ones in order to obtain a reliable diagnosis.'[29]

Whilst the use of a combination of the above techniques can provide information relating to the identification of man-made fibres, it is still not entirely exhaustive. Today, more sophisticated techniques using FTIR and NIR[30] analysis are non-invasive with instantaneous and more accurate results based on the chemical composition of materials are increasingly being used in conservation to identify man-made materials in museum collections.[31] In Haldane's article on identifying man-made fibres at the V&A, mentioned above, she outlines

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[27] The Textile Institute, *Identification of Textile Materials*, Seventh Edition, (Manchester: The Textile Institute, 1985), p. 136.

[28] Clare Lewarne, Irene Karsten and Beverly Lemire, 'Fibre Identification and Condition Charaterization of 1920s and 1930s Rayon Dresses', in *Conservation of Three Dimensional Textiles 7th North American Textile Conservation Conference,* Québec City, Canada, September 30 – 3 October 2009, (Omnipress, 2009), 301-308.

[29] The Textile Institute, (1985), p. 22.

[30] FTIR: Fourier Transform Infrared, NIR: Near Infrared.

[31] Emma Richardson, Graham Martin and Paul Wyeth, 'Collecting a Near Infrared Spectral Database of Modern Textiles for Use of On-Site Characterisation', in *Textiles and Text, Re-establishing the Links between Archival and Object-Based Research, AHRC Third Annual Conference*, (2007), pp. 257-263.

how it may not be a simple task to determine whether a fibre is man-made and, that museum records of fibre types may be inaccurate or missing: 'Existing records for these costumes generally stated that they were either made from silk or did not specify a fibre type.'[32] In the article, she describes the use of FTIR microscopy and NIR spectroscopy on a group of costumes containing man-made fibres including viscose rayon, to provide precise identification of such fibres: 'The aim of testing was to provide accurate information for the curators and also to inform conservation treatment.'[33] Haldane does not however discuss the potential issues with identifying manmade fibres using these techniques, especially of blended fibres. In 2006 however, Emma Richardson when working towards her PhD at the V&A, published an article in which she briefly mentions complications which can be present when using NIR spectroscopy for analysing textiles: '[…] textiles are often complex in nature, so where possible I intend to take into account factors such as polymer blends, surface coatings, additives and age.'[34]

Whilst the use of FTIR and NIR equipment may provide the most accuracy, not all conservators have access to such expensive specialist equipment. This therefore shows the importance of continued research such as that by Lewarne et al. for refining the more accessible *basic* techniques such as microscopy and solubility tests.

Influenced by the literature reviewed above, the use of *basic* methods of fibre identification will be explored initially to identify whether test objects acquired for this project contain viscose rayon, before carrying out any scientific testing. The accuracy of this information will be verified by further use of specialist ATR-FTIR spectroscopy[35] (similar to the FTIR microscopy and NIR spectroscopy). It is hoped in this way that the usefulness of the more *basic* fibre identification methods can be assessed, in order to provide reassurance about their accuracy for conservators without access to specialist equipment.

1.3 Identifying the Gap in Research

In 1975, S. Zeronian wrote that: 'Up to the present time conservators were interested in preserving textiles made from natural fibers. With time, however, as present day materials

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[32] Haldane, (2007), p. 16.

[33] Haldane, (2007), p. 16.

[34] Emma Richardson, 'Towards On-Site Collections Management: NIR Characterisation and Condition Monitoring of Textiles', in *V&A Conservation Journal,* No. 54, Autumn, (2006), p.12.

[35] See Appendix 2 for an explanation of ATR-FTIR spectroscopy.

incorporating man-made fibers are collected, concerns will arise as to their preservation and display.'[36] In 1980, Zeronian further stated that continual developments in the man-made fibre industry make it important for conservators to: '[…] continually update their knowledge of the properties of man-made fibers.'[37]

Despite such statements, in 1999 Lisa Ferreira identified a perceived requirement for more research and publications relating to the conservation of man-made fibres and in particular into viscose rayon, from a survey she conducted and sent to textile conservators working in America: '[…] rayon was identified as the manufactured fiber most in need of conservation attention, and it seems an obvious focus for future research.'[38]

A decade later in 2009 Lewarne et al. describe how there is still a lack of available research into the conservation of early viscose rayon: '[…] decisions about treatment can be difficult with little available research on early rayon and its conservation.'[39]

1.4 Possible Reasons for the Gap in Research

A few possible reasons for the lack of published work on the conservation of viscose rayon have now been identified. (1) The identification of man-made fibres, including viscose rayon, can be difficult and perhaps conservators are not certain what fibre type they are treating. (2) A lot of literature published regarding the conservation of man-made fibres in general has focused on issues relating to problematic degradation of volatile materials such as synthetic rubber and cellulose nitrate.[40] (3) Viscose rayon is a relatively recent addition to the fibre world and, as such, perhaps forms a lower proportion of fibre types brought to textile conservators for treatment.

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[36] Zeronian, (1977), p. 208.

[37] S. Zeronian, 'Preservation Problems Related to Synthetic Fibers', in *Conservazione E Restauro Dei Tessili, Convegno Internazionale*, Como, Italy, 13-18 October 1980, p. 72.

[38] Lisa M. Ferreira, 'Manufactured-Fiber Conservation: A Cause for Concern?', in *AIC Textile Specialty Group Twenty-Sixth Annual Meeting,* (1999), pp. 16.

[39] Lewarne, Karsten and Lemire, (2009), p. 304.

[40] Chris Paulocik and R. Scott Williams, 'Modern Materials in Costume Collections: A Collaboration between Scientist and Conservator', in *North American Textile Conservation Conference*, (2002), pp. 77-89.

Conclusion

The literature surveyed has demonstrated the influence and rationale for this research project, showing that research into the wet cleaning treatment of viscose rayon textiles would have twofold benefits: firstly, the fibre has been described as having a low wet strength and secondly, there is a lack of published research into, and case studies of, wet cleaning viscose rayon.

Additionally, fibre identification has been recognised as somewhat problematic. If, as the literature suggests, viscose rayon's low wet strength has implications for wet cleaning, then fibre identification will be important as a precursor to treatment, to ensure appropriate conservation decisions are made and any potential concerns can be anticipated. Fibre identification will therefore form an aspect of this research.

Finally, there appears to be a lack of clarity over which detergents are most suitable for use with man-made fibres. Whilst it is not possible to research this fully within the confines of this project, this has been taken into account in devising wet cleaning tests.

Chapter 2 – History and Manufacture of Viscose Rayon

Introduction

This chapter will start with the history which led to the invention of viscose rayon. It will briefly consider the similarities between viscose rayon and other regenerated cellulosic fibres in order to contextualise and set-apart viscose rayon from these. The processing of viscose rayon will be described to show its impact on fibre properties: poor wet strength and the tendency of fibres to swell, grow or shrink when wet. To record how the properties of viscose rayon have gradually been improved, changes and modifications to processing will be examined. Lastly, the development of the nomenclature of viscose rayon will be reviewed to identify former meanings of terminology still used today.

2.1 A Short Background to Regenerated Cellulosics and Viscose Rayon

The chemist Robert Hooke has been recognised as the first person to conceive the notion of creating artificial fibre in his 1664 publication *Micrographia.*

> '[…] there might be a way of making an artificial glutinous composition, much resembling, if not full as good, nay better, than that excrement, or whatever other substance it be out of which the silkworm wire-drams his clew.' [41]

However, it was not until the mid-nineteenth century that artificial fibres for use in textiles were invented.

The first man-made fibres were developed to replicate the desirable properties of natural silk, without its high cost. They were named at the time *artificial silks*.[42] These early man-made fibres were created from the naturally occurring polymer cellulose – from wood pulp or cotton linters – dissolved and treated in chemicals, to form a fibre suited for use in textiles. Later, such fibres became known as semi-synthetics, distinct from true synthetic fibres derived entirely from chemicals: such as the first synthetic fibre, nylon, introduced in 1936.[43] Viscose rayon belongs to the group of semi-synthetics known as regenerated cellulosic fibres, so named because their production involves the breaking-down and then

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[41] Robert Hooke, 'Observ. IV. Of Fine Silk, or Taffety', in Micrographia or Some Physiological Descriptions of Minute Bodies Made by Magnifying Glasses with Observations and Inquiries Thereupon, (London: Constable and Company, 1961), p. 7.

[42] Handley, (1999), pp. 16-17.

[43] Hatch, (1993), p. 201.

regenerating of the cellulose molecular structure in filament form. Five processes have been invented to produce regenerated cellulosic fibres. Chronologically, these five processes, as currently named, are; cellulose nitrate (no longer in production), cupro, viscose rayon, modal and lyocell.[44] Of these, viscose rayon has achieved the biggest success in terms of scale and length of production.[45]

Regenerated fibres are composed of cellulose II, different in structure to the cellulose I in natural cellulose fibres such as cotton.[46] During viscose rayon production, R. Moncrieff explains that:

> '[…] the final filament differs chemically from the original cellulose of the wood in only one respect – that it has suffered some degradation during the manufacturing processes: the very long cellulose molecules have been partly hydrolysed and have been broken down into shorter, although still very long, molecules.'[47]

This degradation changes the fibre's properties, giving it low wet strength and a tendency to swell and change in dimensions when wet. These issues are not such a problem for modal and lyocell fibres, manufactured to have improved wet strength.[48] The specific processing stages which affect viscose rayon's properties will be described later in this chapter and the fibre's chemical properties relating to its wet properties are explored in Chapter 3.

Due to the manufacturing processes of regenerated cellulosic fibres, they can all look similar and it can be challenging to distinguish between them during fibre identification analysis. To further complicate matters, they can also look similar to other man-made fibre types, such as acrylic.[49] Problems with the identification of viscose rayon fibres are analysed in Chapter 4.

Outlined below is a short history of the conception of viscose rayon.

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[44] British Standard, (2010), p. 2.

[45] British Man-Made Fibres Federation, Better Living with Man-Made Fibres, (London: British Man-Made Fibres Federation, 1986), p. 12-13.

[46] See, John W. S. Hearle, 'Chapter 8 – Physical Structure and Fibre Properties' , in Calvin Woodings (ed.), Regenerated Cellulosic Fibres, (Cambridge: Woodhead Publishing, 2001), pp. 203-204.

[47] R. W. Moncrieff, 'Chapter 9 - Viscose Rayon', in *Man-Made Fibres*, (London: Butterworth & Co Ltd, 1975), p. 164.

[48] Gohl and Vilensky, (1981), p. 58.

[49] The Textile Institute, (1985), pp. 95-110.

2.1.a The Beginning of Viscose Rayon

Three British chemists, Charles Cross, Clayton Beadle and Edward Bevan, working in laboratories at Kew Royal Botanic Gardens invented the viscose process of regenerating cellulose, patented in 1892.[50-51] Their efforts were not immediately directed at the textile industry. The creation of viscose rayon fibres came instead following, fellow Kew worker, Charles Topham's invention of a centrifugal spinning method for extruding filaments. Topham's supervisor, Stearn, realised the potential of Topham's spinning method for the viscose process to make textile fibres patenting it in 1898.[52] Manufacturers worldwide took up both Cross et al.'s and Topham's patents. Initially, the process proved inconsistent and unreliable. Commercial success for viscose rayon only came after Samuel Courtauld and Co. Ltd – later Courtaulds Ltd – bought the rights to the viscose process in 1904 and worked alongside Kew laboratories to develop a large-scale production process.[53] In 1905 they opened a factory in Coventry and began production of viscose rayon (see Fig. 2).[54]

The viscose process is described below.

Fig. 2 Worker at Samuel Courtauld & Co. Ltd. Coventry Factory, 1908
From, Susannah Handley, *Nylon: The Manmade Fashion Revolution*, (London: Bloomsbury, 1999), p. 22.

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[50] Woodings, (2001a), p. 5.

[51] Textile Mercury Annuals, (1953), p. 666.

[52] Textile Mercury Annuals, (1953), p. 666.

[53] Woodings, (2001a), p. 8.

[54] Handley, (1999), p. 21.

2.2 The Viscose Process

The viscose process consists of four main stages. The following description has been sourced from Gordon Cook's *Handbook of Textile Fibres Vol. II Man-Made Fibres.*[55]

2.2.a Stage One: Formation of Soda Celullose

The wood pulp or cotton linters are formed into thick blotting sheets, treated in a caustic soda solution for an hour and then shredded, forming powdery crumbs which are aged for up to one day. During this time the caustic soda reacts with the cellulose, forming soda cellulose (see Fig. 3). In this process, oxygen from the air breaks down (degrades) the long cellulose molecules to some degree into shorter molecules. This result contributes to viscose rayon's poor wet properties.

2.2.b Stage Two: Production of Sodium Cellulose Xanthate

Following ageing, the crumbs are placed in a revolving drum and carbon disulphide added; the two gradually combine to form the mixture sodium cellulose xanthate (see Fig 3). This sodium cellulose xanthate is then added to a dilute solution of caustic soda, forming a viscous solution (from which the term viscose rayon derives). At this stage the finished fibre's lustre can be dulled by adding a white pigment, usually titanium dioxide: if this is not added the fibre will have a silk-like sheen.

2.2.c Stage Three: Ripening

The viscous solution is ripened for up to a day, while being repeatedly filtered. There is further breakdown of the longer cellulose molecules into shorter ones at this stage, contributing again to the fibre's poor wet properties. Once ripened, the viscosity of the solution rises when the cellulose begins to regenerate as the sodium cellulose xanthate starts to break down.

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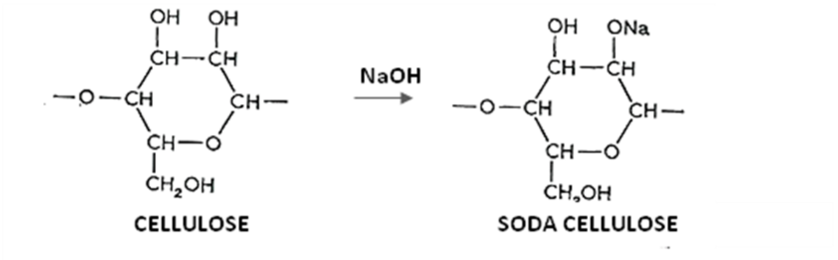
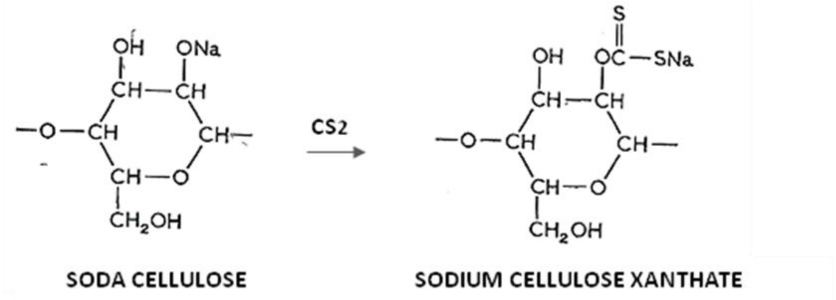
[55] Cook, J. Gordon, 'A: Natural Polymer Fibres, 1: Cellulose Fibres; Rayon', in *Handbook of Textile Fibres Vol. II Man-Made Fibres*, (Cambridge: Woodhead Publishing Ltd, 2009), pp. 11-20.

Fig. 3 The chemical changes occurring during the first three stages of the viscose process
From, J. Gordon Cook, ‘A: Natural Polymer Fibres, 1: Cellulose Fibres; Rayon’, in *Handbook of Textile Fibres Vol. II Man-Made Fibres,* (Cambridge: Woodhead Publishing Ltd, 2009), p. 12.

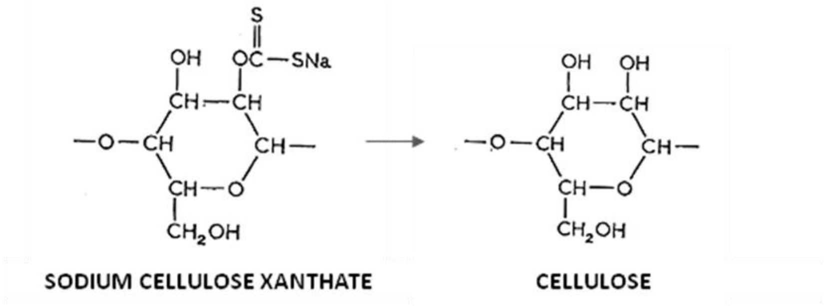
Stage One:



Stage Two:



Stage Three:

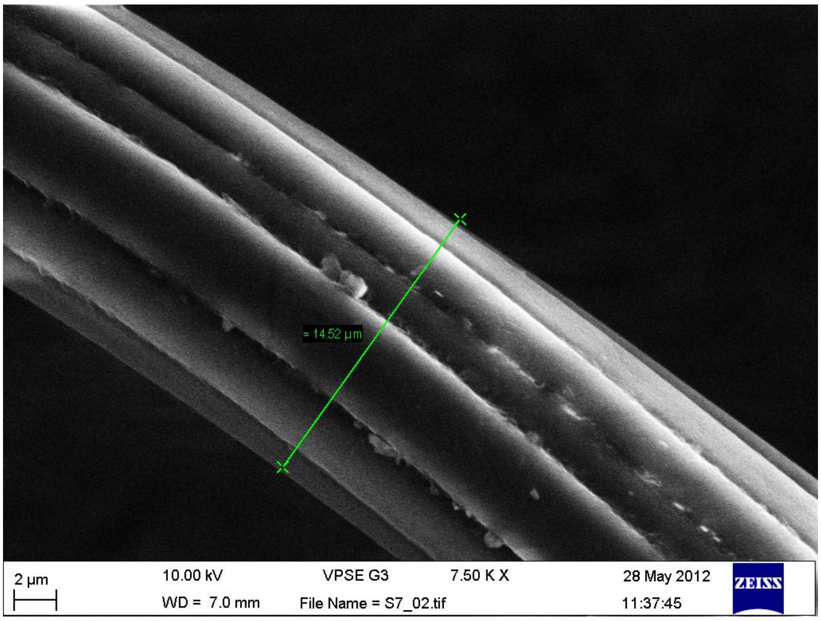


2.2.d Stage Four: Spinning

Finally, the viscous solution is forced through tiny holes in a spinneret (see fig. 4 overleaf), into a coagulating bath containing a mixture of acids and salts (typically sulphuric acid, sodium sulphate and zinc sulphate). At this stage the remaining sodium cellulose xanthate is converted back into cellulose, solubilising in the liquid bath to form filaments. The fibres are ordinarily extruded through circular-shaped holes. However the skin of the fibre dries more quickly than its core. This causes the skin to shrink, forming a striated surface (see fig. 5) that is visible with microscopy.[56]

Fig. 4 Fibres being extruded from a spinneret
From, St Philomenas Textile Dept Daily GCSE Revision Blog, *Properties of Synthetic Fibres*, 29 November 2010, (http://stphilstextiles.blogspot.co.uk/2010/11/properties-of-synthetic-fibres.html), accessed 2nd May 2012.

Fig. 5 Scanning Electron Microscopy image of a viscose rayon fibre, taken using *Carl Zeiss Sigma VP* equipment.



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[56] Hatch, (1993), p. 182.

2.3 Changes to the Processing of Viscose Rayon

Since inception, viscose rayon has been haunted by its poor wet properties, with obvious consequences for domestic washing as illustrated in Fig 6, a 1926 advert offering *rayon* care advice – Production of the fibre, however, has evolved. Modified manufacturing techniques resulted in fibres with improved wet strength and other properties, catering for a variety of end usages.[57] In its natural form, viscose rayon has a silk-like appearance and was first produced like that, but advances in manufacturing have enabled it to be processed with appearance and handle similar to fibres like cotton and wool.[58]

Fig. 6 Lux Soap Advert, 1926
From, Photoplay Vol. 30, July 1926, (Chicago: Photoplay Magazine Publishing Company, 1926).

RAYON

· · NEW · BEAUTIFUL, BUT SO FRAGILE *WHEN WET*

Launder it this one safe gentle way

YOU see it in Paris! Great French houses use rayon in their most stunning dress materials. Famous coutouriers take these and fashion frocks for all the smart world to wear!

You see it in New York! In the inimitable Fifth Avenue stores rayon grows every day more and more popular. New, lustrous, beautiful and such tempting prices!

"But," women ask, "how should we launder our rayon clothes—frocks, underthings, hosiery?"

Rayon is entirely different from silk—different from any other fabric! It is a man-made textile fibre, that temporarily loses much of its strength when wet. You must always launder rayon garments with infinite care!

Your silks and laces, your delicate woolens you've always trusted to Lux. For years Lux has refreshed them without injury. Now wash rayon, too, in Lux! But be sure to follow these washing directions carefully.

The safest way to wash rayon

WHIP up a tablespoonful of Lux in hot water. Add cold water until lukewarm. Take off your rings—they might tear the wet fibres. A rough finger nail, too, may catch in the fabric and cause damage.

Plunge your rayon garments into these fluffy, bubbling, *pure* Lux suds. Swirl them about, gently pressing the suds through the fabric. *Never* rub with a cake of soap! Then squeeze out the suds—*never* wring—and rinse several times in lukewarm water.

To dry, wrap the garment in a towel and squeeze out as much water as possible, do not twist. Then spread on a towel and pull into shape or hang the garment lengthwise over a clothesline or rack. Never use clothespins. Never dry in excessive heat. For rayon garments which require pressing, iron across the weave with a warm, not hot, iron.

Cut out these directions—keep them where you can refer to them next time you wash rayon! Lever Bros. Co., Cambridge, Mass.

Such exquisite underthings this year! In so many new and lovely colors! Silk, crêpe de chine, rayon. Don't ruin them by rubbing with cake soap! Launder them in Lux—directions on the package tell you how

In many of the smart, new frocks rayon is combined with silk, flannel, linen. More important than ever to launder them the safest, gentlest way—in sparkling, bubbling Lux suds—so harmless, so mild!

NOW a big, convenient package, too

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[57] Cook, (2009), p. 11.

[58] Hatch, (1993), p. 187.

The very first viscose rayon fibres from 1905 had very low dry and wet strength, relative to other fibre types, due to the minimal stretch incorporated in the spinning process leading to poorly aligned filaments.[59] Improvements made to manufacturing over proceeding years led to a one hundred percent increase in fibre strength by the 1920s.[60] However, viscose rayon still had low wet strength compared with other fibres, as described in the *Journal of Chemical Education* in 1927: 'The problems which still beset the industry [include] improvement in the strength and elasticity of the rayon particularly by overcoming the peculiar property of rayon which causes it to weaken when wet.'[61] 1920s-1940s research brought development and improvement which included variation of fibre lustre and staple fibre production.[62] Progress since the 1940s has involved physical modifications to viscose rayon fibres, such as alterations to cross-sectional shape and crimping,[63] and chemical modifications, such as cross-linked fibres which allow less movement of molecules, resulting in a reduction in water absorption and swelling when wet, creating a stronger wet fibre with better washability.[64]

Hatch describes the periods of major breakthroughs in viscose rayon production:

> 'The properties of regular viscose rayon remained about the same until 1940, when high-tenacity rayon was commercialized. Continued research led to what is now considered the greatest technological breakthrough in rayon fiber development – high-wet modulus rayon, which was commercially available in 1955. Crimped high-performance rayon followed in 1977. Differences in the dry and wet stress-stain [sic] properties of these fibres make significant differences in the maintenance of fabrics made from them.'[65]

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[59] Teresa A. Summers, Billie J. Collier, John R. Collier and Janice L. Haynes, 'History of Viscose Rayon', in Raymond B. Seymour and Roger S. Porter (eds.) *Manmade Fibers: Their Origin and Development*, (Essex: Elsevier Science Publishers Ltd, 1993), p. 77.

[60] John W. S. Hearle, 'Chapter 8 – Physical Structure and Fibre Properties', in Calvin Woodings (ed.), *Regenerated Cellulosic Fibres*, (Cambridge: Woodhead Publishing in association with The Textile Institute, 2001), p. 214.

[61] Milton J. Shoemaker, 'Viscose Rayon', in *Journal of Chemical Education,* October 1927 (USA: ACS Publications, 1927), p. 1268.

[62] Summers, (1993), p. 80.

[63] Cook, (2009), p.21.

[64] Cook, (2009), p. 38.

[65] Hatch, (1993), p. 186.

It is evident that processing variations can produce viscose rayon fibres with quite different properties. Hearle explains that: '[…] the minor differences between fibres of nominally the same type from different manufacturers or from different dates, lead to a wide range of values of physical properties.'[66]

For conservation it would be extremely difficult to determine the exact processing of a particular example of viscose rayon and use it to predict that fibre's properties. Awareness, however, of the variety of different processing techniques exploited in viscose rayon production clarifies how and why these fibres may behave in quite different ways. Therefore for conservation, it should be understood that viscose rayon fibres may not necessarily act within a common format. Knowledge of manufacturing changes reveals that older examples are likely to display poorer wet properties than later examples.

2.4 Nomenclature

Viscose rayon has had a number of names. Below are details of the evolving nomenclature which could help elucidate the true meaning of fibre names provided in labels on historical objects made of man-made fibres. In addition, Appendix 1 lists some past trade names for viscose rayon and other regenerated cellulosic fibres.

As previously mentioned, early man-made fibres were all marketed under the generic term *artificial silks*. This began to be seen as a misnomer, as illustrated by J. Merritt Matthews in 1913: 'It would be better to call the artificial product "imitation silk," or give it some name more distinctive of its origin and true nature [...].'[67] Hatch describes how: 'Consumers and retailers were confused about the identity of the fiber.'[68] A new generic name was sought to replace *artificial silk.* A 1925 American competition led to the new name *rayon* ('ray of light') derived from the French, in reference to the lustre of these early fibres.[69] *Rayon* was officially recognised by the United States Federal Trade Commission in the same year.[70] The first sanctioned classification of rayon came from the American Society for Testing Materials in 1926 where it was defined as: '[…] A generic term for filaments made from

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[66] Hearle, (2001)p. 208.

[67] J. Merritt Matthews, *The Textile Fibres: Their Physical Microscopical and Chemical Properties*, (London: Chapman & Hall, 1913), p. 352.

[68] Hatch, (1993), p. 181.

[69] Summers, (1993), pp. 78-79.

[70] Handley, (1999), pp. 24-25.

various solutions of modified cellulose by pressing or drawing the cellulose solution through an orifice and solidifying it in the form of a filament, or filaments, by means of some precipitating medium.'[71] This definition included cellulose acetate as well as regenerated cellulosic fibres.

In 1926, the Silk Association of Great Britain approved the term rayon and it was later adopted by Courtaulds in 1929 – although in Britain the term was used for any man-made fibre, not just those obtained from cellulose.[72-73]

Despite the creation and sanctioning of this new name, the term was not adopted universally as shown by Fig. 7 (overleaf), a 1937 British *Needlewoman* magazine advert for 'artificial silk'. However, an example of the use of the term 'rayon' is given in an advert from a 1938 *Needlewoman* edition, Fig. 8 (overleaf).

The American Federal Trade Commission refined the classification of rayon in 1954, to refer solely to regenerated cellulosic fibres. By 1956 it appears that the same classification had also been adopted in Britain, as illustrated in an industry publication from the time: 'There has been a tendency to include both viscose rayon and acetate under the title "rayon", but this is misleading and both in Great Britain and the U.S.A., acetate is now sold under its own name.'[74]

In 1960, the *1958 Federal Trade Commision Fiber Identification Act* came into force, making the use of generic names in America compulsory.[75] In this, rayon was defined as: 'A manufactured fibre composed of regenerated cellulose, as well as manufactured fibres composed of regenerated cellulose in which substituents have replaced not more than 15 per cent of the hydrogens of the hydroxyl groups.'[76] This still remains its classification today.

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[71] Summers, (1993), pp. 78-79.

[72] Handley, (1999), pp. 24-25.

[73] Textile Mercury Annuals, (1953), p. 666.

[74] *Man-Made Fibres*, (London: n.p., 1956), p. 6.

[75] Cook, (2009), pp. xxvi-xxviii

[76] Cook, (2009), pp. xxvi-xxviii

Fig. 7 Huttons Fabrics Advert, 1937
From, *Needlewomen*, Vol. 15, No. 178, June 1937, (London: n.p., 1937), p. 3.

Make your Holiday Frocks from HUTTONS Fabrics—

cool, colourful, smart!

Lovely linens and chic linen tweeds, charming new prints in profusion, cool gay cottons—Huttons choice of Summer Frock Fabrics is more enchanting than ever this season! They're materials that make up beautifully, that wash to perfection . . . *and they offer you really worthwhile savings at Huttons Bargain Prices.* Send for Free Patterns.

"NEVER-FADE" IRISH LINEN

A smooth fine weave that makes up delightfully. All the 44 exquisite shades are fast to sun, soap and sea air. Width, 36-in.
Per Yard 3/-

CHIC LINEN TWEED

One of the most fashionable dress materials of the season. In fine Standard Quality, 12 charming colourings. 36-in. wide.
Per yard 2/11

FADELESS DRESS POPLIN

Firm and beautifully silky in finish. Many attractive shades to choose from—all guaranteed fast. Width, 36-in.
Per Yard 3/-

FLOWERED DRESS PRINTS

Demure and gay in small three-tone designs. Easy to wash and iron. Width, 36-in. Amazing value!
Per Yard 11d.

SPUN SHEEN

A lovely artificial silk fabric, patterned in clever dot and floral designs, with good choice of both light and dark tones. Width, 36-in.
Per Yard 2/1½d.

UNCRUSHABLE CREPE

Printed in charming multi-coloured effects. Marvellously practical for wash and wear. Width, 36-in.
Per Yard 1/0½d.

PRINTED MAROCAIN

A fine silky crepe that makes up admirably. Dark backgrounds prevail—brown, black, royal and navy—patterned in distinctive new designs. Width, 36-in.
Per Yard 2/11

Frocks made to measure

If you haven't time for dress-making, remember that Huttons make up your frocks for you at very low cost. All the fabrics listed here can be made to measure in a choice of Ten New Paris Styles, one of which is illustrated above. Post the Coupon for Patterns, Illustrated Frock Leaflet and Self-measurement Chart.

LINENS FOR EMBROIDERY too—

A NEW FANCY HEAVY EMBROIDERY LINEN CRASH, Natural shade, made with a beautiful round thread so easy to count and draw, suitable for wool or ordinary embroidery. 18-in wide.
Per yard 1/6

36-in. wide, **2/9 per yard.**
41-in. wide, **3/2 per yard.**
45-in. wide, **3/6 per yard.**

CREAM EMBROIDERY LINEN for all classes of embroidery work. 45-in. wide.
Per yard 2/2

54-in. wide, **2/6 per yard.**

TRACED IRISH LINEN NOVELTIES.—Write for illustrated Leaflet. Patterns of our embroidery Linens and Crashes also sent free on request. There are amazing values in Remnant Bundles of Irish Linen for Needlework, too. Complete Bargain Catalogue FREE

Huttons Goods are ALL BRITISH. NO CUSTOMS BARRIER BETWEEN GREAT BRITAIN AND N. IRELAND. Postage paid on Orders 20/- or over—under 20/- add postage. This postage applies to U.K. only. C.O.D. orders welcomed, but it is cheaper to send cash for small orders, which can then be sent to you by letter post. Goods returnable for exchange or refund of money if not satisfied.

Huttons

Post this Coupon Now!

G. R. HUTTON & Co. Ltd.,
90, Main St., Larne, Ulster,
N. Ireland.

Please send me FREE, Patterns of your Summer Frock Fabrics and leaflet illustrating the made-to-measure styles available.

Name ....

Address ....

....

....

(Post in unsealed envelope, stamped ½d.—U.K. only.)

Fig. 8 Sparva Fabric Advert, 1938
From, *Needlewomen*, Vol. 16, No. 188, April 1938, (London: n.p., 1938), p. 31.

YOU can have more lovely frocks and undies, in brighter, fresher styles, and still save on your dress bills, by choosing "SPARVA"! The low prices of "SPARVA" mean that you can look and feel your best on every occasion with the minimum expenditure. 100 beautiful shades and a host of printed patterns to choose from. Use "SPARVA" for casements, too—it is colour-fast to sunlight, sea and the wash-tub.

THERE are several imitations of these beautiful fabrics. Look for the name on the selvedge—it is your guarantee of good service.

TWO charming sister fabrics—"SPARVASYLK," spun in fine Rayon in plain and fancy weaves; "SPARVA"-SPUN, a printed material in delightful designs, with a lovely, lasting lustre. Both 36 inches wide, 1s. 0d. per yard.

ASK to see patterns at your usual Drapers or Stores. If any difficulty, write for shade card and name of nearest retailer to—"SPARVA," 45, Sparva House, York Street, Manchester.

When replying to advertisements always mention "The Needlewoman."

In 1977 the International Organisation for Standardisation (ISO) published *ISO 2076:1977(E)* stating that the terms cupro, viscose and modal would be used by them to identify the separate regenerated cellulose fibres (lyocell not in production yet) without reference to the word *rayon* because, it concluded, this meant different things in different countries.[77] However, this practice was not enforced internationally, and fibre names still differ between countries. For example, Gohl and Vilensky explain in *Textile Science,* 1983, that the terms *cuprammonium* and *polynosic* are used to refer to *cupro* and *modal* respectively in Australia.[78]

Today, British Standard *BS ISO 2076:2010* enforces the use of the names *cupro*, *viscose*, *modal* and *lyocell*, in Britain, to classify the different processing of each.[79] Differences still exist internationally however. For example, in America under the Federal Trade Commission Textile and Wool Acts, the term *rayon* is preferred to *viscose*,[80] although both are legal. For the purpose of this paper the name *viscose rayon*, as used in a variety of textile literature,[81-82] has been employed to provide clarity for conservators in different countries.

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[77] Gohl, (1983), p. 54.

[78] Gohl, (1983), p. 54.

[79] British Standard, (2010), p. 2.

[80] Bureau of Consumer Protection Business Center, *Threading Your Way Through the Labeling Requirements Under the Textile and Wool Acts,* May 2005, (http://business.ftc.gov/documents/bus21-threading-your-way-through-labeling-requirements-under-textile-and-wool-acts#names), accessed 5th June 2012.

[81] Cook, (2009), p. 11.

When researching available conservation literature, no discussion was found of whether the changes in meaning outlined above could result in old labels or past museum records relaying old terminology with connotations different to current usage. There is potential here for misidentification of fibres. This issue combined with difficulties in identifying man-made fibres (see Chapter 4) could have repercussions for conservation treatment decisions, especially in relation to the wet strength of viscose rayon.

Conclusion

This chapter has highlighted the history of viscose rayon production, alongside the history of its poor wet properties. This has shown that older versions of viscose rayon have poorer wet properties than later versions, with significant improvements to fibres occurring from the 1940s onwards. For textile conservation, poor wet strength has clear implications for wet cleaning treatments.

The range of manufacturing modifications has also been described which give rise to a diversity of fibre properties for different examples of viscose rayon. This could complicate predicting how viscose rayon fibres may behave during wet cleaning.

This chapter has also introduced issues surrounding the fibre identification of viscose rayon and other man-made fibres which are explored in Chapter 4. This has relevance for wet cleaning treatment decisions if early viscose rayon does show a marked weakness when wet.

The information provided in this chapter has been the impetus for fibre identification analysis and strength testing, described in Chapters 4 and 5.

In the next chapter the chemical properties of viscose rayon are discussed, to show why viscose rayon fibres have such poor wet properties.

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[82] Morton, (1986).

Chapter 3 – Fibre Properties Relating to Wet Strength

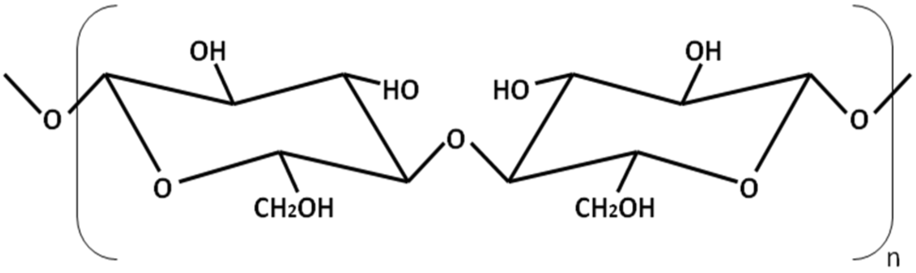
Introduction

This chapter deals with the fibre properties which dictate viscose rayon's poor wet properties. Comparisons are made between rayon and some other fibre types for contextualisation.

3.1 Chemical Properties

Fig. 9 shows the cellulobiose monomer unit of the viscose rayon polymer, identical to that of cotton and other natural cellulosic fibres. As detailed in Chapter 2, the structural alignment of viscose rayon cellulose is affected by its processing. Cook explains: 'The cellulose of rayon has been modified to some degree during manufacture, and the alignment of the molecules is not identical with that of natural fibres such as cotton.'[83] During regeneration, the cellulose crystallises in the form of cellulose II with polymer-chains orientated anti-parallel.[84] This is different to the natural cellulose I form of cotton, in which the polymer-chains are orientated parallel.[85] The anti-parallel orientation of cellulose II in viscose rayon contributes to its reduced strength.[86]

Fig. 9 Cellubiose Unit



Viscose rayon has a high degree of amorphous regions, typically between 60-65 percent amorphous, with 35-40 percent crystalline regions.[87-88] As water is able to enter and be

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[83] Cook, (2009), p. 35.

[84] Hearle, (2001), pp. 203-204.

[85] Hearle, (2001), pp. 203-204.

[86] Hearle, (2001), p. 206.

[87] Hatch, (1993), p. 182.

absorbed by fibres in its amorphous regions, viscose rayon a very absorbent fibre.[89] The high absorbency of viscose rayon causes the fibres to swell in diameter when wet between 50-133 percent, as well as to grow in length.[90]

3.2 Degree of Polymerisation (DP)

DP relates to polymer chain lengths within a fibre. Chapter 2 described how viscose rayon processing causes some degradation of its cellulose, resulting in shorter polymer chains. Viscose rayon's DP is approximately 400.[91] This is markedly different compared to natural cellulose fibre cotton which has a DP of 6,000-10,000. The low DP of viscose rayon cellulose contributes to its reduced strength. Hearle explains: 'Degree of polymerisation affects the strength of all fibres […] short segments will break first and longer ones later. The low strengths at low DP are due to the fact that the molecules are not long enough to bridge many gaps and many free ends will emerge from crystallites.'[92]

3.3 Degree of Polymerisation, Orientation and Amorphousness and their Affects on Wet Properties

Low DP, poor orientation and high absorption all contribute to viscose rayon's poor wet strength. The low DP and poor orientation of the cellulose II molecules results in fewer hydrogen bonds in the fibres than are possible in natural cellulose:[93] hydrogen bonding being in part responsible for the high strength of the natural cellulose fibre cotton.[94] The highly amorphous structure allows easy entry of water molecules during wetting, causing a significant amount of direct hydrogen bonds to break.[95] The result is a much weaker fibre when wet. Gohl and Vilensky have described wet viscose rayon as: 'only half as strong as when dry.'[96]

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[88] Amorphous regions are weaker than crystalline regions in textile fibres. See: Gohl and Vilensky, (1981), p. 28.

[89] Hearle, (2001), p. 230.

[90] Hatch, (1993), p. 184.

[91] Hatch, (1993), p. 182.

[92] Hearle, (2001), pp. 204-205.

[93] Gohl and Vilensky (1981), p. 58.

[94] Gohl and Vilensky, (1981), p. 45.

[95] Hearle, (2001), p. 219.

[96] Gohl and Vilensky (1981), p. 58.

As already mentioned, another implication of the high absorbency of viscose rayon fibres is its effect on dimensions.[97]

These characteristics have clear implications for wet cleaning. As Hatch describes, 'It is difficult to launder regular rayon fabrics due to the behaviour of rayon fibres in water and to the use of water-soluble sizings. Drycleaning [sic] is generally recommended.'[98] Although there was not time to investigate it within the scope of this project, a comparison between conservation solvent cleaning (dry cleaning) and wet cleaning would be useful.

Conclusion

The information gathered above elucidates viscose rayon's poor wet properties. It has been decided to focus on viscose rayon's low wet strength for testing. Observations relating to dimensional change in wet viscose rayon fibres, as mentioned above, will also be made during strength testing though it was not possible to include this within the restraint of this paper and these will therefore be included in an appendix as reference for potential future research.

Part Two of this paper now presents analysis and testing carried out for this project, based on research discussed in Part One.

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[97] Hatch, (1993), p. 184.

[98] Hatch, (1993), p. 184.

Chapter 4 – Fibre Identification Analysis

Introduction

Fibre identification analysis undertaken on objects acquired for research is described in this chapter. The aim of this chapter was to illustrate how successful *basic* analytical techniques are for the accurate identification of viscose rayon alongside other man-made fibres. In addition, it was intended that analysis would inform which three objects were carried forward for testing described in Chapter 5.

First, the rationale for fibre identification analysis will be presented, followed by a description of identifying features of viscose rayon alongside other man-made fibres. The main fibre identification analysis follows, beginning by presenting the objects acquired for analysis and ending with the results from five fibre identification techniques which were chosen for analysis: any fibre content information on garment labels, sensory perceptions, microscopy, acetone solubility tests and ATR-FTIR spectroscopy.

4.1 Rationale for Fibre Identification Analysis

The ability to distinguish between the many man-made fibres has been problematic for a long time. The 1953 *Textile Mercury Cotton Yearbook* states*:* 'With the introduction of new yarns and fibres every year, their accurate identification becomes increasingly difficult and more and more a job for the specialist.'[99]

The 1985 Edition of *Identification of Textile Materials (ITM)* provides a helpful photomicrograph catalogue including some man-made fibres.[100] However, studying these reveals that cross-characteristics exist between viscose rayon and other man-made fibres (see fig. 10: although, it should be noted that calcium alginate is used mainly for medical dressings and is unlikely to be encountered by textile conservators).[101] Similarly, viscose-rayon's cross-section resembles that of other man-made fibres (see fig. 11).

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[99] Textile Mercury Annuals, (1953), p. 700.

[100] The Textile Institute, (1985), pp. 51-133.

[101] The Textile Institute, (1985), p. 29.

Fig. 10 Similarities in microscopic appearance: viscose rayon (left), cellulose acetate (middle), calcium alginate (right).
From, The Textile Institute, *Identification of Textile Materials*, Seventh Edition, (Manchester: The Textile Institute, 1985), pp. 95-104.

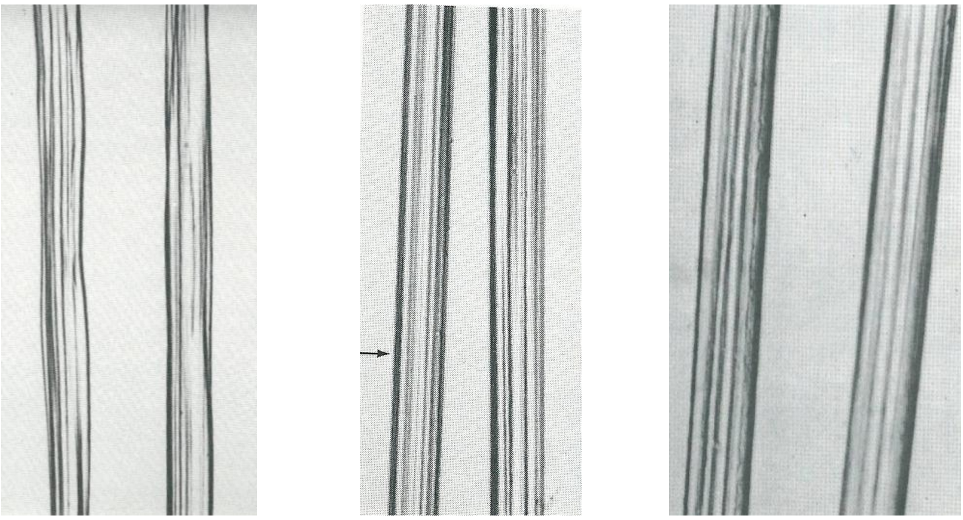
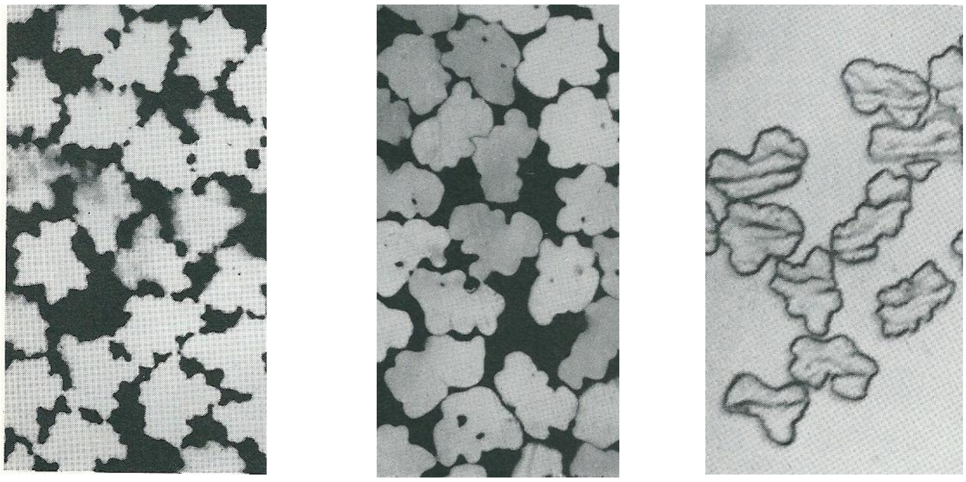


Fig. 11 Similarities in cross-sections: viscose rayon (left), cellulose acetate (middle), calcium alginate (right).
From, The Textile Institute, *Identification of Textile Materials*, Seventh Edition, (Manchester: The Textile Institute, 1985), pp. 95-104.



ITM states that one can differentiate between man-made fibres by refining microscopy techniques. However, the techniques described are complex, involving the use of birefringence and interference microscopy with a polarising microscope:[102] techniques used in forensics which require specialist training.[103] For the average conservator, results using

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[102] The Textile Institute, *Identification of Textile Materials*, Seventh Edition, (Manchester: The Textile Institute, 1985), pp. 138-141.

[103] Lewarne, Karsten and Lemire, (2009), p. 301.

these methods are not easily interpretable. *ITM* explains how fibre identification can be complicated by different finishing techniques: ‘crimping, bulking, or texturizing processes, chemical treatments, and finishing processes can very easily distort the original form, especially of man-made fibres [...].’[104]

Recognising the problems in distinguishing man-made fibres, Lewarne et al. in 2009 investigated polarising microscopy and other techniques to develop a ‘protocol’ for discerning viscose rayon from other man-made fibres: ‘It was found that polarizing microscopy was not as effective a tool for identifying rayon as anticipated […] birefringence did not consistently lead to successful fibre identification.’[105] Furthermore: ‘Many factors, including dark dyes and the high level of experience required of the operator, confounded results.’[106] The ‘protocol’ developed by Lewarne et al. incorporated a combination of fibre identification tools to progressively eliminate possible fibre types. They described their own success in using a combination of *basic* microscopy, acetone solubility tests and hot-stage microscopy to identify viscose rayon from other fibres.[107]

The use of infrared spectroscopy (FTIR and NIR) in fibre identification was outlined in 1985 in *ITM*.[108] More recently, there have been a number of publications written by museum professionals on the use of this technique to distinguish between hard-to-identify man-made materials, including textiles. In 2002 Chris Paulocik and R. Scott Williams discussed the superiority of infrared spectroscopy over other techniques.[109] Then, in 2007 Emma Richardson, Graham Martin and Paul Wyeth published two articles each discussing the use of NIR spectroscopy at a different museum,[110-111] to quickly identify modern materials: ‘The ability to characterise such collections quickly, on site and without the need for sampling,

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[104] The Textile Institute, (1985), p. 136.

[105] Lewarne, Karsten and Lemire, (2009), p. 303.

[106] Lewarne, Karsten and Lemire, (2009), p. 303.

[107] Lewarne, Karsten and Lemire, (2009), pp. 303-304.

[108] The Textile Institute, (1985), p190-194.

[109] Paulocik and Williams, (2002), p. 82.

[110] Richardson, Martin and Wyeth, (2007), p. 257-263.

[111] Emma Richardson, Graham Martin and Paul Wyeth, ‘On-Site Collections Management: NIR Characterisation and Condition Monitoring of Modern Textiles’, in Conservation Science 2007, Papers from the Conference held in Milan, Italy, 10-11 May 2007, (London: Archetype, 2008), pp. 262-269.

would offer a major advancement in object-based research and the care and curation of artefacts.'[112]

Reviewing information found during research on identifying viscose rayon fibres, it was decided it would be useful to supplement this project with an analysis of the value of using a combination of fibre identification techniques on objects acquired for research. It was intended that analysis would firstly add to the existing body of knowledge relating to the identification of viscose rayon and man-made fibres. Secondly, it would illustrate how successful the use of a combination of *basic* techniques was for objects acquired for this research, which will be useful for conservators who do not have access to infrared analytical equipment. Additionally, the use of ATR-FTIR would demonstrate how accurate these *basic* methods are.

One object acquired for analysis was chosen as it was labelled as the regenerated cellulosic fibre 'lyocell'. It was felt that this would be useful for comparison against viscose rayon, to show any similarities and differences in appearance between the two. In addition, two objects of viscose rayon fibre blended with another fibre type were obtained for analysis, to analysis how fibre blends, made of yarns containing more than one fibre type, can affect fibre identification.

Below will now be described those features typical of viscose rayon.

4.2 Appearance of Viscose Rayon Fibres

The microscopic appearances attributed to man-made fibres as a whole is described in *ITM:* 'Smooth profile without scales or convolutions, with or without striations – indicating man-made fibres or silk.'[113] These common features make the task of identifying man-made fibres a challenge: with the additional similarity in appearance to silk.

Normal viscose rayon fibres do however have some definition in appearance, caused by how the fibres are processed, as described in Chapter 2. They typically have a microscopic appearance which shows striations longitudinal to fibre length and a cross-section showing lobed-edges (see figs. 10-11 above). Although, as mentioned above, these features are also present on some other fibre types.

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[112] Richardson, Martin and Wyeth, (2007), p. 262.

[113] The Textile Institute, (1985), p. 136.

4.3 Objects Acquired for Analysis

Eleven objects were acquired from different eras covering the history of viscose rayon, with the supposition that they could be, or were, examples of viscose rayon. Little or no information was available on the provenance of these objects. Dating was based on construction and style. It was considered acceptable to take samples from these objects, as they were not precious and the research carried out would be beneficial for conservation. The objects used in analysis are now shown in figs. 12-22.

Fig. 12 Object 1, child’s jacket, circa 1920s

Above right: Maker’s label

Fig. 13 Object 2, woman’s printed scarf, circa 1930s

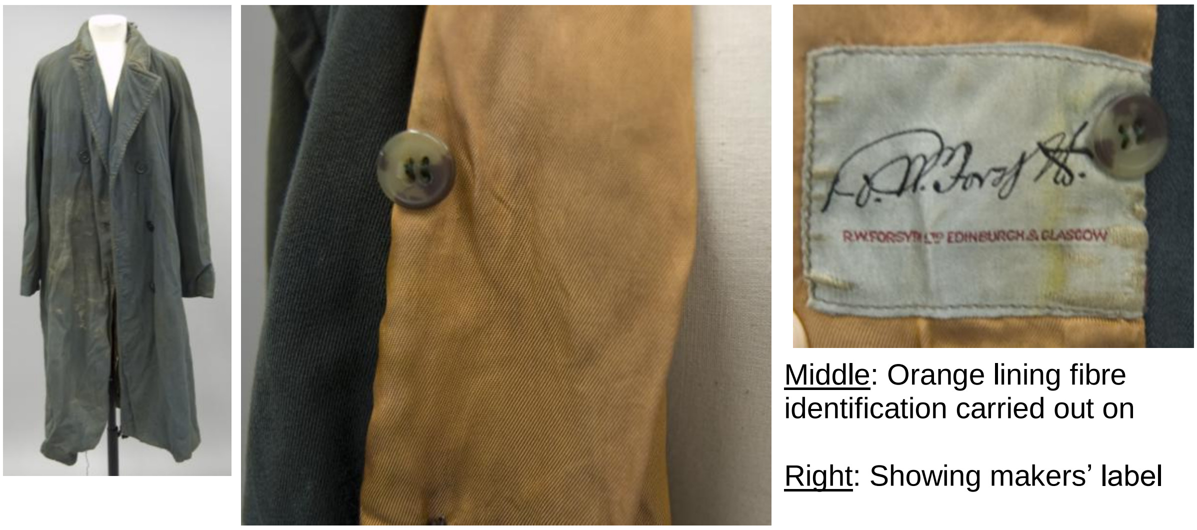
Above right: Fabric content label

Fig. 14 Object 3, woman’s printed dress, circa 1940s

Fig. 15 Object 4, Courtaulds fabric, circa 1940s

Above right: Courtaulds stamp

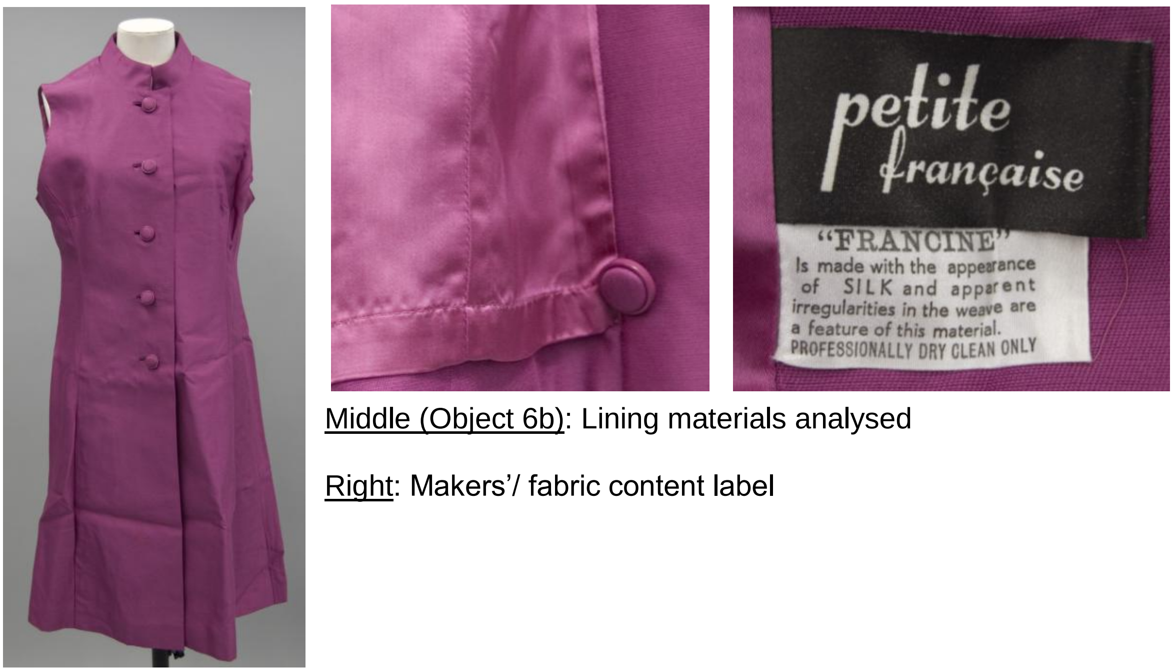
Fig. 16 Object 5, man's coat orange lining, circa 1950/60s



Middle: Orange lining fibre identification carried out on

Right: Showing makers' label

Fig. 17 Object 6a (outer material) / 6b (lining), woman's dress, circa 1960s

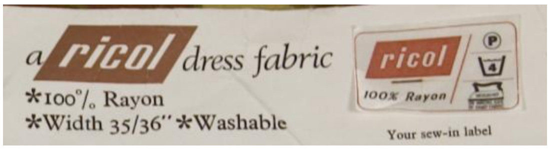


"FRANCINE"
Is made with the appearance of SILK and apparent irregularities in the weave are a feature of this material.
PROFESSIONALLY DRY CLEAN ONLY

Middle (Object 6b): Lining materials analysed

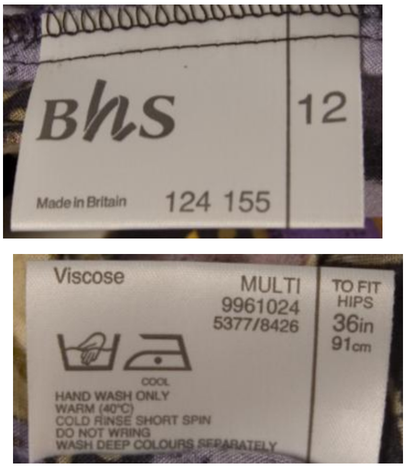
Right: Makers'/ fabric content label

Fig. 18 Object 7, printed fabric, circa 1960s



Right: Makers'/fabric content label

Fig. 19 Object 8, woman's printed trousers, circa 1980/90s



Top/Bottom Right:
Makers'/fabric content label

Fig. 20 Object 9, woman’s dress, circa 1990/2000s

Middle and Right:
Makers’/fabric content label

Fig. 21 Object 10, viscose and acetate blend, 2012

Fig. 22 Object 11, viscose and silk blend, 2012

4.4 Fibre Identification Analyses

For analysis, it was decided to include information from fabric content labels, as it was felt that this may illustrate issues with past connotations of nomenclature, described in Chapter 2. It was also decided that sensory perceptions would show how far touch and appearance can be tools for identifying man-made fibres. In addition, there were three fibre identification techniques available at the centre for analysis: microscopy, solubility tests and ATR-FTIR spectroscopy. Analysis was conducted in this order:

1. Fabric Content Labels
2. Sensory Perceptions
3. Microscopy
4. Acetone solubility test
5. ATR-FTIR

Thoughts on possible fibre type after each analysis were informed by any preceding analyses, to build a collective picture of what each object's fibre type was likely to be. The results of all analyses are now given in order over the next pages.

4.4.a Analysis 1: Fabric Content Labels

Table 1 below summarises information in any garment labels pertaining to fabric content. This information took account of issues raised by past nomenclature.

Table 1 Fibre identification from garment labels

Fibre ID Object Number	Fabric Content Stated in Garment Labels
1	None
2	‘All Foreign Rayon’
3	None
4	None
5	None
6a	‘Francine is made with the appearance of silk’
6b	‘Francine is made with the appearance of silk’
7	‘100% Rayon’
8	‘Viscose’
9	‘100% Tencel® Lyocell’
10	‘Viscose & Acetate Blend’
11	‘Viscose & Silk Blend’

4.4.b Analysis 2: Sensory Perceptions

Table 2 below gives results following sensory perceptions, based on fibre stiffness, typically associated with man-made fibres, and high lustre, also typically associated with earlier man-made fibres (although older man-made fibres often have less sheen).

Table 2 Fibre identification from sensory perceptions

Fibre ID Object Number	Possible fibre type?
1	Silk
2	Man-made fibre, possibly viscose rayon or cellulose acetate
3	Viscose rayon
4	Cotton or man-made, possibly viscose rayon
5	Man-made, possibly viscose rayon
6a	Man-made
6b	Man-made, possibly viscose rayon or cellulose acetate
7	Man-made, possibly viscose rayon
8	Viscose rayon
9	Polynosic rayon
10	Viscose & Acetate Blend
11	Viscose & Silk Blend

4.4.c Analysis 3: Microscopy

Fibre samples were taken from both weave directions for each object, labelled yarn 1 and yarn 2 as warp and weft were indiscernible for most objects.

Fibre samples were mounted onto slides using *Eukitt® quick-hardening mounting medium*[114] and observed under a micrscope at different magnifications (see fig 23).

Photomicrographs of all samples were taken under 10x magnification with a *Leica ATC 200* transmissive microscope. These are shown in figs.24-35. For each, characteristics observed during microscopy which influenced thoughts of possible fibre type, as presented in Table 3 below, are given.

It was noted during microscopy that dark specks – possibly dye present in fibres – on some fibre samples made it difficult to discern features. For example, fig. 31 shows yarn 2 of Object 7, where it was difficult to ascertain whether striations were present.

Fig. 23 Conducting microscopy

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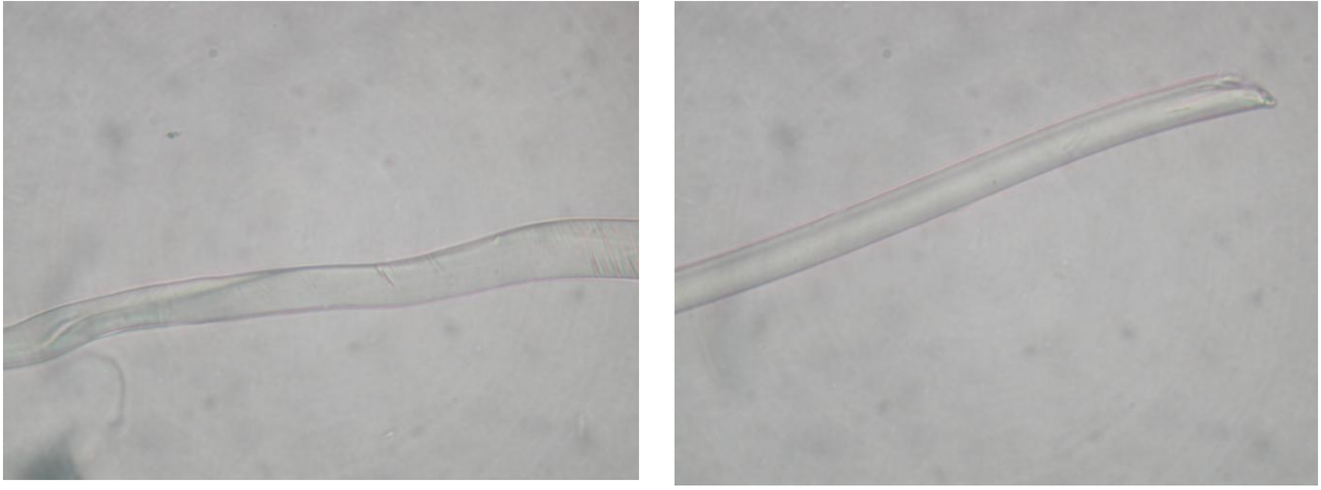
[114] Eukitt®: Poly(butyl methacrylate-co-methyl methacrylate) - manufactured by Fluka Analytical, BioChemika.

Table 3 Fibre identification following microscopy

Fibre ID Object Number	Possible fibre type?	
	Yarn 1	Yarn 2
1	Silk	
2	Man-made, possibly cuprammonium rayon or cellulose acetate	
3	Viscose rayon	
4	Cotton	Man-made, possibly viscose rayon
5	Man-made, possibly viscose rayon	
6a	Man-made, possibly viscose rayon	
6b	Man-made, possibly viscose rayon	
7	Man-made, possibly viscose rayon or cellulose acetate	
8	Viscose rayon	
9	Polynosic rayon	
10	Viscose & Acetate Blend	
11	Viscose & Silk Blend	

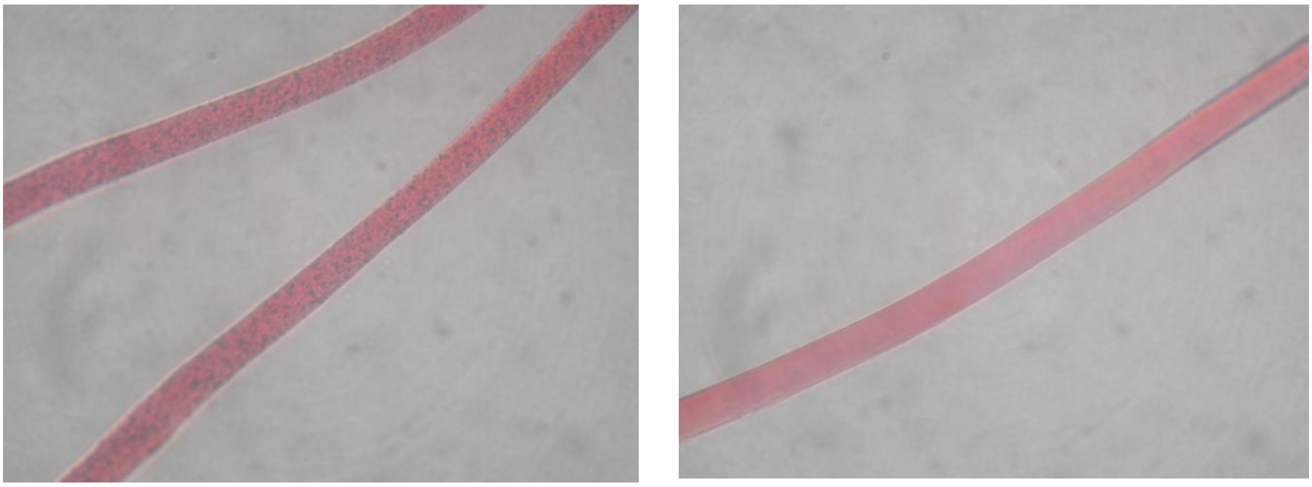
Fig. 24 Photomicrographs: Object 1



Yarn 1:smooth, flat, slight twisting

Yarn 2: smooth, round

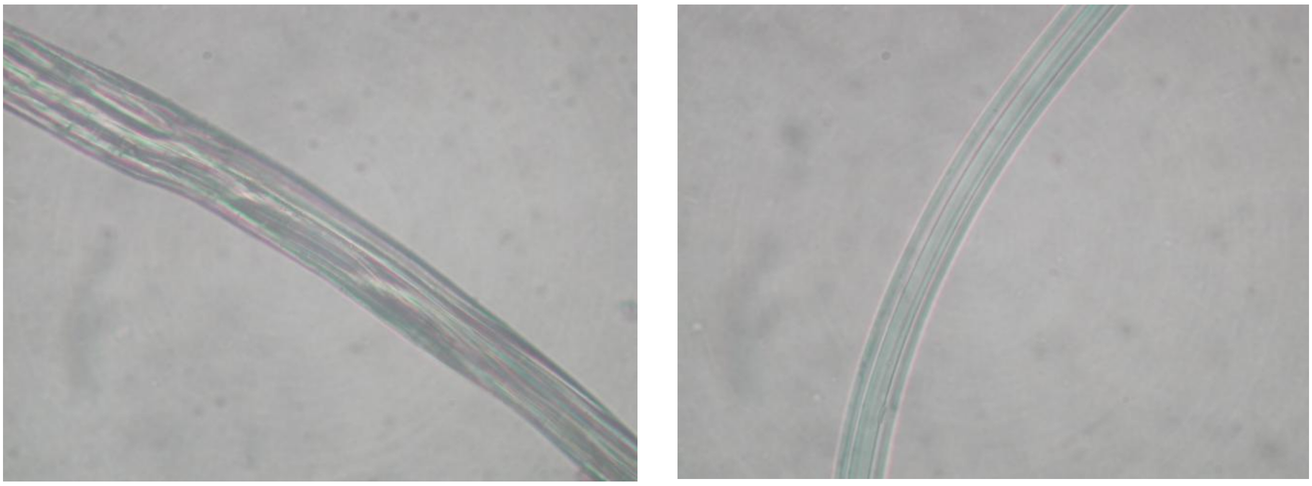
Fig. 25 Photomicrographs: Object 2



Yarn 1:smooth, round, dark specks

Yarn 2: smooth, round

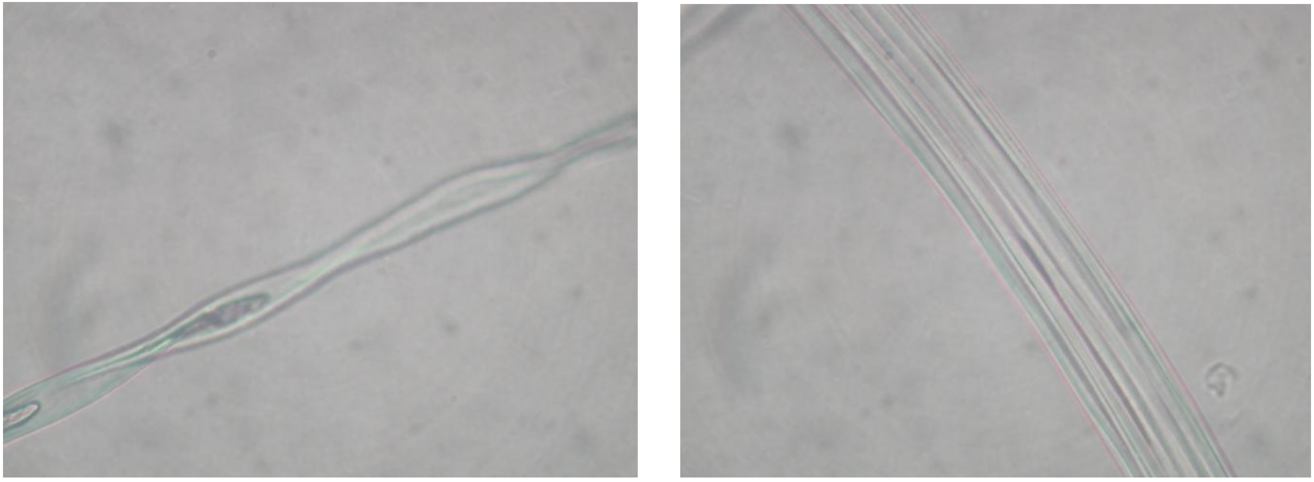
Fig. 26 Photomicrographs: Object 3



Yarn 1:round, well-defined striations

Yarn 2: smooth, round, striations

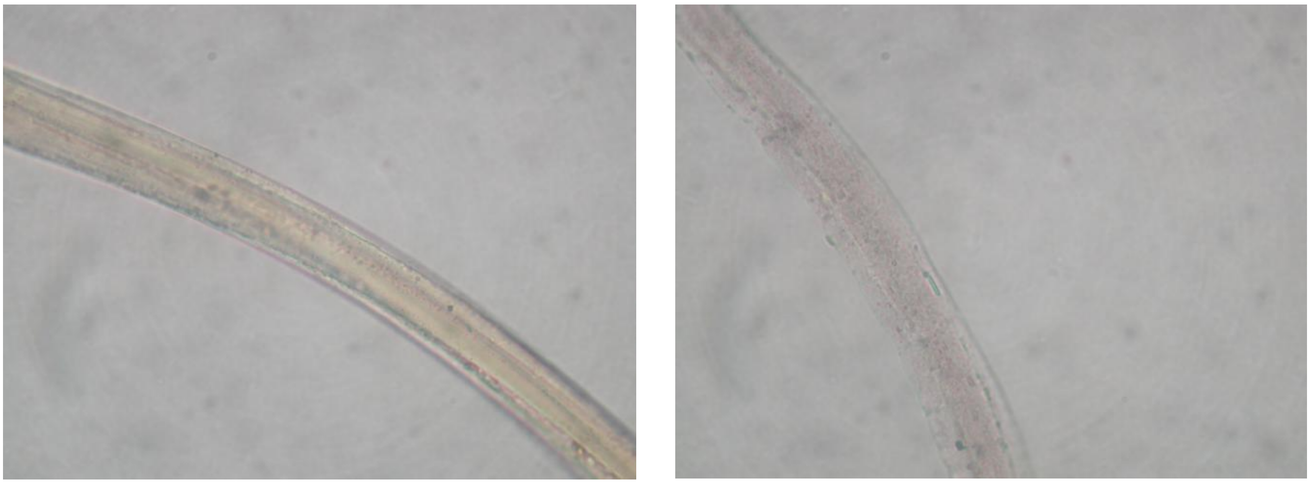
Fig. 27 Photomicrographs: Object 4



Yarn 1:flat, twisted

Yarn 2: round, well-defined striations

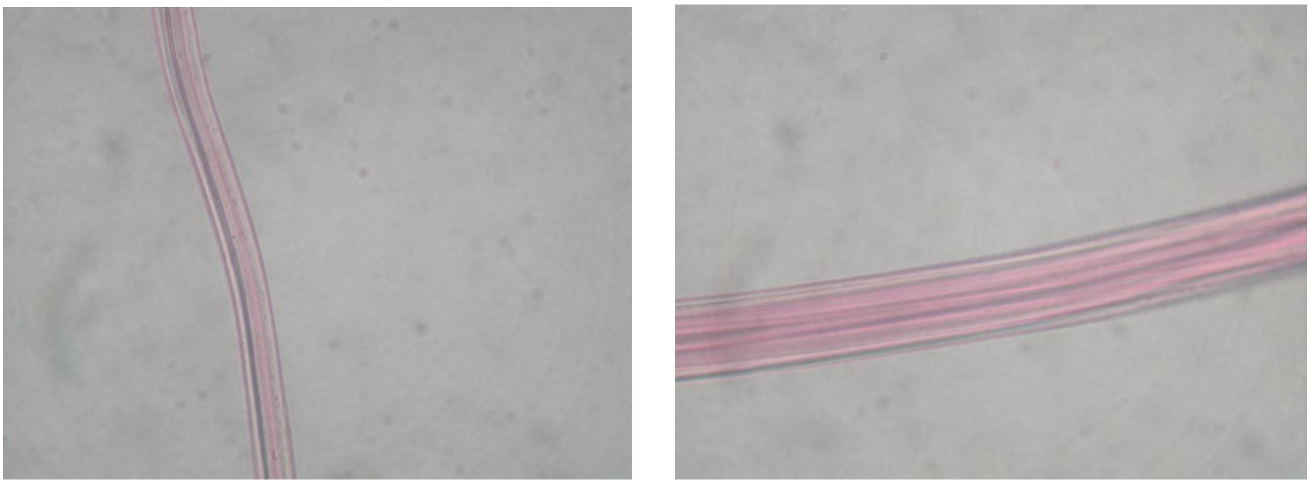
Fig. 28 Photomicrographs: Object 5



Yarn 1:round, some striations

Yarn 2: round, some darker specks

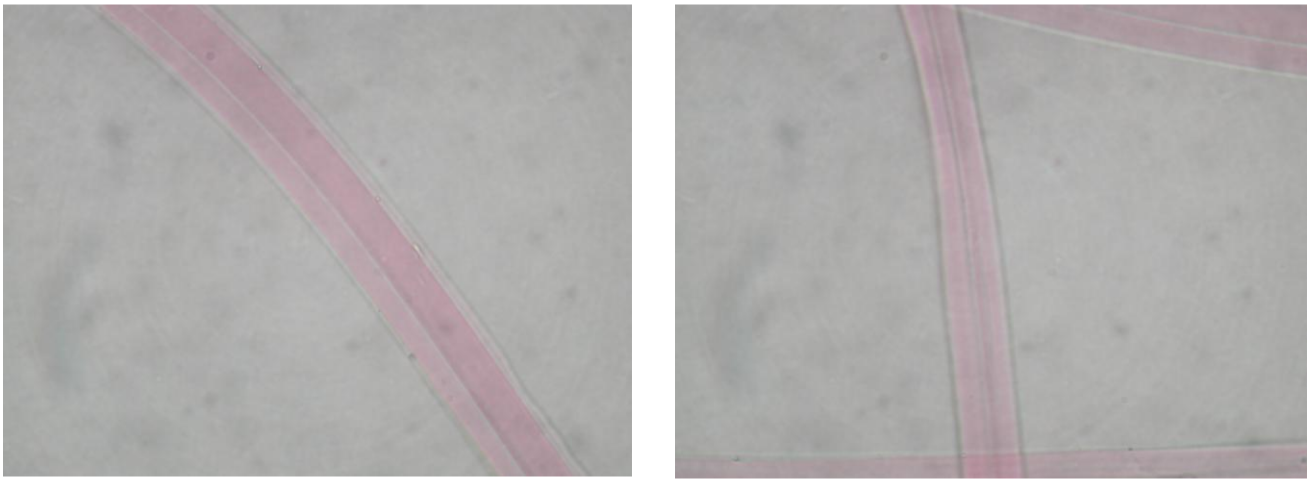
Fig. 29 Photomicrographs: Object 6a



Yarn 1:smooth, round with striations

Yarn 2: smooth, round, well-defined striations

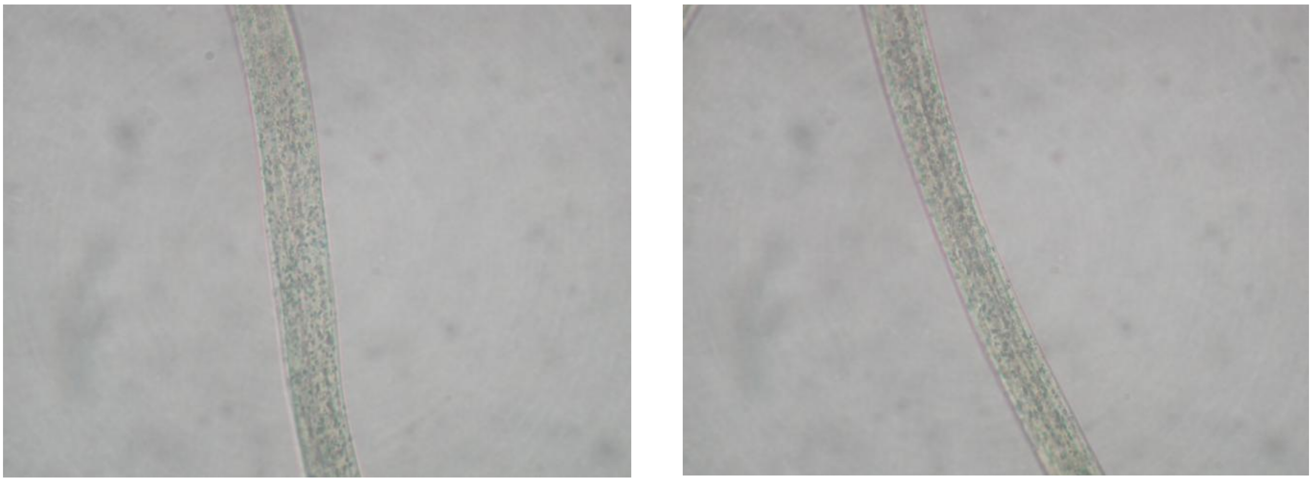
Fig. 30 Photomicrographs: Object 6b



Yarn 1:smooth, round, slight striations

Yarn 2: smooth, round, slight striations

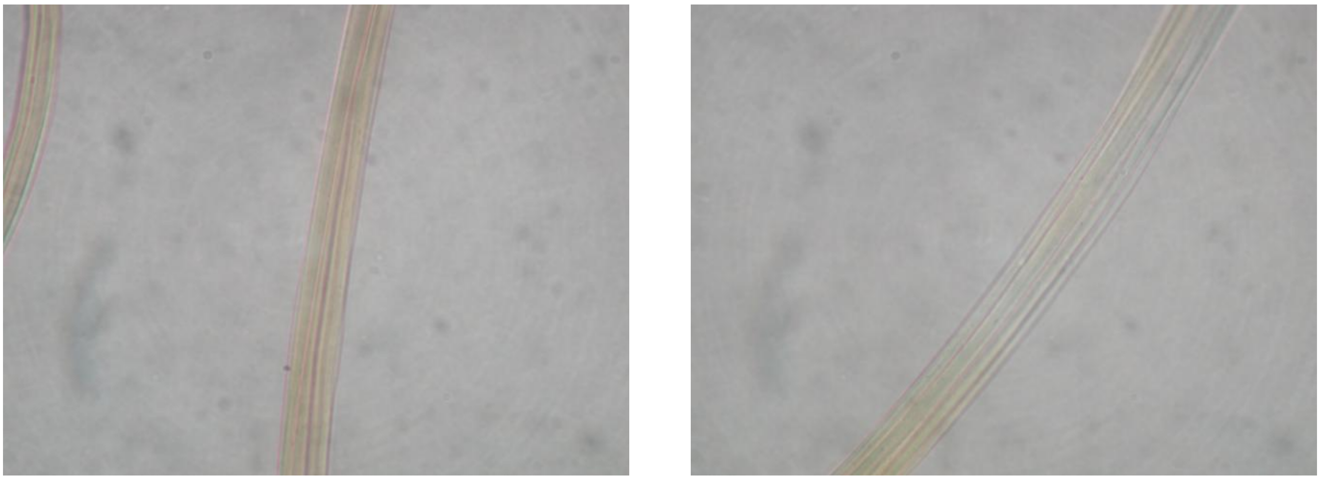
Fig. 31 Photomicrographs: Object 7



Yarn 1:smooth, round, dark specks

Yarn 2: smooth, round, dark specks, possibly striations

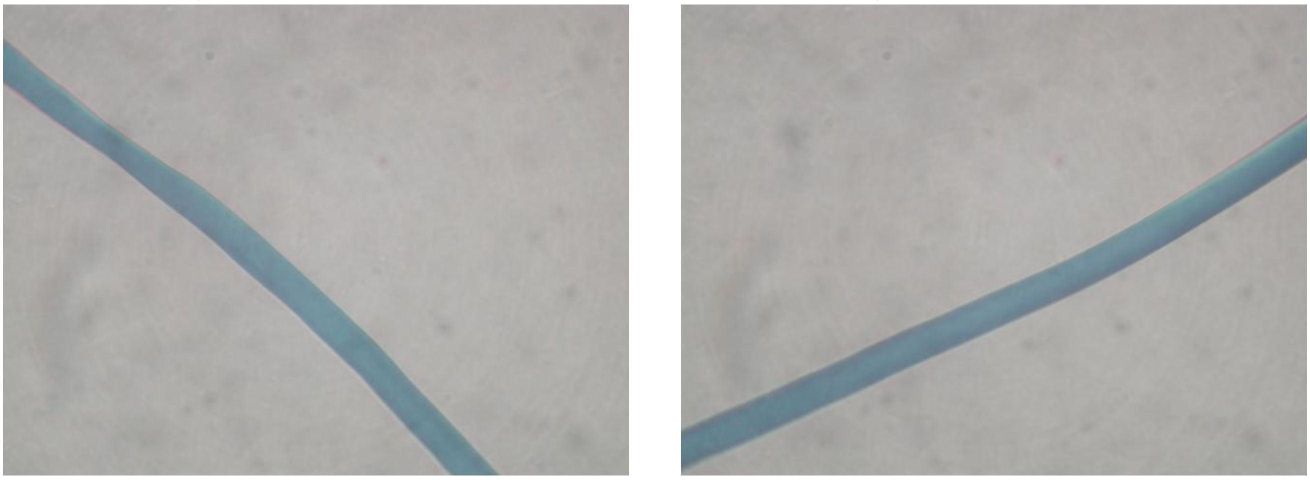
Fig. 32 Photomicrographs: Object 8



Yarn 1:smooth, round striations

Yarn 2: smooth, round striations

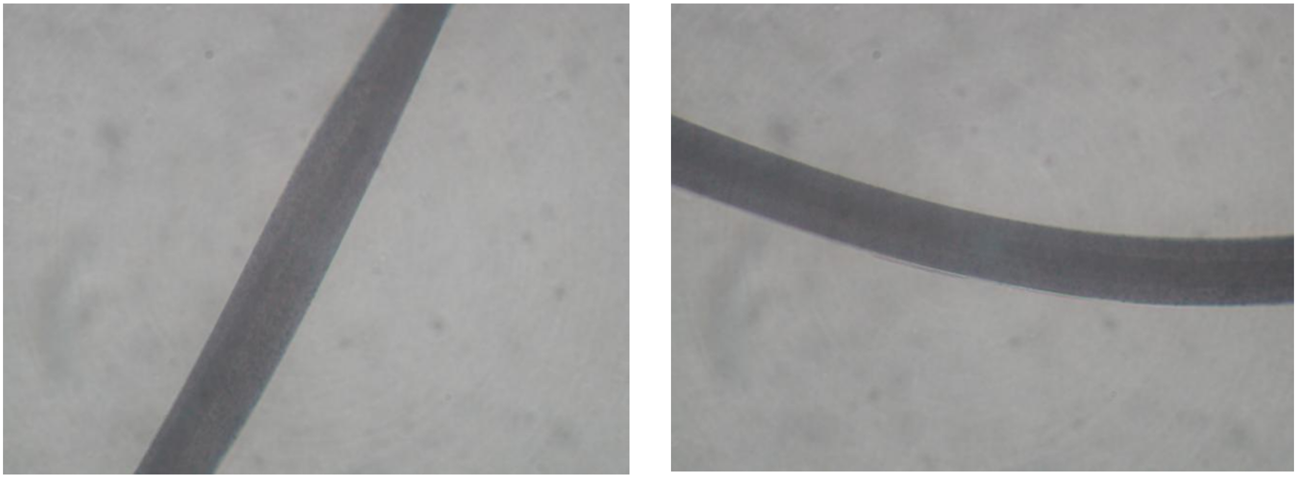
Fig. 33 Photomicrographs: Object 9



Yarn 1:smooth, round

Yarn 2: smooth, round

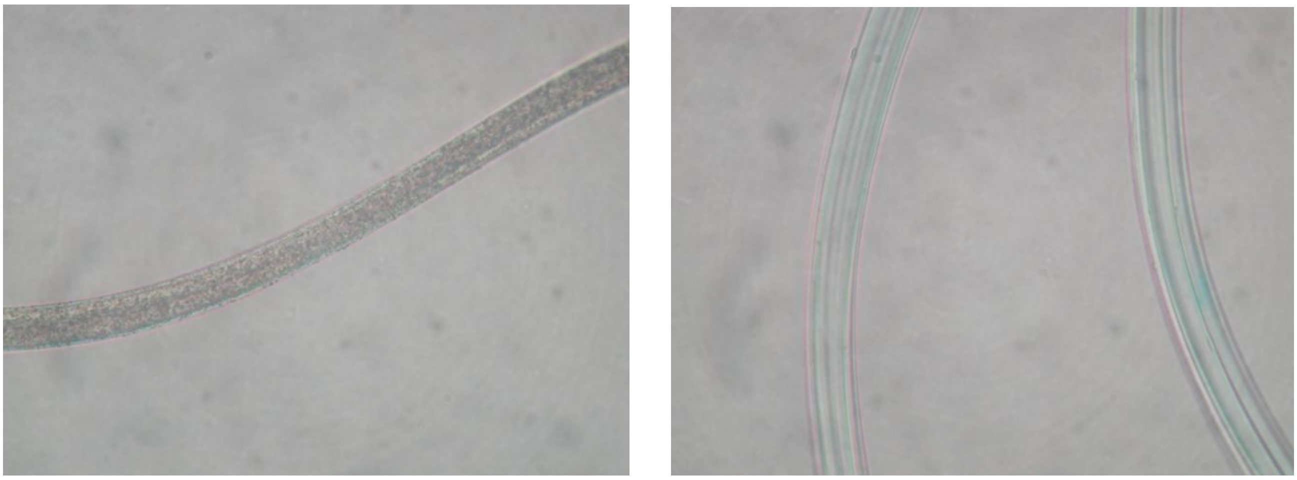
Fig. 34 Photomicrographs: Object 10



Yarn 1:smooth, round

Yarn 2: smooth, round

Fig. 35 Photomicrographs: Object 11



Yarn 1:smooth, round, dark specks

Yarn 2: smooth, round, well-defined striations

4.4.d Analysis 4: Solubility Tests

Acetone[115] solubility tests were conducted to try to eliminate any objects which may be cellulose acetate and which, as illustrated earlier, can have a similar microscopic appearance to viscose rayon. Under a fume hood (fig. 36), samples of both yarns from each object were placed in a glass Petri dish and covered with a few drops of neat acetone using a glass pipette. If the fibre was solubilised it could be identified as cellulose acetate (see fig. 37).[116] Any fibres identified as cellulose acetate are shown in Table 4 overleaf.

Fig. 36 Conducting solubility tests

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[115] Acetone manufactured by Fisher Chemical, Code A/0520/PB17, general purpose grade.

[116] The Textile Institute, (1985), 182-183.

Fig. 37 Fibre sample from Object 6b solubilised in acetone, indicating the fibre is cellulose acetate

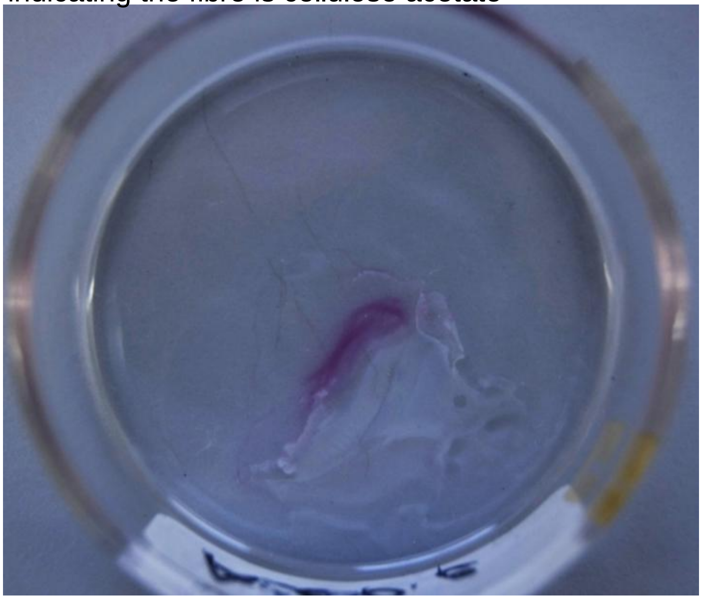

Table 4 Results following solubility tests

Fibre ID Object Number	Possible fibre type?	
	Yarn 1	Yarn 2
1	Silk	
2	Possibly cuprammonium rayon	
3	Viscose rayon	
4	Cotton	Viscose rayon
5	Viscose rayon	Cellulose acetate
6a	Man-made, possibly viscose rayon	
6b	Cellulose acetate	
7	Viscose rayon	
8	Viscose rayon	
9	Polynosic rayon	
10	Viscose & Acetate Blend (fibres partially solubilised)	
11	Viscose & Silk Blend	

4.4.e Analysis 5: ATR-FTIR Spectroscopy

ATR-FTIR Spectroscopy was carried out using a *Perkin Elmer Spectrum One FT-IR Spectrometer* and *Spectrum* version 5.0.1 software. A total of 16 scans were done for each spectrum. This technique was quick and non-destructive, allowing fibres to be analysed in situ (see fig. 38). Prior to testing, reference spectra of fabric swatches obtained from *Whaleys (Bradford) Ltd*[117] were taken, to use for comparison with spectra from objects. Thoughts on fibre type following ATR-FTIR are shown in Table 5 overleaf, followed with a discussion of observations from analysis.

Fig. 38 Object 9 during ATR-FTIR analysis

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[117] http://www.whaleys-bradford.ltd.uk/

Table 5 Results following ATR-FTIR Spectrocospy

Fibre ID Object Number	Fibre type identified with ATR-FTIR Spectroscopy	
	Yarn 1	Yarn 2
1	Silk	
2	Cuprammonium rayon	
3	Viscose rayon	
4	Cotton	Viscose rayon
5	Viscose rayon	Cellulose acetate
6a	Viscose rayon	
6b	Cellulose acetate	
7	Viscose rayon	
8	Viscose rayon	
9	Polynosic rayon	
10	Spectra showed as cellulose acetate	
11	Spectra showed as viscose rayon	

Initially, just the front surface of fabric was analysed for each object. However, it was realised that fabric mixes,[118] as determined from solubility tests, gave ambiguous results using this method. For example, the spectra of the front surface for Object 5 indicated closest resemblance to cellulose acetate (see Graph 1). But, solubility tests had shown yarn 1 was cellulose acetate; yarn 2 was not. Yarns 1 and 2 from Object 5 were then retested individually producing different results for each yarn in coherence with solubility test results (see Graphs 2 and 3). It was therefore decided to test yarns 1 and 2 separately for all ATR-FTIR analysis and the results discussed now have been obtained as such. It should be noted that this did necessitate removal of original stitching in some objects, to expose a raw fabric edge from which to separate yarn 1 from yarn 2 (warp from weft), this may be undesirable in the context of conservation where it is ideal to avoid removing original stitching.

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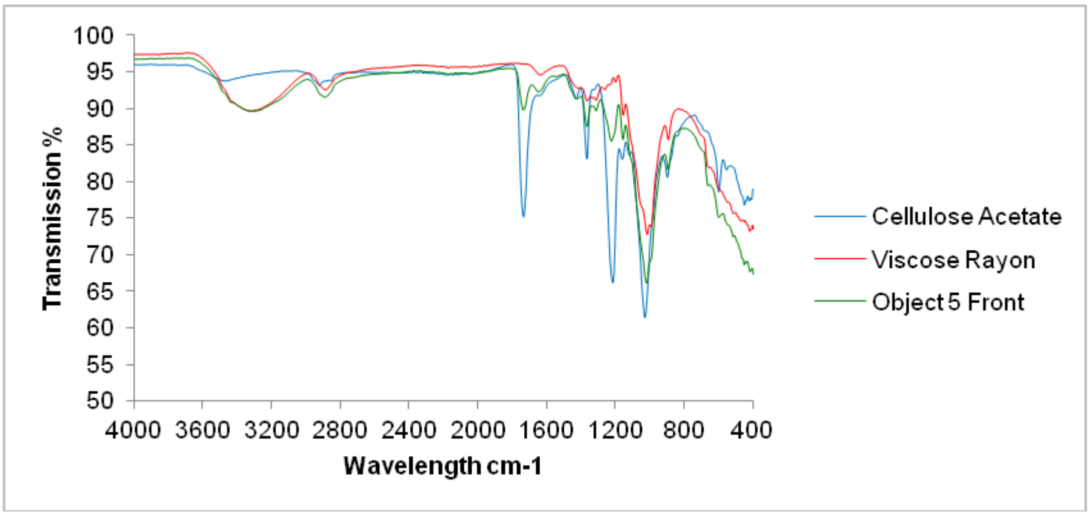
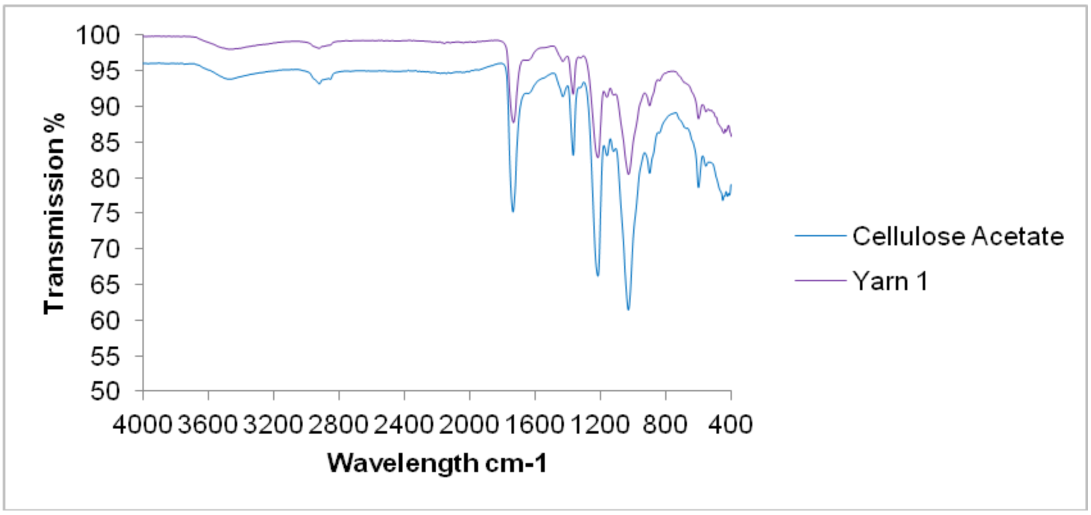
[118] *Fabric mixes* contain a different fibre type for warp and weft. *Fibre blends* are different and involve the use of a mixture of fibres in a single yarn/filament.

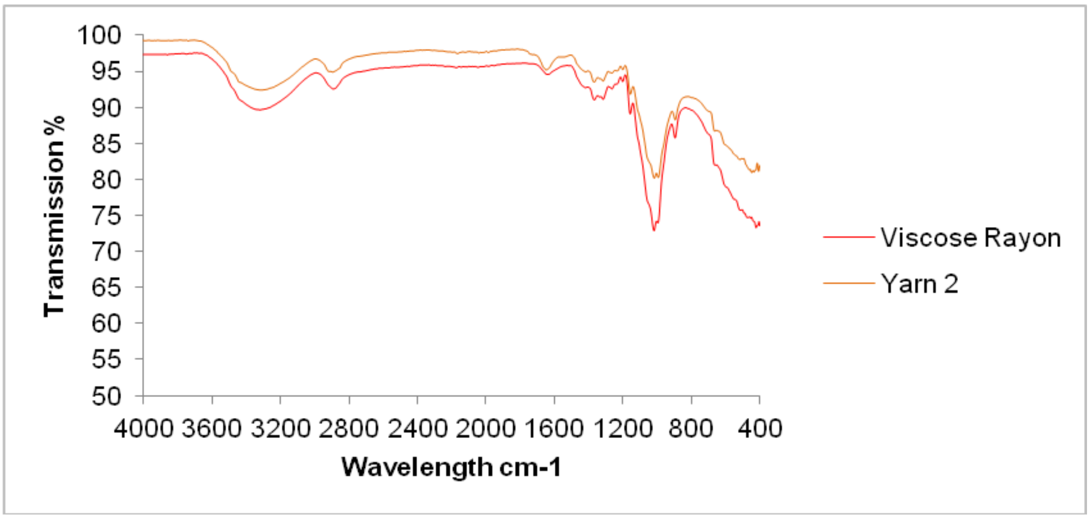
Graph 1 ATR-FTIR Spectra, Object 5, fabric front, with viscose rayon and cellulose acetate references



Graph 2 – ATR-FTIR Spectra, Object 5, yarn 1, with reference of cellulose acetate



Graph 3 ATR-FTIR Spectra, Object 5, yarn 2, with reference of viscose rayon



ATR-FTIR spectroscopy was successful in distinguishing between the majority of different fibre types in objects, which included viscose rayon, cellulose acetate, silk and cotton with the exception of Objects 10 and 11 which contained fibre blends. (These are discussed in more detail later.) Some of the ATR-FTIR spectra are now used to illustrate observations during analysis. All other spectra are in Appendix 2 for reference.

Before analysis, it was considered that there may be issues in using ATR-FTIR to distinguish between different cellulosic fibres which are all composed of cellubiose, as outlined in Chapter 3. Whilst the natural cellulose fibres cotton and flax have distinguishing features identifiable via microscopy (see fig. 39), it was felt that, as mercerised cotton fibres are smooth and round in appearance, similar to cupro, modal and lyocell (see fig. 40), this could potentially cause uncertainty in results. During ATR-FTIR spectroscopy the opportunity to test this issue arose when analysing Object 4, which had shown during microscopy that yarn 1 was cotton, yarn 2 possibly a man-made fibre. When analysing the spectra for Object 4, a subtle distinction in each yarn was observed and, following research, this difference was shown to exist in the area of the spectra which recorded hydrogen bonding on cellulose, as described below.

Fig. 39 Microscopy images of cotton (left) and flax (right)
From, The Textile Institute, *Identification of Textile Materials, Seventh Edition*,
(Manchester: The Textile Institute, 1985), pp. 74 and 78.

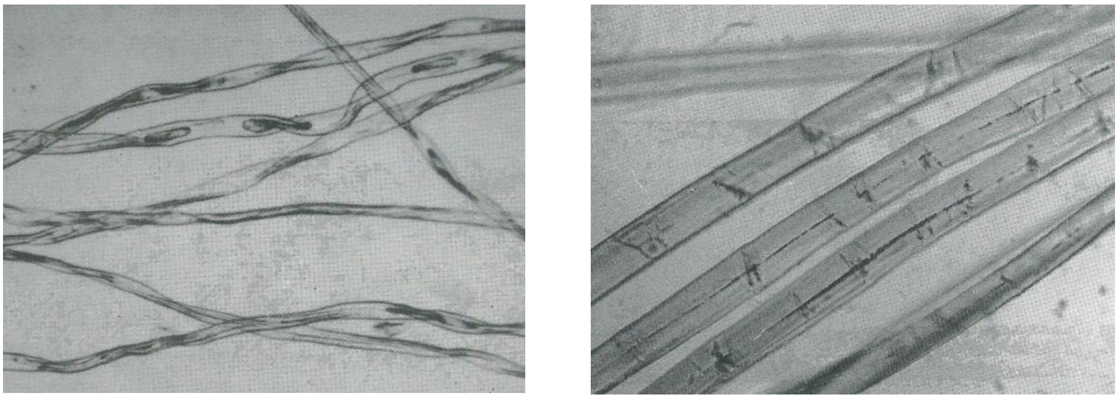
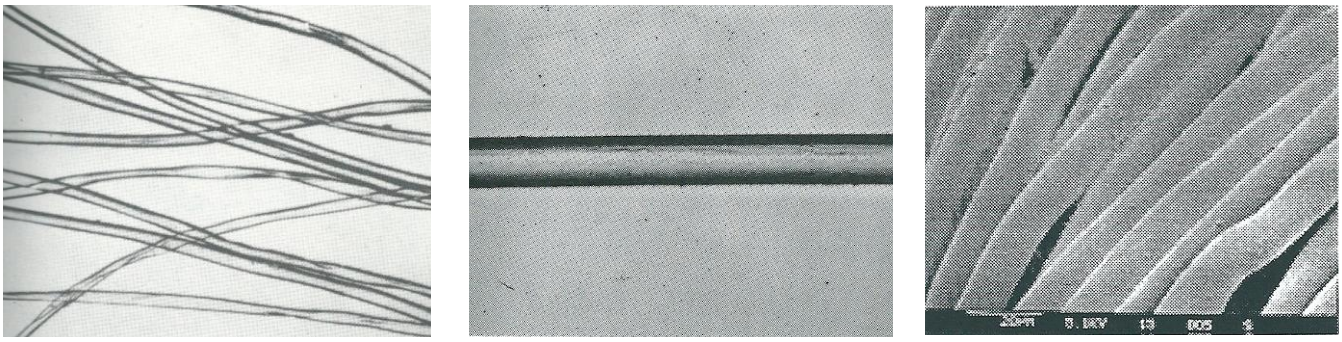
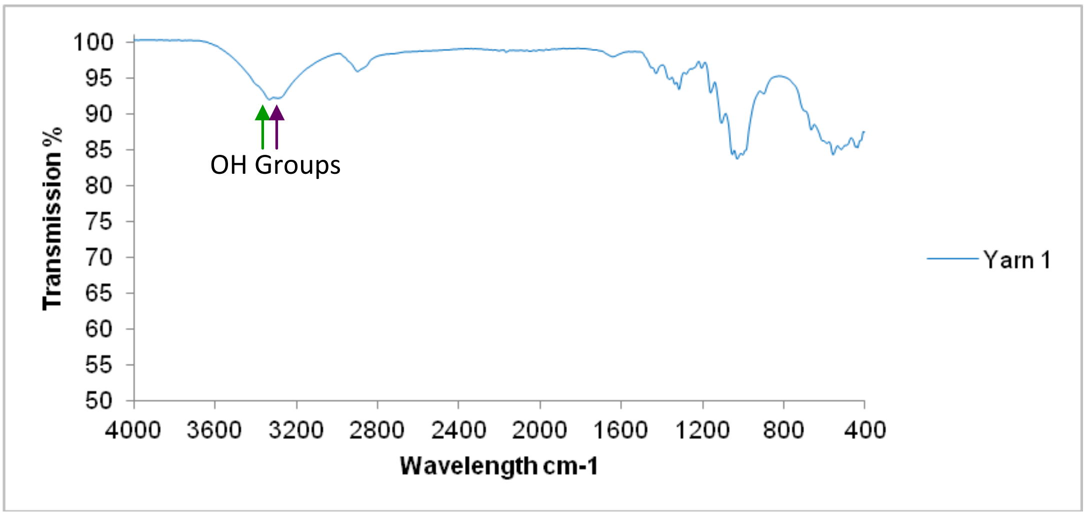


Fig. 40 Microscopy images of mercerised cotton (left), cupro (middle) and lyocell (right)
From, The Textile Institute, *Identification of Textile Materials, Seventh Edition*, (Manchester: The Textile Institute, 1985), pp. 75 and 101.
And, Kathryn L. Hatch, *Textile Science,* (St. Paul, USA: West Publishing Company, 1993), p. 197.



Graph 4 shows the spectrum for yarn 1, cotton, from Object 4. The trough in the area marked 'OH groups' is the area of the spectrum associated with OH bonding which accounts for the main hydrogen bonding present in the polymer of cotton.[119-120] A dip in spectra around 3,335 cm^{-}1 is attributed to intra-molecular hydrogen bonds, [121] as represented by the green arrow. And, a dip in spectra around 3,285 cm^{-}1 is attributed to intermolecular hydrogen bonds, [122] as represented by the purple arrow.

Graph 4 ATR-FTIR Spectra, Object 4, yarn 1: cotton



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[119] Noureddine Abidi, Eric Hequet and Luis Cabrales, 'Applications of Fourier Transform Infrared Spectroscopy to Study Cotton Fibers', in Goran Nikolic (Ed.), *Fourier Transforms - New Analytical Approaches and FTIR Strategies,* (N.p.: Intech, 2011), pp. 94-95.

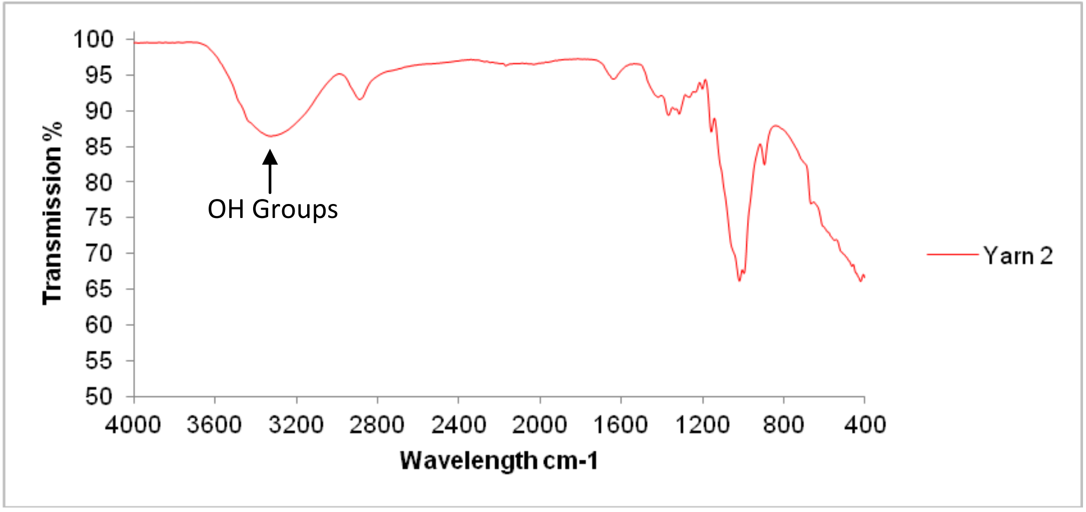
[120] Gohl and Vilensky, (1981), p. 17.

[121] Abidi, (2011), pp. 94-95.

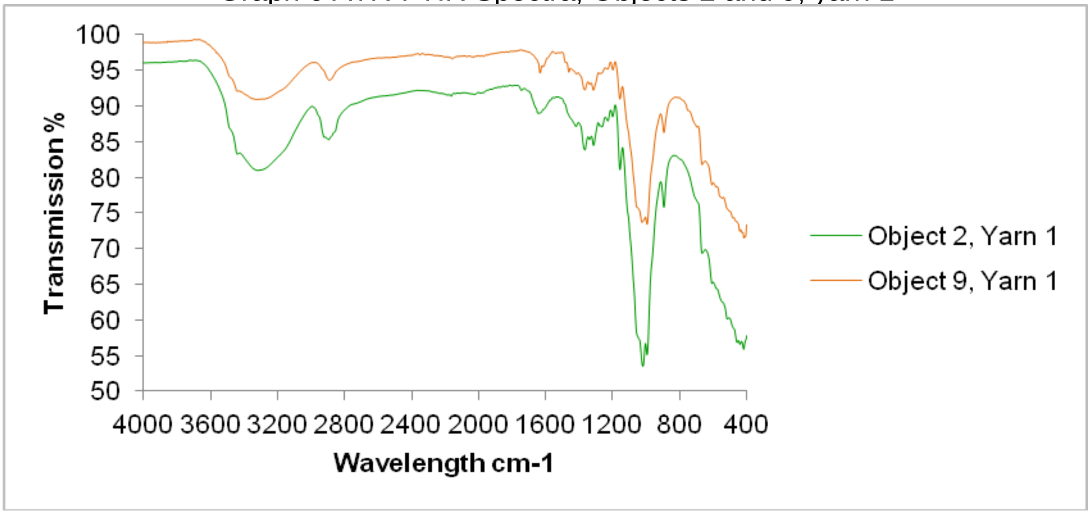
[122] Abidi, (2011), pp. 94-95.

The spectra of yarn 2, Object 4, identified it as a cellulosic fibre and using this in combination with microscopy, it was identified as viscose rayon. Graph 5 shows the spectrum for yarn 2, Object 4. Whilst this spectrum resembles that of the cotton fibre in Graph 4, it does not however have the same double dip around the OH groups. This was the same for all samples identified as viscose rayon (see Appendix 2) as well as for the other fibres identified as regenerated cellulosics from ATR-FTIR: Object 9 identified as lyocell fibre as per its garment label (see Graph 6), and, Object 2 as cupro from its smooth round microscopic appearance (see Graph 6). It is speculated that the reduction in definition present in the ATR-FTIR spectra of regenerated cellulosic fibres like viscose rayon, is due to the fewer number of hydrogen bonds present in comparison with natural cellulose fibres, as described in Chapter 3.[123] No specific reference to this was found in literature acquired during research.

Graph 5 ATR-FTIR Spectra, Object 4, yarn 2: viscose rayon



Graph 6 ATR-FTIR Spectra, Objects 2 and 9, yarn 1



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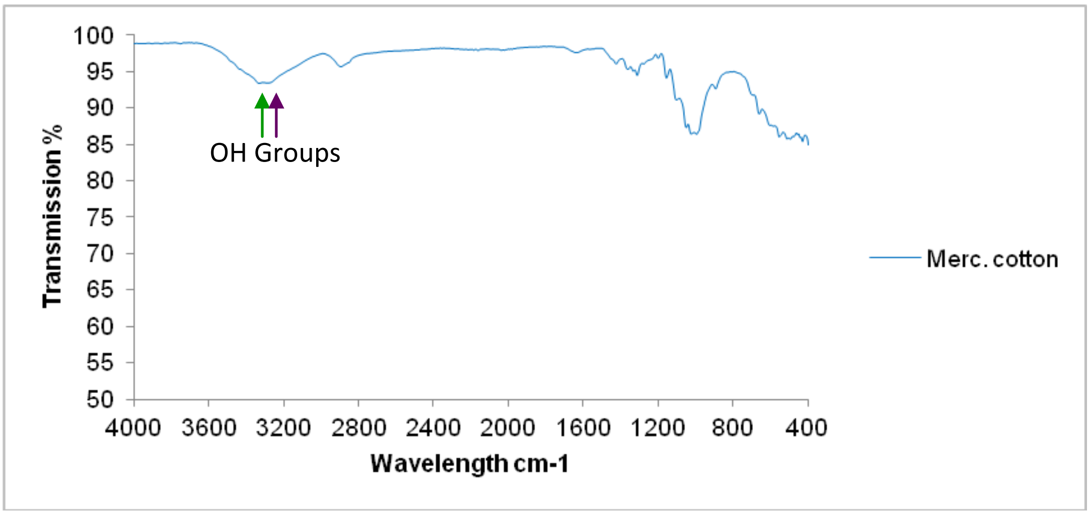
[123] Gohl and Vilensky, (1981), p. 58.

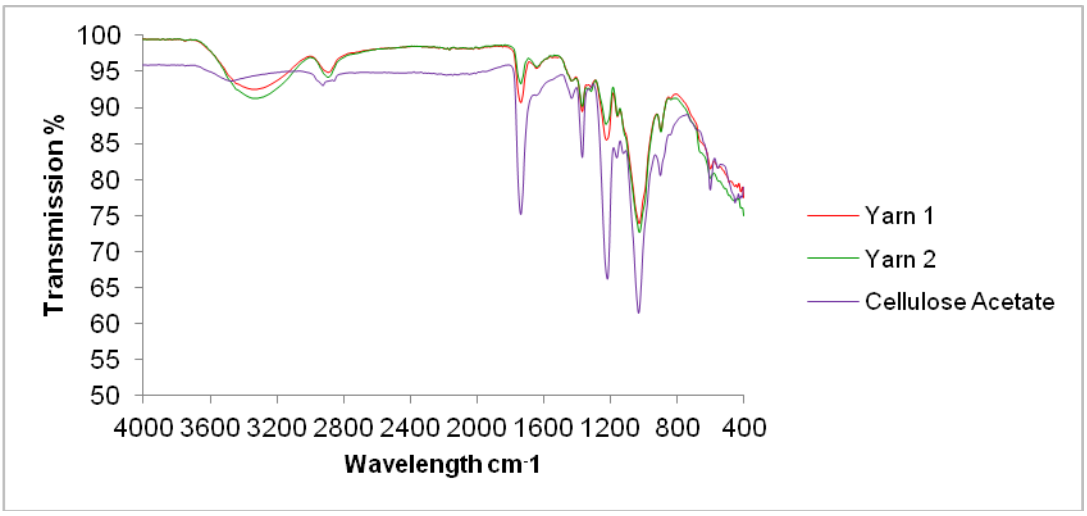
A reference material of mercerised cotton was analysed using ATR-FTIR, to show whether the same OH group pattern existed in its spectrum as for normal cotton, whose microscopic appearance is similar to regenerated cellulosic fibres (see fig. 39). This spectrum, shown in Graph 7, recorded the same double dip trough as for normal cotton, showing that it is possible to distinguish mercerised cotton from regenerated cellulosic fibres with ATR-FTIR Spectroscopy.

Graph 7 ATR-FTIR spectrum, mercerised cotton

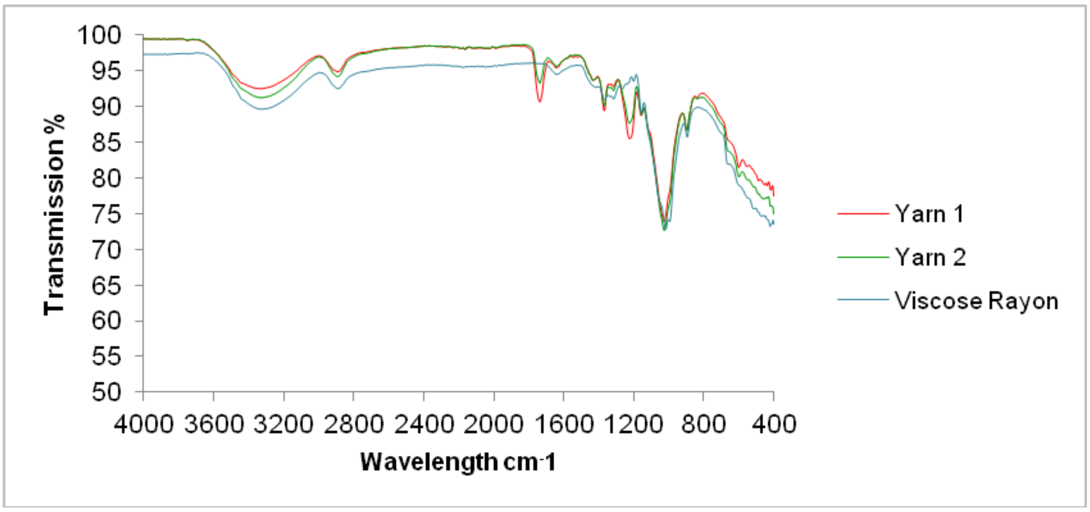


The spectra produced for Objects 10 and 11 both proved inconclusive with just a single fibre type being dominant in yarns of both directions. The labelling from the manufacture stated that Object 10 was a blend of viscose rayon and cellulose acetate but both yarn directions showed predominately as cellulose acetate, although the OH bonding area between 3000-3600cm^{-}1 was the same as the reference for viscose rayon, as shown in Graphs 8-9. Object 11 was labelled as a blend of viscose rayon and silk. Its spectrum however showed both yarn directions to be viscose rayon (see Graphs 10-11).

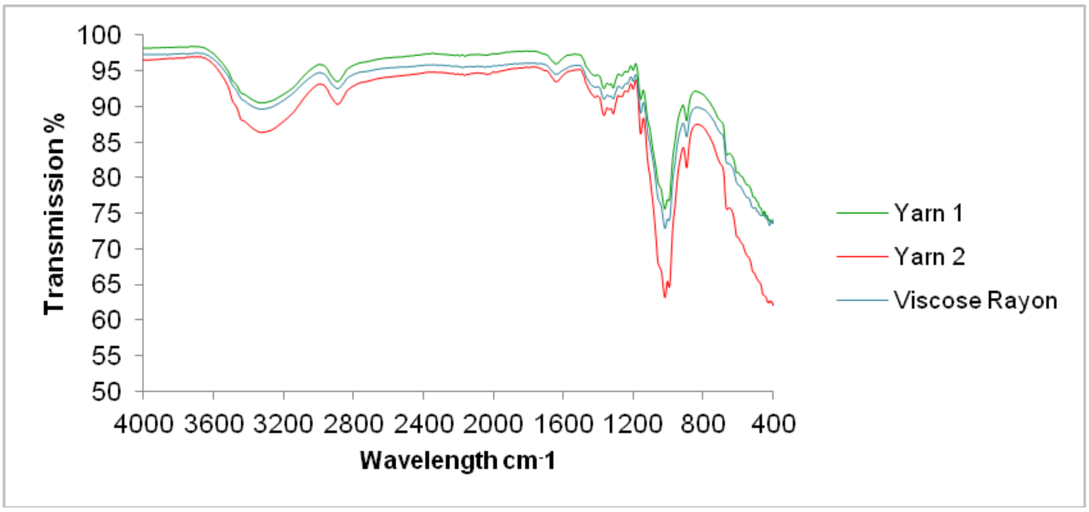
Graph 8 ATR-FTIR Spectra, Object 10, with reference of cellulose acetate



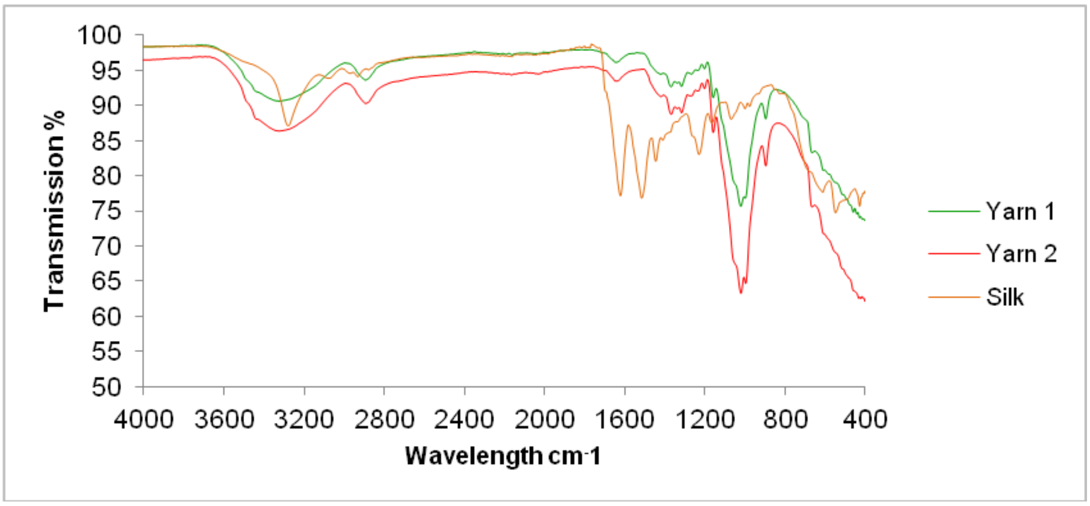
Graph 9 ATR-FTIR Spectra, Object 10, with reference of viscose rayon



Graph 10 ATR-FTIR Spectra, Object 11, with reference of viscose rayon

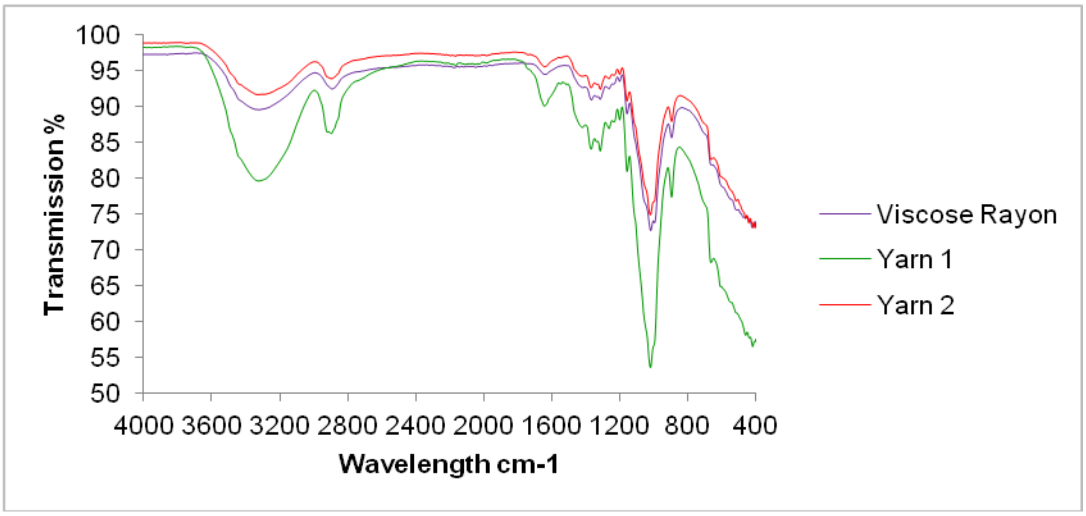


Graph 11 ATR-FTIR Spectra, Object 11, with reference of silk

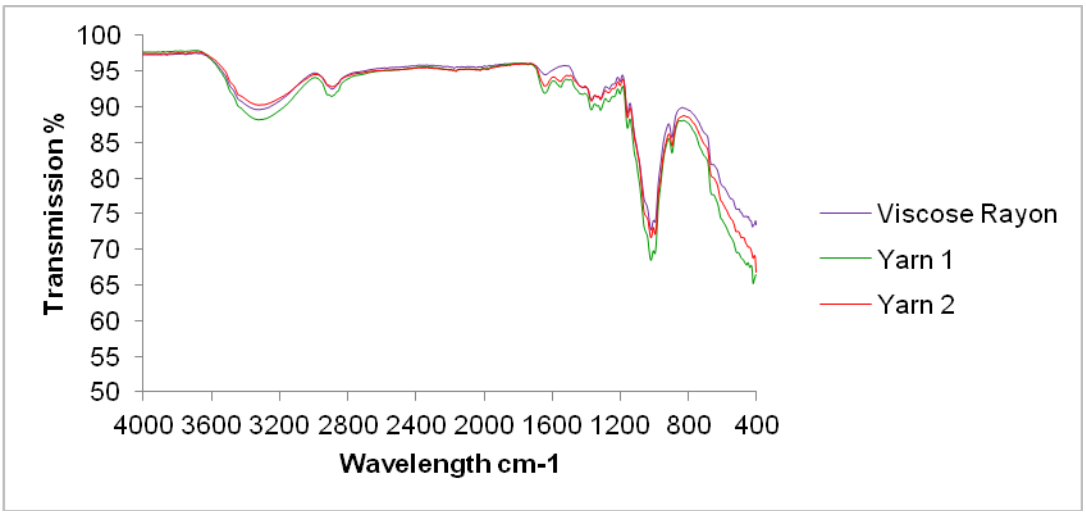


Final analysis using ATR-FTIR resulted in the selection of three objects identified as viscose rayon that were suitable for testing described in Chapter 5: Objects 3, 7 and 8. The spectra of these are shown in Graphs 12-13 below, compared against a reference spectrum of viscose rayon.

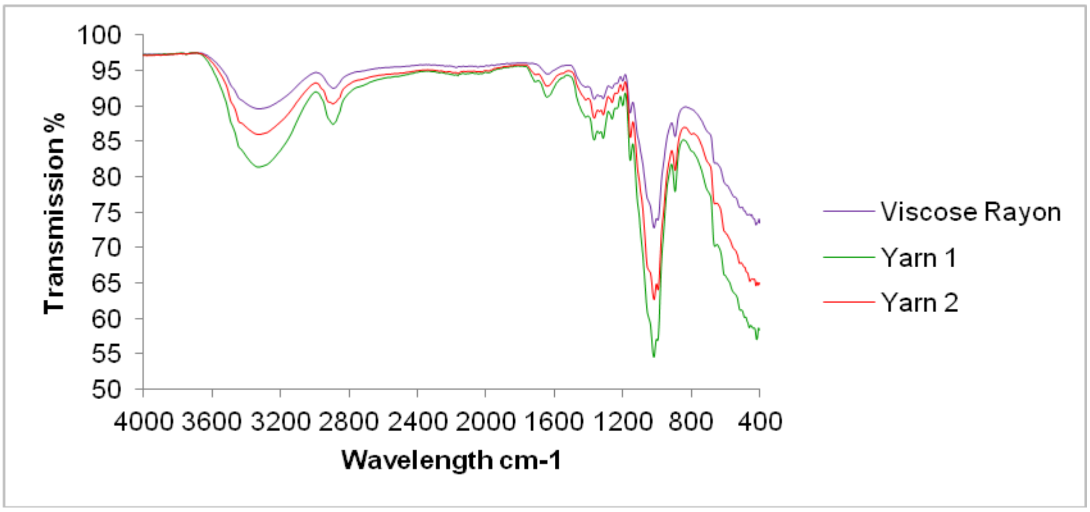
Graph 12 ATR-FTIR Spectra, Object 3



Graph 13 ATR-FTIR Spectra, Object 7



Graph 14 ATR-FTIR Spectra, Object 8



Discussion of Results

This discussion amalgamates observations and results from the different analyses, comparing similarities and ambiguities.

Early on, sensory perceptions indicated that object 1 was silk. Results from microscopy showed smooth round fibres typical of silk (and man-made fibres) but confirmation of the initial silk prognosis was clarified with ATR-FTIR.

During microscopy, a number of samples from different objects were observed to have striations. This included both yarns from Object 3, 6a, 6b and 8, and also, yarn 2 of Object

4, yarn 1 of Object 5, possible striations on yarn 2 of Object 7 and finally yarn 2 of Object 11 (see figs. 23-34). Most of these fibres were later confirmed as viscose rayon following ATR-FTIR spectroscopy with the exception of two; the yarns of 6b which following solubility testing were confirmed as cellulose acetate and, yarn 2 of Object 11 which was already known from manufacturing labelling as a viscose and silk blend.

During microscopy, it was observed that fibres of Object 5 yarn 2 and both yarns of Object 6b had unfocused edges (see figs. 27 and 29). These fibres were all identified as cellulose acetate following solubility testing. Lewarne et al. provide information that cellulose acetate has the same refractive index as liquid paraffin mountant causing loss of edge definition in microscopy.[124] This concurs with the observations above and could be useful for determining cellulose acetate from viscose rayon.

Object 2 was labelled ‘All Foreign Rayon’. Microscopy revealed an undefined smooth round fibre and ATR-FTIR showed a regenerated cellulose fibre. With these results, and the 1930s dating of the object eliminating modal and lyocell fibre types, it was determined that the fibre was cupro. Today in Britain and America, cupro would not be allowed to be labelled as rayon, see Chapter 2. The dress label, describing a textile manufactured to resemble silk, of Objects 6a and 6b, proved vague, with no distinction between the different materials of the lining (6b) and outer material (6b); analysis showed 6a was cellulose acetate and 6b viscose rayon. Fabric content labels on Object 8 ‘viscose’ and Object 9 ‘lyocell’, both manufactured since the introduction of compulsory labelling, proved to be accurate.

The blended fibres of Objects 10 and 11 proved difficult to identify. Both yarns of Object 10 were smooth and undefined and acetone solubility testing did not result in any visible solubilisation of fibres although, ATR-FTIR showed an apparent predominance of cellulose acetate. Yarn 1 of Object 11 was smooth and undefined whereas yarn 2 had striations. Again, ATR-FTIR only showed one predominant fibre: viscose rayon.

Specks observed on some yarns during microscopy made it difficult to discern distinguishing features (see earlier figs 23-34), as also described by Lewarne et al. earlier.

There were diversities in the macroscopic appearance of the fabrics identified as viscose rayon following analysis. Object 3 (1940s) had the high-sheen and smoothness associated

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[124] Lewarne, Karsten and Lemire, (2009), p. 303.

with early viscose rayon. Object 6a's surface was matte and coarse. The fabric of Object 7 (ricol) was akin to wool and without lustre. Object 8 (BHS) was soft and cotton-like without lustre. This demonstrates the versatility in appearance possible as a result of processing developments outlined in Chapter 2.

Conclusion

Fibre identification analysis has provided a catalogue of viscose rayon fibres as well as some other man-made fibre types, showing similarities and distinctions between them.

During ATR-FTIR Spectroscopy a distinction was made in the OH group recorded in the spectra between regenerated cellulosic and natural cellulosic fibres.

On the whole, ATR- FTIR proved a useful tool for verification of fibre types. However, this also supported the accuracy that can be obtained from the use of a combination of *basic* fibre identification techniques. Knowledge acquired during research which highlighted some subtle differences and similarities in the appearance of different man-made fibres greatly helped with fibre identification. This shows the value of such knowledge acquisition for accurate fibre identification by conservators.

Analysis has enabled three viscose rayon objects to be chosen for testing from different time periods. These objects will now be referenced by new letter classification during testing, as follows:

- Object 8 (1980/90s): Test Object A
- Object 7 (1960s): Test Object B
- Object 3 (1940s): Test Object C

The next chapter presents testing done on these objects.

Chapter 5 – Testing

Introduction

This chapter describes tensile strength tests carried out to determine the impact that a conservation wet cleaning treatment has for the strength of test specimens from three objects made of viscose rayon.

Each object is from a different period in viscose rayon's history, to highlight whether manufacturing changes mean that earlier versions of viscose rayon have poorer wet properties than later versions, and to what extent.

The rationale for testing is outlined first. This is followed with details of test object choice, test specimen preparation, test variables, test groups/rounds and the controlled wet cleaning treatment. The presentation and analysis of results from tensile strength tests are then given, followed by a summary of these results.

5.1 Rationale for Testing

Viscose rayon has always had problems with its wet properties. Industrial developments have progressively improved wet strength and wet dimensional stability. However, poor wet properties are still an issue for regular viscose rayon fibres produced today.

From a conservation perspective no research was found into the implications that wet cleaning treatments have for viscose rayon objects. It was decided that it would be useful to conduct tests which assessed the impact that a controlled conservation wet cleaning treatment had on the strength of test specimens of viscose rayon. As textile conservators deal with objects from the past, it was considered particularly worthwhile to test an early version of viscose rayon alongside later versions, in order to show whether wet cleaning is implicated in any significant differences in the wet properties of each.

Testing aimed to indicate whether viscose rayon from different time periods are suitable candidates for conservation wet cleaning treatments and, to highlight any implications that the poor wet properties of viscose rayon have for such treatments.

5.2 Test Object Choice

Appendix 3 contains research on the dating of each test object and a sample of each fabric.

The three test objects, as outlined at the end of Chapter 4, were chosen on the following basis; (1) they were identified during fibre analysis as viscose rayon, (2) they contained enough fabric to use for all testing, (3) the dating of each corresponded to a separate period associated with an advancement in viscose rayon processing which resulted in improved wet properties, (4) the fabric structure of each was as similar as possible given the *vintage* nature of the objects. The latter two bases are now described in more detail below.

Chapter 2 detailed improvements to viscose rayon's wet properties occurring in the 1940s, 1950s and 1970s.[125] Each object chosen corresponds with one of these dates: Object A c.1980/90s, Object B c.1960s and Object C c.1940s. This aims to highlight any significant contrasts between the wet properties of viscose rayon from different eras, caused by wet cleaning. It was hoped to obtain an earlier viscose rayon object, dated 1905-1920, as fibres produced in this period had reportedly the lowest wet strength of all, described in Chapter 2. Unfortunately no such examples were found.

It was recognised that each test object would have been subjected to varying degrees of natural ageing.[126] It was contemplated that test specimens from Objects A and B could be artificially aged to represent the level of ageing undergone by Object C. However, upon researching artificial ageing methods it was determined that accurately representing a specific time span and level of ageing could be difficult and this was therefore not followed through.[127] In order to proceed with testing, it was decided that the variable of ageing was an inherent necessity given the scope of this project, but that it would be considered when analysing test results.

Another inherent variable in using *vintage* objects was the lack of choice of weave type and density. With the resources available for this project, it was not possible to acquire test objects of the same weave. The three test objects chosen therefore have different weaves:

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[125] Hatch, (1993), p. 186.

[126] Natural ageing is caused by different agents of deterioration including; light, relative humidity, temperature and environment. See, Sarah Staniforth, 'Chapter 5: Agents of Deterioration', in *The National Trust Manual of Housekeeping*, (Oxford: Elsevier, 2006), pp. 45-53.

[127] Robert L. Feller, 'Chapter 4 Prediction of Useful Lifetime', in *Accelerated Aging: Photochemical and Thermal Aspects*, (USA: The Getty Institute, 1994), pp. 37-43.

Object A plain weave, Object B twill weave and Object C crepe weave. Due to the difference in weaves, it was decided that it would not be possible to make direct strength comparisons, as a twill weave for example may be slightly stronger than a plain weave. Instead the *tenacity*, or *specific stress*, for each sample has been determined. *Tenacity,* a measurement of equivalent strength between specimens of different *linear density,*[128] is used in the textile industry for comparing different fabric strengths.[129]

It was hoped to find white or un-dyed fabrics for testing to reduce the possibility that different colourants may have affected fibres. However, again due to limited resources this was not possible. The test objects chosen each have printed designs on them and obviously the effect that printing colourants may have had on fibres will need to be considered when analysing results.

5.3 Test Specimen Preparation

BS 13934-1:1999[130] was used where appropriate to control specimen preparation. All test specimens were identically prepared.

Before cutting specimens, test fabric was ironed on a low setting to remove any creasing which could have caused uneven stress on specimens, affecting test results.

Specimens were cut into rectangular fabric strips 100mm by 25mm, with the edges cut parallel to the line of the weave. In accordance with BS 13934-1:1999, test specimens were prepared by cutting them slightly larger than the required size and then pulling threads away from the edges until the correct measurement was achieved, leaving a fringe smaller than 5mm. The actual test area was 80mm by 25mm, as the space at the top and bottom of specimens was used for clamping them into the tensile strength tester.[131]

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[128] *Linear density* is the mass (Kg) / Unit Length (m) of a specimen. See, W. E. Morton and J. W. S. Hearle, ‘Chapter 13: Tensile Properties’, in *Physical Properties of Textile Fibres: Student Edition,* (Manchester: The Textile Institute, 1986), p. 267.

[129] Morton and Hearle, (1986), pp. 267-269.

[130] British Standard, *Textiles – Tensile Properties of Fabrics, Part 1: Determination of Maximum Force and Elongation at Maximum Force Using the Strip Method BS EN ISO 13934-1:1999*, (London: British Standard Institute, 1999), p. 6.

[131] It was aimed to have a test area of 100/25 mm, half the recommended size in BS 13934-1:1999. However, allowing excess fabric for clamping specimens in the Instron device was overlooked and, due to a limited supply of test fabric, the test area had to be reduced.

For each test round, ten specimens were tested, five cut with the length in the warp direction, five with the length in the weft direction (see figs. 41-42) in accordance with BS 13934-1:1999.[132] Both warp and weft specimen sets were tested separately, as the warp tends to be stronger than the weft which can affect test results. Test specimens cut in the warp direction are referred to as *warp specimens* henceforth and those cut in the weft direction, as *weft specimens*.

Fig.41 Test specimen cut in the warp direction

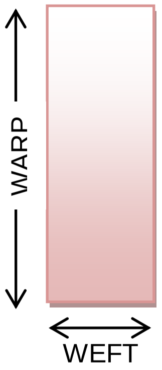
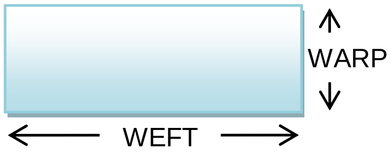
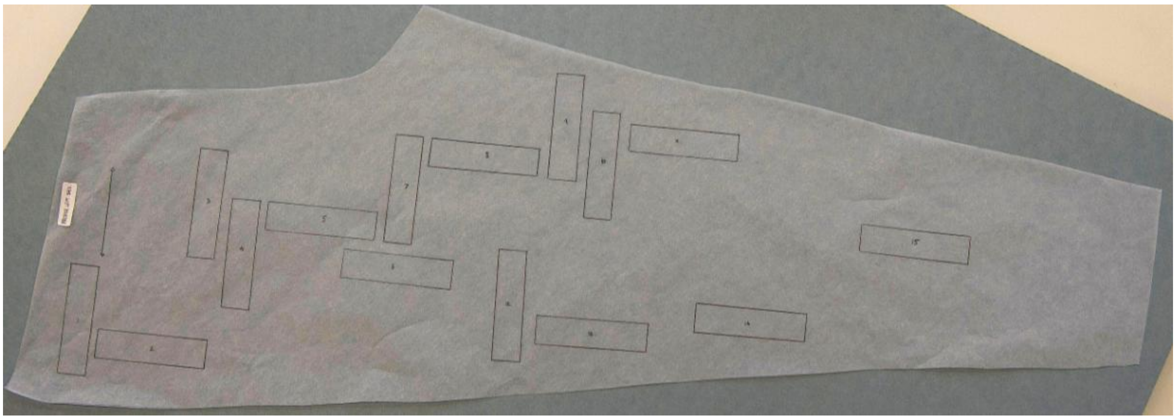


Fig.42 Test specimen cut in the weft direction



BS 13934-1:1999 suggests test specimens be cut with no two specimens containing the same line of warps and wefts as another,[133] to reduce the possibility of weave discrepancies in a fabric affecting results. However, due to the limited amount of fabric available in test objects, it was necessary to adapt that method for this project. A cutting plan was devised which, rather than having test specimens that contained *none* of the same warps and wefts, instead involved no two test specimens containing *exactly the same* line of warp and weft threads (see fig. 43).

Fig. 43 Test specimen cutting plan for Test Object A



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[132] British Standard, (1999), p. 5.

[133] British Standard, (1999), p. 5.

5.4 Summary of Test Variables

Listed below are the variables for testing. Given first are the variables present within the test objects, followed by the variables set for testing.

Test Object Variables:

- Date of production
- Weave structure
- Colourants (print)

Test Variables:

- Dry untreated specimens
- Wet treated specimens
    - Three wash solutions used for treatment:
        - Non-ionic detergent
        - Anionic detergent
        - Soft water
- Dry treated specimens
    - Three wash solutions used for treatment:
        - Non-ionic detergent
        - Anionic detergent
        - Soft water

5.5 Test Groups and Rounds

The test groups and rounds devised are now outlined in Table 6 overleaf. Individual test rounds are ordered in three main test groups; Test Group 1 dry untreated specimens, Test Group 2 wet treated specimens and Test Group 3 dry treated specimens. A total of 21 test rounds were conducted, highlighted in bold. *Treated* specimens are those which have undergone a controlled wet cleaning treatment, as described under the next heading.

Table 6 Test structure for tensile strength testing

Test Group	State of Specimens During Testing	Test Subgroup (Treatment)		Test Object	**Test Round**	No. Warp Specimens Tested	No. Weft Specimens Tested
1	Dry	None		A	**1-A**	5	5
				B	**1-B**	5	5
				C	**1-C**	5	5
2	Wet	Wet cleaning treatment	1 Non-ionic detergent	A	**2-1-A**	5	5
				B	**2-1-B**	5	5
				C	**2-1-C**	5	5
			2 Anionic detergent	A	**2-2-A**	5	5
				B	**2-2-B**	5	5
				C	**2-2-C**	5	5
			3 Soft water	A	**2-3-A**	5	5
				B	**2-3-B**	5	5
				C	**2-3-C**	5	5
3	Dry	Wet cleaning treatment, then allowed to air dry for 72 hours	1 Non-ionic detergent	A	**3-1-A**	5	5
				B	**3-1-B**	5	5
				C	**3-1-C**	5	5
			2 Anionic detergent	A	**3-2-A**	5	5
				B	**3-2-B**	5	5
				C	**3-2-C**	5	5
			3 Soft water	A	**3-3-A**	5	5
				B	**3-3-B**	5	5
				C	**3-3-C**	5	5

5.6 Test Group 2 and 3: Conservation Wet Cleaning Method

All testing for specimens from Test Group 2 and 3 was preceded by a controlled wet cleaning treatment, to replicate the stress and strain placed on objects during such treatments in conservation. It was felt that this would most meaningfully assess the impact for conservation of any reduction in strength as a result of wetting.

The controlled wet cleaning treatment was based on normal wash treatments carried out previously on textiles at the *CTCTAH.*[134] This involved the use of a wash solution combined with gentle sponging followed by soft water rinses to flush detergent away from the object and finalised with a de-ionised water bath.

The wash bath used was a small plastic tray lined with a layer of Melinex® (see figs. 44-45). Test specimens were placed in the wash bath and the wash solution (described below) gently poured in through hands, to soften the flow. To ensure all test specimens underwent the same extent of treatment, a standardised treatment cycle was devised, shown in Table 7. The deionised water used for final baths came from an *ELGA B114 Deioniser* with cartridge type *C114*.

Table 7 Wet cleaning treatment cycle

Wet Cleaning Stage	**Cycle**	**Time (minutes)**
Wash Solution added	Front: gently sponged	4
	Left	6
	Front: gently sponged	4
	Left	6
	Front: gently sponged	4
	Left	6
	Back: gently sponged	4
	Left	6
	Back: gently sponged	4
	Left	6
	Back: gently sponged	4
Soft water rinses	6 bath rinses	16
De-ionised final rinse	1 bath	10
	Total wet cleaning time:	**80**

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[134] CTCTAH (Centre for Textile Conservation and Technical Art History), University of Glasgow

During wet cleaning a few threads loosened from the edge of some test specimens. Lost threads did not notably reduce the size of affected specimens and it was felt that these instances were unavoidable due to the gentle sponging required to replicate normal wet cleaning treatment conditions.

Following wet cleaning treatment, wet specimens from Test Group 2 were transferred to the tensile strength testing area sandwiched in between two layers of Melinex® to inhibit water evaporation. Each single specimen was released from the Melinex® sandwich only when ready for testing, to prevent any differential evaporation of water from specimens which may have affected results. Treated specimens from Test Group 3 were placed on blue paper towel and allowed to air dry for 72 hours[135] before tensile strength testing.

5.6.a Wash Solution Choice

The cellulosic nature of viscose rayon means that an anionic detergent may be most suited for use with it, see Chapter 1. However, in the absence of available research into detergents suited for use with viscose rayon, it was decided to test both the anionic detergent Orvus WA® and the non-ionic detergent Dehypon LS45® separately, to determine whether either caused a greater reduction in strength to test specimens. Orvus WA® and Dehypon LS45® were chosen as the only detergents available at the *CTCTAH* at the time of testing. Also, they are both commonly used in textile conservation.

For consistency, both detergents were used at a concentration of five times their critical micelle concentration (cmc) in soft water from the tap.[136] The anti-soil redeposition agent sodium carboxylmethyl cellulose was added to each detergent wash solution, as is common in conservation wet cleaning. Detergent wash solution calculations are given in Appendix 4. One batch of each detergent wash solution was made for use in all wet cleaning treatments. As a control, a third wash solution of soft water only was also tested. All wash solutions were used at room temperature.

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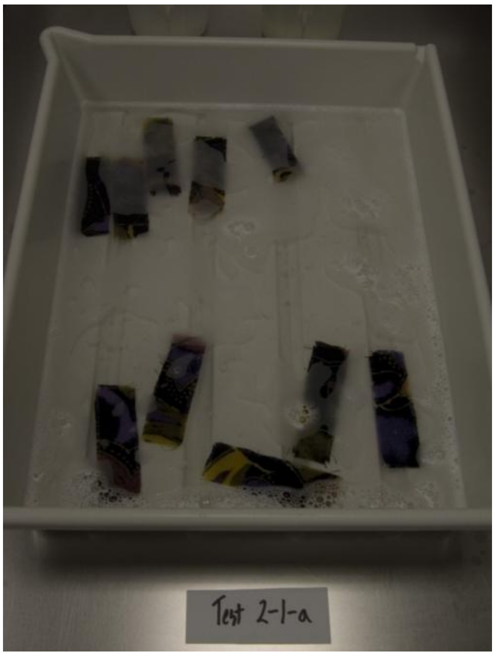
[135] The time of 72 hours was partly arbitrary as testing was started on a Friday and access to the *CTCATH* was not available over the weekend.

[136] Sourced from soft water geographical region (Glasgow).

Fig. 44 Conducting wet cleaning treatment on test specimens

Fig. 45 Test 2-1-A specimens in the wash bath



5.7 Tensile Strength Testing

Tensile strength testing was conducted using an *Instron 5544 Tensile Strength Tester* and *Bluehill* software version 1.4 (see fig. 46). This equipment measures strength by applying an even force to specimens held between two jaws and measuring the force required to break them. The Instron device was set to an extension speed of 10mm per minute, with a load cell of 1000 Newtons. Test specimens were mounted in the device clamps with pre-tension. All testing was carried out in an uncontrolled room environment.[137]

In addition to testing wet treated specimens (Test Group 2), specimens that had been wet cleaned and then air dried were also tested separately (Test Group 3) to show any immediate permanent effects on strength.
As mentioned earlier, Test Group 2 specimens were immediately taken following wet cleaning to be tensile strength tested. Before testing, each wet specimen was briefly placed onto a single sheet of blue paper towel to remove excess water, as suggested by British Standard (BS) 13934-1:1999,[138] and tested immediately.

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[137] See Appendix 5 for room conditions for each test round.

[138] British Standard, (1999), p. 7.

As mentioned in Chapter 3, observations of dimensional change in wet specimens were also taken as reference for future research and these are included in Appendix 6.

Analysis of results is presented in two sections; (1) comparing dry untreated specimens against wet treated specimens, (2) comparing dry untreated specimens against dry treated specimens.

Fig. 46 Mounting Object C test specimen in the Instron device

5.7.a Tensile Strength Test Observations

Described now are any prominent observations on how test specimens reacted during tensile strength testing. An analysis of the results will follow under the next heading.

It should be noted that specimens from Test Group 2 did not show any visible signs of weakening as a result of the wet cleaning treatment, before tensile strength testing.

As said, the warp tends to be stronger than weft and there was a unanimous distinction in strength between test specimens cut in the different yarn directions from all test objects. This enabled the identification of warp and weft in Test Objects A and C, where no selvedge was present. It was noted during tensile strength testing that all weft specimens fractured at a lower load than their warp partners but, weft specimens elongated more before breaking than warp specimens.

Test Group 1: Dry untreated specimens

During tensile strength testing of specimens from Test Group 1, the first sign of fracture came from audible tearing, soon after followed with a broad fracturing of fibres across the surface of the test specimen. Despite fracturing, when test specimens were removed from the Instron device they were still intact (see figs. 47-49).

Fig. 47 Warp Specimen Test 1-A

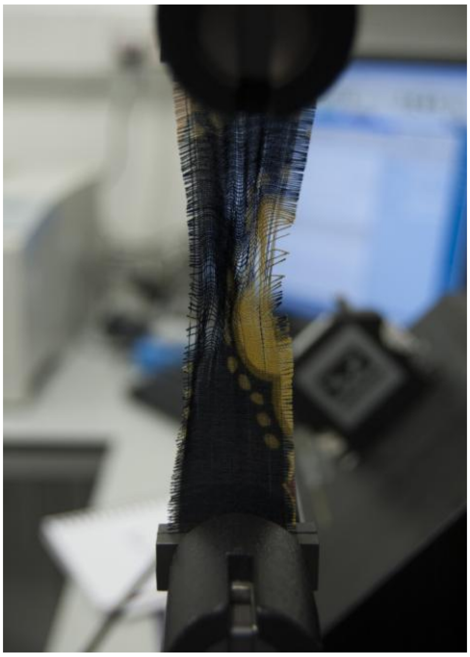


Fig. 48 Weft Specimen Test 1-B

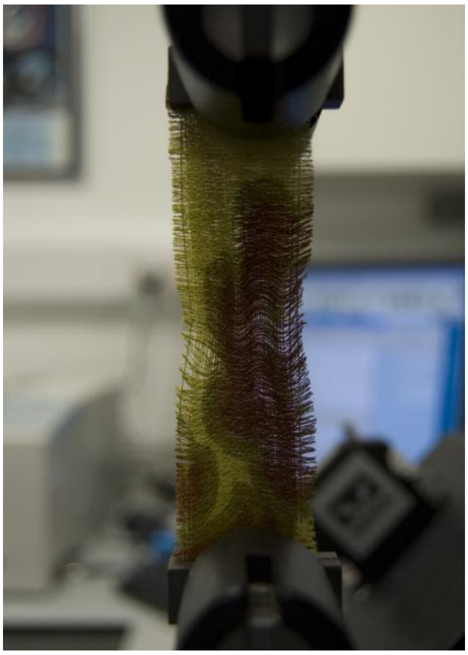
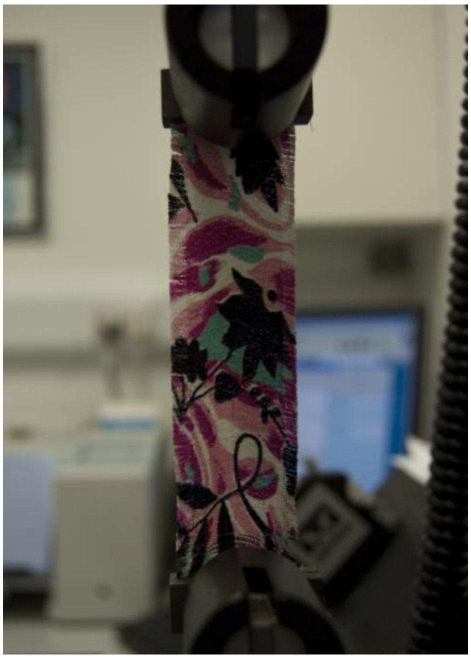


Fig. 49 Weft Specimen Test 1-C



Test Group 2: Wet treated specimens

During tensile strength testing of wet specimens from Test Group 2, breakage happened quickly with audible tearing and fracture occurring simultaneously. Fracturing was more destructive on wet specimens, compared with dry specimens in Test Group 1, with yarns across the entire width of most specimens fracturing in a line all at once so that these specimens were split in two (see figs. 50-52).

Fig. 50 Warp Specimen Test 2-1-A

Fig. 51 Warp Specimen Test 2-2-B

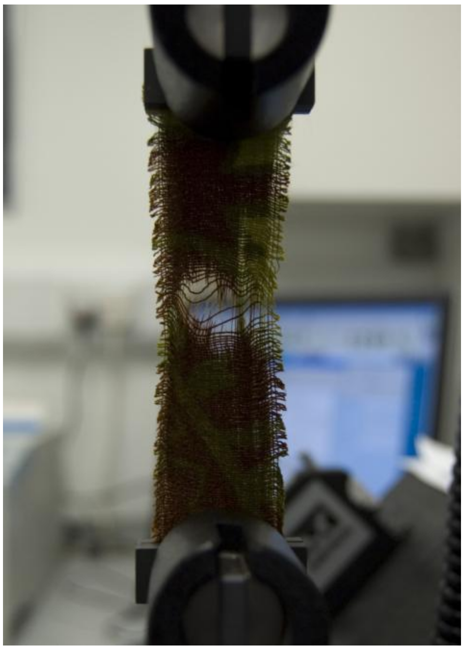
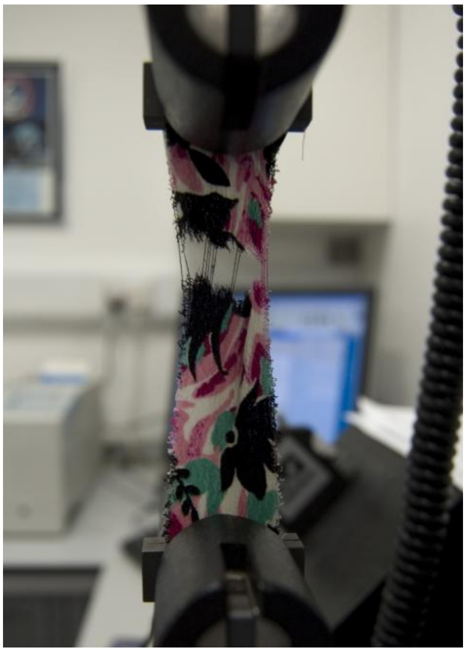


Fig. 52 Warp Specimen Test 2-3-C



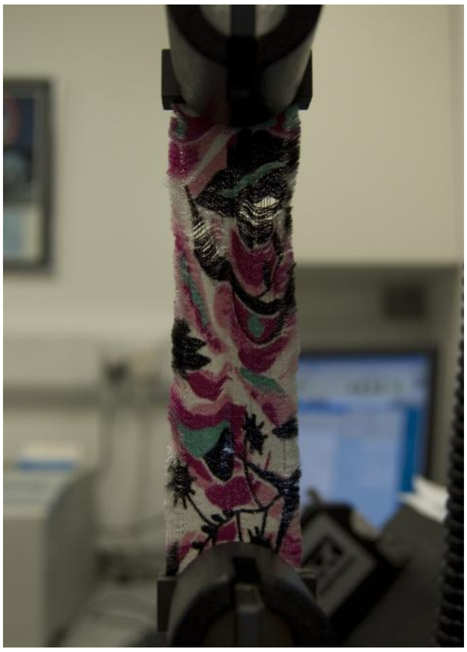
Test Group 3: dry treated specimens

Test specimens fractured in the same way as for Test Group 1 described above, with specimens still intact following testing (see figs. 53-55).

Fig. 53 Weft Specimen Test 3-2-A

Fig. 54 Weft Specimen Test 3-3-B

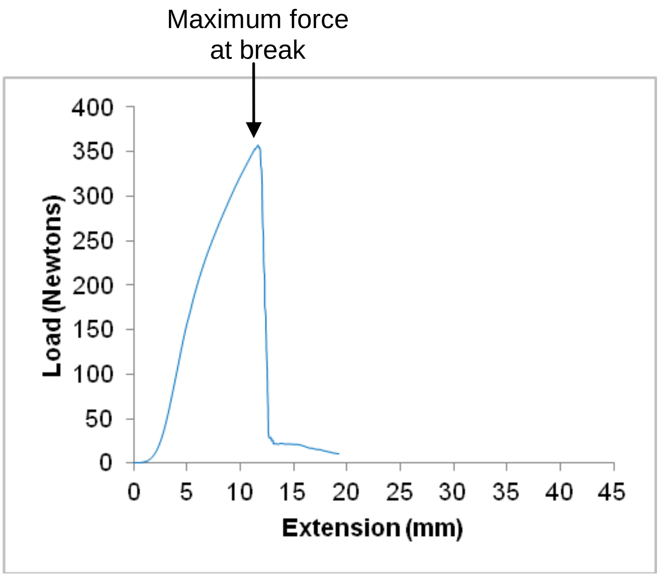
Fig. 55 Weft Specimen Test 3-1-C



5.7.b Tensile Strength Test Results Analysis

In tensile strength testing, strength is determined by a material's maximum force (load) applied at break, measured in Newtons (N). This is represented as the peak in the load-extension curve in Graph 15 below. The initial curve upwards before the peak represents the gradual increase in force being applied to a specimen and its resistance to this. The curve downwards after the peak represents the force required to fracture any yarns remaining intact following the initial fracture, until all yarns have failed. If all yarns fail at once, then a steep drop occurs immediately following the peak, as shown in Graph 15.

Graph 15 Load-extension curve, warp specimen 2, Test 1-B, showing maximum force at break



As explained, analysis of testing has been done by calculating the *tenacity* of each specimen from the different test rounds. The calculation used for determining tenacity is as follows:[139]

$$\frac{\text{Load (N)}}{(\text{Mass in Kg / Unit length in metres})} \times 10^{-6} = \textit{tenacity} \text{ in N/tex}$$

Percentage changes of mean *tenacity* for each test round were then calculated using the formula below, to show the relative drop in percentage strength for each different object.

$$\frac{(\text{Original } \textit{tenacity} \text{ mean} - \text{New } \textit{tenacity} \text{ mean})}{\text{Original } \textit{tenacity} \text{ mean}} \times 100 = \% \text{ Change in } \textit{tenacity}$$

---

[139] Calculation from: Morton and Hearle, (1986), p. 267.

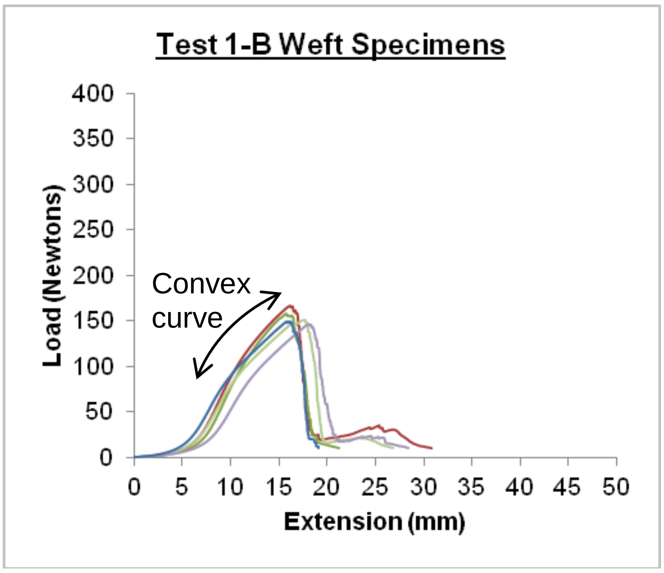
The first section below analyses the wet strength of test specimens from each object following wet cleaning, by comparing test round results from Test Group 1 (dry untreated) and 2 (wet treated). The next section analyses any permanent change in strength in specimens as a result of wet cleaning, by comparing test round results from Test Group 1 (dry untreated) and 3 (dry treated).

Section One: Test Group 1 versus Test Group 2

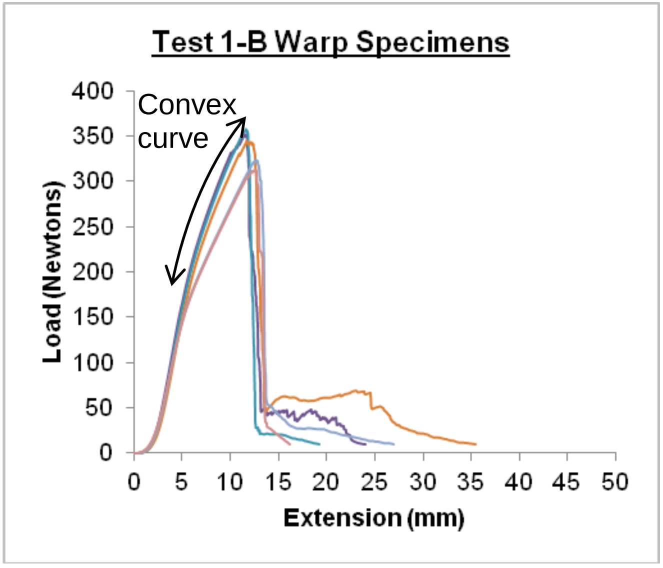
Firstly, load-extension curves from some test rounds are given to illustrate specific behavioural patterns of specimens during different test rounds. (Load-extension curves of all test rounds from Test Groups 1-3 are provided for reference in Appendix 7.) Next follows the main comparison of results from tensile strength testing. The mean results for warp and weft specimens from Test Group 1 and 2 have firstly been amalgamated into two bar graphs for ease of comparison between dry and wet strength. Secondly, two tables show the changes in *tenacity* for warp and weft specimens.

Graphs 16-17 show load-extension curves from dry specimens in Test 1-B. The initial curve before the peak is convex to the extension axis, showing some level of resistance from test specimens to the increased load. Graphs 18-19 show load-extension curves from wet specimens in Test 2-1-B. Here, the initial curve before the peak is concave to the extension axis, showing little resistance to the increased load in wet fibres, which ultimately resulted in the wet specimens breaking at a lower load than the dry specimens in Graphs 16-17. The steep drop following the peak in Graph 19 shows that almost all the specimens fractured immediately and this was typical for most wet test specimens although, as Graph 18 shows, sometimes a few yarns in wet specimens took some more force to fracture. This indicates that, if a viscose rayon object becomes sufficiently fragile during a conservation wet cleaning treatment, splitting may occur quickly at the point of weakness.

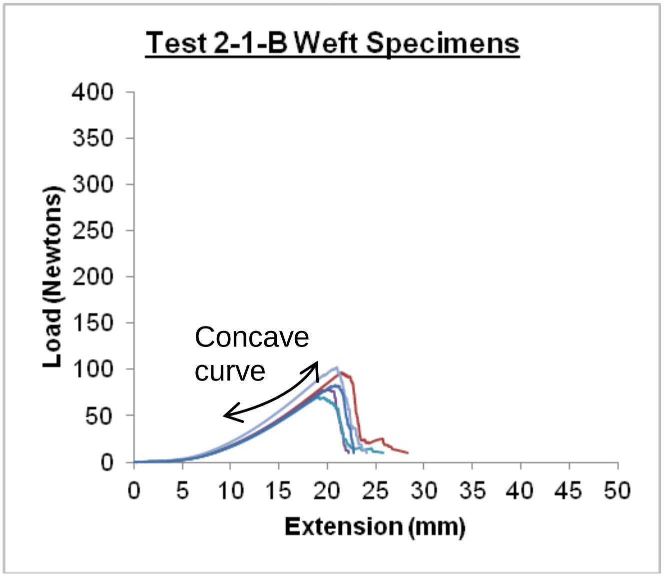
Graph 16 Load-extension graph, weft specimen, Test 1-B



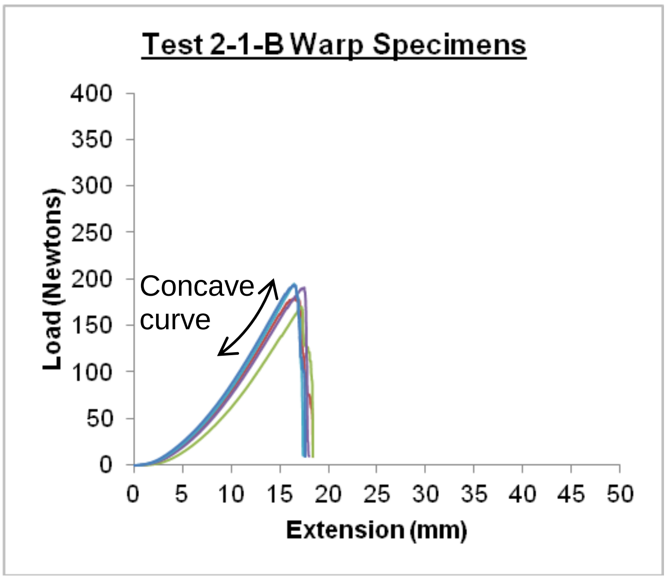
Graph 17 Load-extension graph, warp specimen, Test 1-B



Graph 18 Load-extension graph, weft specimen, Test 2-1-B

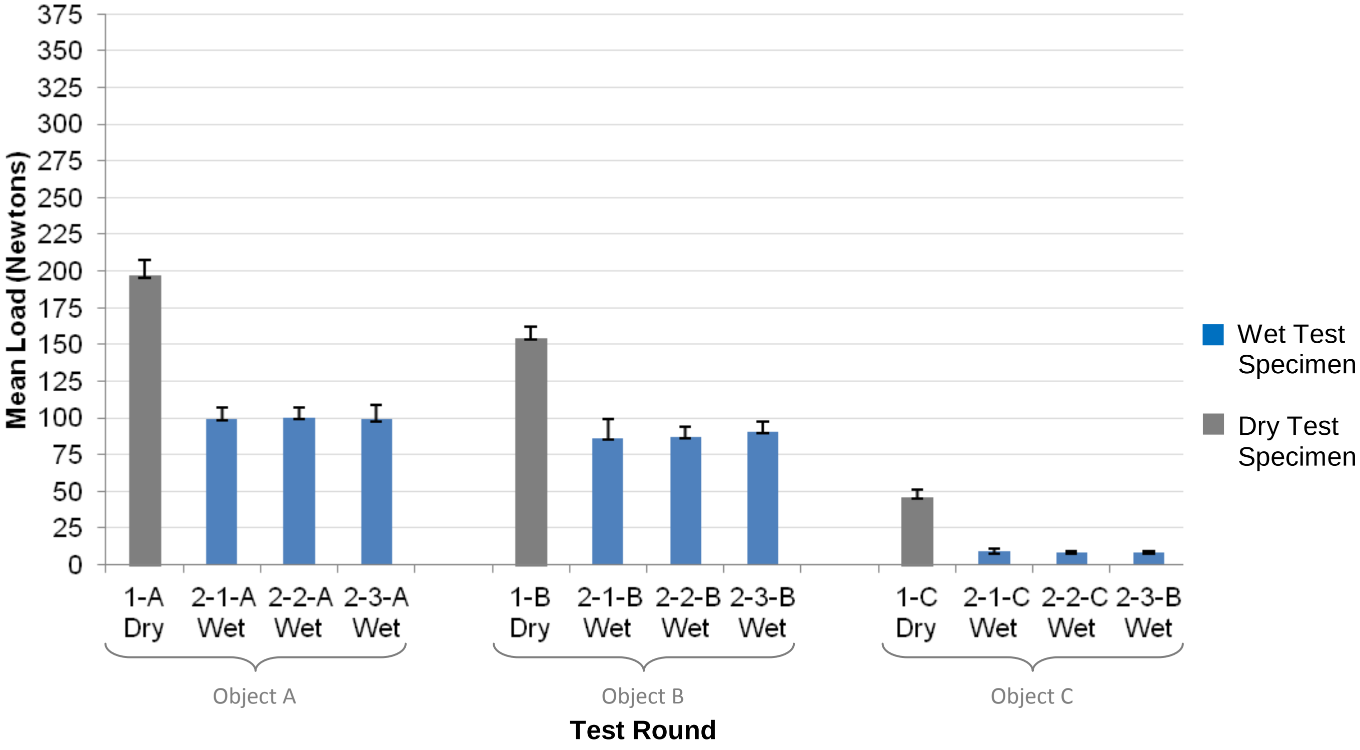


Graph 19 Load-extension graph, warp specimen, Test 2-1-B

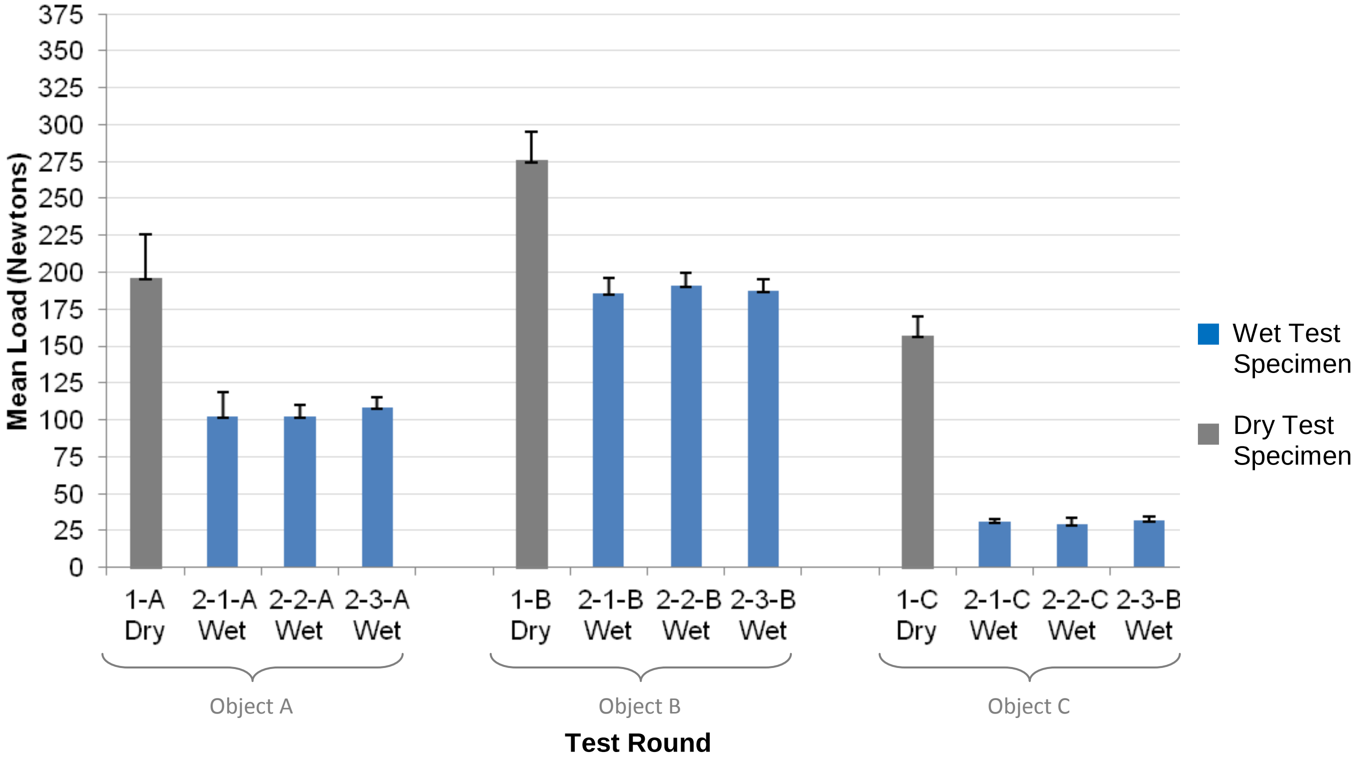


Graphs 20-21 below compare the mean wet and dry strength of specimens from Test Group 1 and 2. Both graphs show a marked drop in strength in wet specimens from all objects. Object C shows the greatest drop in strength from dry to wet specimens. These results however, do not take into account *linear density.* Further interpretation, in the form of *tenacity* figures, is therefore provided on the next page.

Graph 20 Test Group 1 versus Test Group 2 (Dry Untreated versus Wet Treated) Weft Specimens



Graph 21 Test Group 1 versus Test Group 2 (Dry Untreated versus Wet Treated)
Warp Specimens



Tables 8-9 below, show changes in *tenacity* from dry to wet specimens in the different test rounds. Object C showed a significantly greater decline in *tenacity* in specimens tested during Test Group 2, than that observed in Object's A and B (see Tables 8-9). For Object C, weft specimens lost between 75.6 - 78% in strength, warp specimens between 79.3 - 81%. Objects A and B had comparable loss in strength. For Object A, weft specimens lost between 48.9-51.4%, warp specimens between 41.8-47%. For Object B, weft specimens lost between 40-40.7%, warp specimens between 45.7-46%.

There was a marked difference in the original dry strength of untreated specimens between each object. The weft of Object C was 85.5% weaker in the dry state than Object A and, the weft of Object B was £52.1% weaker in the dry state than Object A. The warp of Object C was only 43% weaker than the warp of Object A but, the warp of Object B was 19.8% stronger than the warp of Object A. This shows that the weave structure of Object B affects weft and warp differently to that of Objects A and C. This also shows a marked difference in the dry strength of Object C compared with Objects A and B.

None of the three wash solutions – non-ionic detergent, anionic detergent and soft water – used for wet cleaning specimens in Test Group 2 resulted in significantly more loss of strength than another and it was determined neither had a significant impact on strength. The relative percentage differences in warp and weft strength between the different rounds in Test Group 2 being less than 5.3% (see Tables 8-9).

Statistical analysis, using the *Student's t-test*,[140] was done to show whether differences between tensile strength test load-extension graphs for dry and wet specimens were real and significant. These determined that there was at least a 1:100 probability of replication of results. *Student's t-test* calculations are provided in Appendix 8 for reference.

It was noted that wet specimens from Object C consistently fractured initially in areas of black print (see fig. 56) during tensile strength testing, although, the same observation was not made for dry specimens in Test Group 1 from Object C. It is likely that the black colourant has caused degradation in these areas weakening fibres, and, wetting the fibres has caused further weakening here resulting in a great reduction in strength, as seen by percentage changes in *tenacity* (see Table 8-9). No visible weakness was seen on wet specimens from Object C in black areas before testing. It is conceivable that, because Object C is dated c.1940s when wet strength was lower than later versions of viscose rayon, this degradation has had more of an impact on the fibres than it might have done in viscose rayon fibres produced later.

Table 8 Tensile strength from Test Group 1 and 2 (Dry Untreated and Wet Treated) Weft Specimens

Test Round	**Object A**	**Object B**	**Object C**
Dry tenacity - Test 1 (N/tex)	0.0564	0.027	0.0082
Wet tenacity - Test 2-1 (N/tex)	0.0288	0.016	0.0018
Wet tenacity - Test 2-2 (N/tex)	0.0282	0.016	0.002
Wet tenacity - Test 2-3 (N/tex)	0.0274	0.0162	0.002
Percentage drop in strength	51.4 - 48.9%	40.7 - 40%	78 - 75.6%

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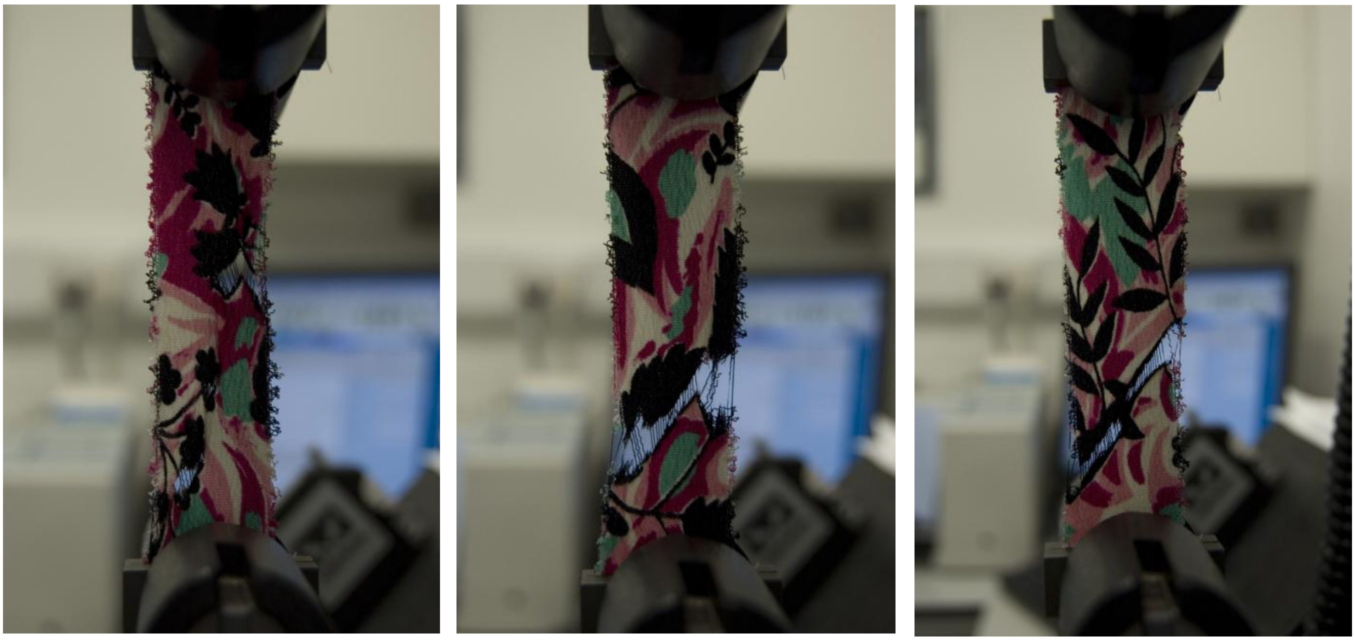
[140] The *Student's t-test* calculates the significance of results and shows the probability that future tests would replicate the same contrast between the dry and wet strength.

Table 9 Tensile strength from Test Group 1 and 2 (Dry Untreated and Wet Treated) Warp Specimens

Test Round	**Object A**	**Object B**	**Object C**
Dry tenacity - Test 1 (N/tex)	0.0526	0.063	0.03
Wet tenacity - Test 2-1 (N/tex)	0.028	0.0338	0.0062
Wet tenacity - Test 2-2 (N/tex)	0.0278	0.0342	0.0056
Wet tenacity - Test 2-3 (N/tex)	0.0306	0.0336	0.0062
Percentage drop in strength	41.8 - 47.1%	45.7 - 46.7%	79.3 - 81.3%

Fig. 56 Fracturing in areas of black dye on three specimens from Object C, Test 2-1-C

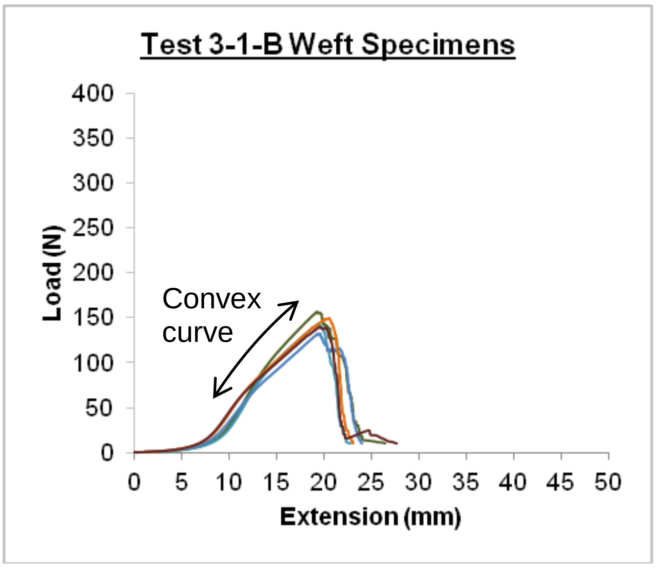


Section Two: Test Group 1 versus Test Group 3

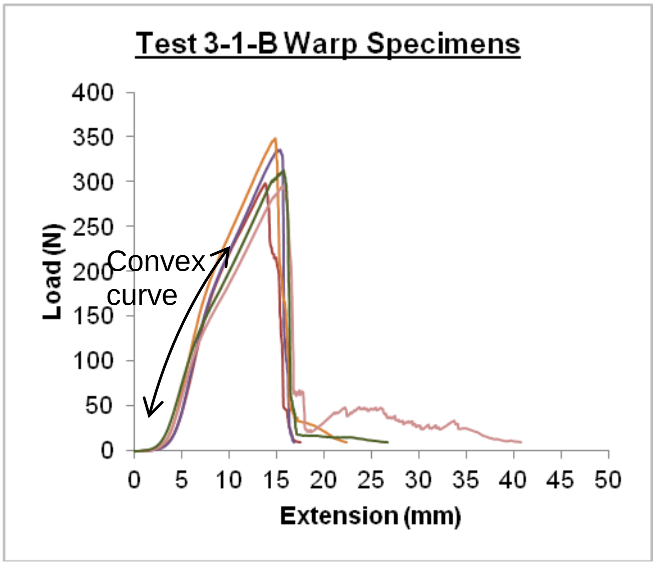
The results analysed below, comparing tensile strength test results from Test Group 3 against those from Test Group 1, are presented in the same manner and order as for *Section One* above.

The load-extension curves of specimens from Test Group 3 were similar in nature to those of Test Group 1. Graphs 22-23 show load-extension curves convex to the extension axis and this was the case for test specimens from both test groups, indicating some resistance from fibres to the increased load. This suggests that the dramatic loss in strength on specimens in the wet state following wet cleaning (see results from Test Group 1 and 2 above) was to some degree reversed upon air drying.

Graph 22 load-extension graph, weft specimens, Test 3-1-B

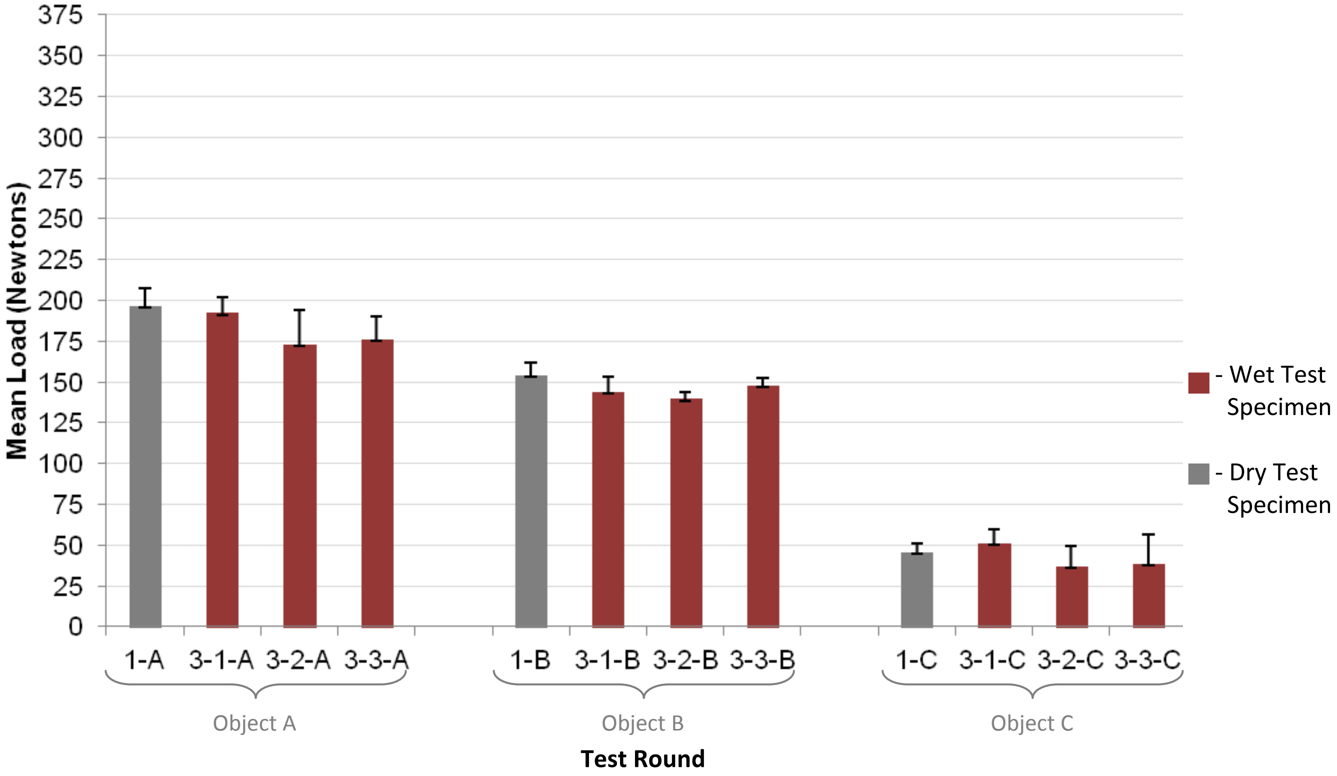


Graph 23 load-extension graph, warp specimens, Test 3-1-B

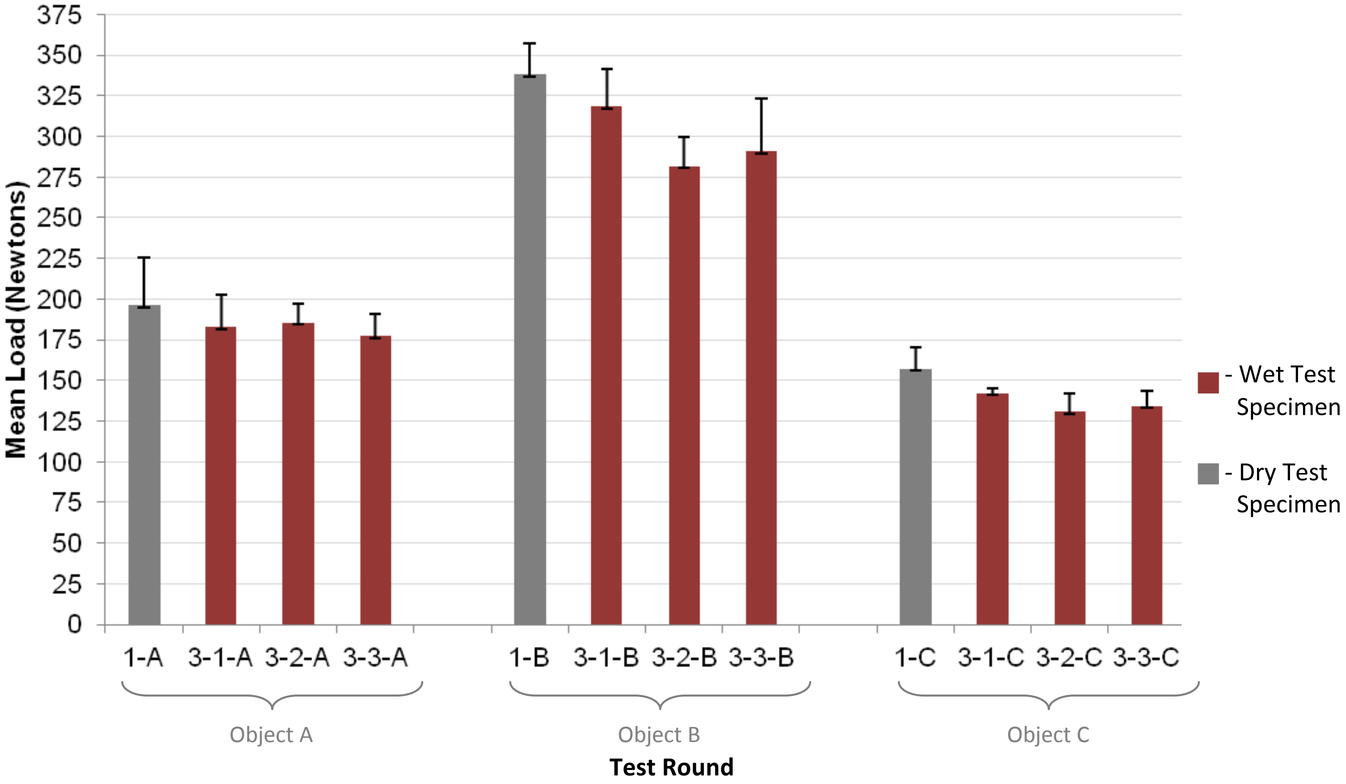


Graphs 24-25 below compare the mean dry strengths of untreated and treated weft and warp specimens from the different test rounds. Both graphs show that there was some variation in results between Test Group 1 and 3. These graphs appear to show that a significant amount of the strength lost from wetting was returned upon drying, although it appears like there was some slight reduction in strength incurred in all specimens in Test Group 3. However, these results do not take into account any differences in the *linear density* between test specimens of the same object. *Tenacity* figures given on the next page provide a truer equivalent strength for comparisons between Test Group 1 and 3. These are analysed next.

Graph 24 Test Group 1 versus Test Group 3 (Dry Untreated versus Dry Untreated)
Weft Specimens



Graph 25 Test Group 1 versus Test Group 3 (Dry Untreated versus Dry Untreated)
Warp Specimens



Tables 10-11 overleaf show that in terms of *tenacity*, there was a range of differences in mean strength between Test Group 1 and 3 for all three test objects. These results appear significant, with ranges of a **loss** in *tenacity* of 4.6% in weft specimens from Test 3-1-A to 23.8% in warp specimens from Test 3-3-B and a **gain** in *tenacity* of +0.8% in warp specimens from Test 3-2-A and +24.4% in warp specimens from Test 3-1-C.

However, *Student's t-test* calculations were again done to show the real significance of these different results, looking at the load-extension graphs of specimens and the probability of them being replicated in future results. These showed that the strength changes between Test Group 1 and 3 for the majority of test rounds were in fact insignificant and had a low chance of replication in future tests (see Appendix 8 for calculations). With the exception of changes in the following test rounds, shown next to their percentage change in dimensions and ratio probability of replication:

- Weft specimens 3-1-B, $^{-}$4.4% - 1:20 ratio
- Weft specimens 3-2-B, $^{-}$4.4% - 1:20 ratio
- Warp specimens 3-2-C, $^{-}$14% - 1:20 ratio
- Warp specimens 3-2-B, $^{-}$20.9% - 1:100 ratio

Student's t-test calculations suggest that wet cleaning with detergent wash solutions has had some effect on permanent strength for Objects B and C, but no effect on Object A. The soft water control wash solution did not have a significantly greater effect on strength for any test specimens. Test specimens from Object B incurred the greatest overall loss in strength in Test Group 3, with the result for warp specimens in Test 3-2-B showing significant reduction in strength with a high probability of replication of this result. The majority of specimens which suffered significant loss in strength had been wet cleaned in an Orvus WA® (anionic) detergent solution. As results were not unanimous throughout specimens however, it is not possible to suggest that this detergent is likely to a definitive permanent effect on strength in viscose rayon. More research is therefore required before conclusions can be drawn.

Specimens from Object C did not just fracture first in areas of black colourant as had been observed for Test Group 2, suggesting the likelihood that any reduction in strength in these areas was reversed upon air drying.

Table 10 Tensile strength from Test Group 1 and 3 (Dry Untreated and Dry Treated)
Weft Specimens

Test Round	**Object A**	**Object B**	**Object C**
Dry tenacity - Test 1 (N/tex)	0.0564	0.027	0.0082
Dry tenacity - Test 3-1 (N/tex)	0.0538	0.0258	0.0102
Dry tenacity - Test 3-2 (N/tex)	0.0484	0.0252	0.0072
Dry tenacity - Test 3-3 (N/tex)	0.0512	0.0278	0.0078
Percentage drop in strength	$^{-}$4.6 - $^{-}$14.2%	$^{+}$3 - $^{-}$6.7%	$^{+}$24.4 - $^{-}$14%

Table 11 Tensile strength from Test Group 1 and 3 (Dry Untreated and Dry Treated)
Warp Specimens

Test Round	**Object A**	**Object B**	**Object C**
Dry tenacity - Test 1 (N/tex)	0.0526	0.063	0.03
Dry tenacity - Test 3-1 (N/tex)	0.0502	0.0582	0.0284
Dry tenacity - Test 3-2 (N/tex)	0.053	0.0498	0.0258
Dry tenacity - Test 3-3 (N/tex)	0.048	0.048	0.0276
Percentage drop in strength	$^{+}$0.8 - $^{-}$8.75%	$^{-}$7.6 - $^{-}$23.8%	$^{-}$5.3 - $^{-}$14%

Conclusion and Summary of Results

During tensile strength testing, wet specimens put up little resistance to an increased load and tended to fracture quickly, causing the specimens to split in two. Following a controlled wet cleaning treatment, specimens from Object A c.1980-90s and Object B c. 1960s both suffered a reduction in *tenacity* of around half. Tensile strength test results for Object C c.1940s showed it was most affected by the controlled wet cleaning treatment, with a decrease in *tenacity* of up to 81%. However, it appears that wet tensile strength testing for Object C was affected by the black colourant present in the printing on the fabric. Statistical analysis showed there was a 1:100 probability that these same results would be achieved in any retesting. No one detergent type was shown to affect specimens more than another.

Mixed results were obtained from tensile strength tests designed to show whether reductions in strength caused by the controlled wet cleaning treatment were reversed upon air drying. However, statistical analysis comparing the load-extension graphs of each showed that most of these changes were insignificant. Of those which were shown as significant, these did not unanimously show that either detergent tested had a significant effect on permanent strength.

It should be noted that although comparison of results using *tenacity* took into account the *linear density* of specimens from the three test objects, the weave type may have affected results to some extent, with some weave constructions producing stronger fabrics than others. In addition, it was not known whether any finishes were applied to the fibres during manufacture and this may have had some impact on results. However, as results for both Object A and B were similar, this may indicate that these factors had little effect on specimens from either object. In terms of the different levels of ageing of the different fabrics, it is clear that degradation over time has had some impact on specimens form Test Object C, see above. The similar results obtained for Objects A and B however, suggest that differential levels of ageing may not have significantly impacted on tensile strength test results.

Conclusion

Evaluation of Project

The research aims and objectives for this project where largely fulfilled, with the exception of the aspects detailed below. Changes and improvements that would be made, were this project to be repeated, are listed subsequently.

Dimensional change observations were made however it was not possible to discuss these within the main body of this paper due to its limitations: though these have been included in Appendix ? for reference.

It was not possible to locate equipment to complete cross-section analysis for the fibre identification chapter.

Changes and Improvements

- Tensile strength test specimens would have a test area of 100/25mm, half the size recommended by BS 13934-1:1999. This is a standardised specimen size and would allow better comparison with testing carried out in other research.
- Reduce the number of tests rounds to allow more detailed comparisons

Summary of Findings and Recommendations for Conservation

A summary of Part One of this paper is now given below.

The literature review highlighted a need for more research into the conservation of viscose rayon, such as that conducted for this project.

Research into the history and manufacture of viscose rayon highlighted the plethora of manufacturing processing finishes used and how these may affect fibres differently. In particular, viscose rayon can be produced to appear like silk, cotton or wool. Conservators should be aware of the diversity of appearances and properties possible for this fibre.

Research showed that early viscose rayon, pre-1940s, may have even poorer wet strength than the dated examples tested for this project. However, this could not be investigated, as an example of viscose rayon from this early date was not found.

Reviewing fibre properties showed the similarities and differences between the wet properties of different regenerated cellulosic fibres as well as difficulties with telling them apart, as they are all essentially composed of the same molecular structure: cellulose II. In terms of conservation, this has pertinence for fibre identification and determining the level and extent of any treatment undertaken, albeit, viscose rayon has a typical microscopic appearance of longitudinal striations different to other regenerated cellulosics, which would usually set it apart.

A summary of Part Two of this paper is now given.

Fibre Identification Analysis

The use of a combination of *basic* techniques was shown to be more successful for distinguishing some fibres than others, as verified with ATR-FTIR Spectroscopy.

Basic techniques proved successful for identifying viscose rayon from cellulose acetate. Viewed under a microscope, both cellulose acetate and viscose rayon fibres had surface striations, cellulose acetate’s fibres however had blurred edges in contrast to viscose rayon’s focused edges. Acetone solubility tests dissolved cellulose acetate samples, verifying observations from microscopy, and further ATR-FTIR findings showed identification to be accurate.

Basic techniques were less successful at identifying cupro and lyocell, both having a smooth undefined surface under the microscope, potentially being one of a number of man-made fibres. It took ATR-FTIR Spectroscopy to identify both as regenerated cellulosic fibres.

Both ATR-FTIR and *basic* techniques were inconclusive for identifying the two samples of blended fibres. More research and information needs to be available to conservators on blended fibres.

ATR-FTIR proved to be successful at differentiating natural cellulose fibres from regenerated cellulose fibres by the part of spectra which show OH groups. In combination with microscopy, ATR-FTIR was shown successful at distinguishing mercerised cotton from regenerated cellulosic fibres.

Garment labels present in two of the older objects proved vague. The 1930s object which was identified as cupro was labelled ‘All-Foreign Rayon’. It would not be possible to class cupro as rayon today in Britain. The pink 1960s dress which was labelled as ‘Francine’ a material ‘made to look like silk’ was shown to contain cellulose acetate in the lining and viscose rayon in the outer material, however, no distinction was made between the two different fibres. This shows that conservators should be aware of obsolete past connotations of fibre names still in use today, as well as of vagueness in distinction between materials in older labels.

Tensile Strength and Dimensional Change Testing

Testing conducted showed that both older and more recent examples of viscose rayon all display poor wet properties.

During wet cleaning treatments, prior to testing, fibres did not show signs of any visible weakening. However, tensile strength tests highlighted a significant loss in strength within all specimens. Specimens from c. 1960s and c.1980/90s showed a reduction in *tenacity* of around 50%. Specimens from c.1940s showed a reduction in *tenacity* of around 80%, though, it is likely degradation inflicted by the black colourant in these affected results to some extent. The impact that the degradation had on c.1940s specimens may indicate that, the reduction in strength that occurs when viscose rayon fibres are wetted greatly exacerbates weaknesses from degradation. It is possible that the way the c.1940s specimens behaved during testing could highlight an issue with viscose rayon textiles in general. Areas which may appear strong prior to wet cleaning but which have undergone a certain level of degradation, may become weakened enough through wetting for splitting to occur in those areas. This may be difficult for conservators to predict and may possibly be an issue in the future as viscose rayon textiles become older and more degraded, though more research is required into this.

It is evident that the loss in strength incurred on viscose rayon during wet cleaning resulted in causing stress on fibres of whatever age. Whereas tests on specimens dried following wet cleaning indicated that most did not suffer significant permanent strength loss, more research is required to verify how representative these results are, because a few specimens did show more significant strength loss.

It is concluded that wet cleaning can be a suitable treatment option for viscose rayon from different times providing objects are acutely monitored during treatment for any signs of weakening from wetting which could cause splitting. Tensile strength tests conducted on wet specimens resulted in quick fracturing, causing whole specimens to be split in two. It is possible that this behaviour may be replicated in any viscose rayon fibres which become sufficiently weakened to fracture in the wash bath. The number of wet cleaning treatments viscose rayon objects are subjected to should be minimised, to reduce the number of times wetting puts stress on fibres. If fibres are visibly degraded in any way, wet cleaning may not be appropriate as fibres are likely to be weakened by at least around 50%, significantly increasing the risk of further damage to fibres during such treatment. Thompson, and, Tímár-Balázsy and Eastop, mention using a framed net support when cleaning viscose rayon and this seems like good advice, to reduce strain put on fibres.[141-142]

Neither Orvus WA® (anionic) nor Dehypon LS45® (non-ionic) detergents appeared to affect wet strength more than the other and therefore it is concluded that both are suitable for use for wet cleaning viscose rayon fibres. More research is required into any permanent strength loss as a result of using these detergents, as described above.

Suggestions for Future Research

Below are suggestions for future research into the conservation of viscose rayon:

- Build up more photomicrograph references showing the different variations of appearance of viscose rayon and other man-made fibre types, to help conservators with fibre identification.
- Build up more knowledge so conservators can identify blended fibres.
- Research the behaviour of viscose rayon blended fibres during wet cleaning treatment.
- Research carried out for this project has indicated that wet cleaning treatments significantly weaken viscose rayon from between c.1940s-1990s. Would a solvent cleaning treatment have less of an impact on viscose rayon?
- To what extent does degradation, such as that observed in Test Object C, contribute to reducing wet strength in viscose rayon fibres?
- How does the presence of colourants in viscose rayon fibres affect their degradation?
- What detergents are most suitable to use with other man-made fibres?

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[141] Thompson, (1992), p. 17.

[142] Tímár-Balázsy and Eastop, (1998), p. 142.

- Conduct more research testing the wet properties of viscose rayon, to build up a more substantial body of knowledge which will help to show how representative results obtained during this project are, relating to how the strength and dimensions of viscose rayon are affected by wet cleaning treatments.
- Do dimensional changes (see Appendix 6) that occur with viscose rayon as a result of wetting revert upon drying?
- To what extent are examples of viscose rayon from 1905-1940s affected by conservation wet cleaning treatments?
- Do high relative humidity levels affect the strength of viscose rayon textiles?

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Appendix 1 – Viscose Rayon Trade Names

Below is a list of trade names given to viscose rayon fibres processed in various ways, manufactured from around the world, for the textile industry. The list provided should not be viewed as extensive.

This list has been comprised from information provided in the following sources:

- ASTM Textile Mercury Annuals International, *ASTM D276 – 12,* *(*http://www.astm.org/Standards/D276.htm)
- British Man-Made Fibres Federation, *Better Living with Man-Made Fibres*
- Bruce A. Townsend, 'Full Circle with Cellulose, in Raymond B. Seymour and Roger S. Porter (eds.) *Manmade Fibers: Their Origin and Development*
- R. W. Moncrieff, 'Appendix – Commercial Man-Made Fibres', in *Man-Made Fibres*

A
Absorbit
Alastin
Alastra
Amplum
Asahi
Avila
Avisco

B
Bijohai
Blackbird
Bobal
Bodana
Bodanella
Bodanita
Bredanese
Brenka
Brenkona
Briglo
Britenka
Bubblfil

C
Calyx
Cantona
Caste Tulle
Celta
Cheviot
Chevisol
Cisalpha
Colcesa
Colnova
Colomat
Coloray
Colva
Colvalan
Conyma
Cordenka
Corval
Courdulla
Cova
Crepesyl
Crispella

D
Daifuki
Danufil
Danuflor
Decora
Delustra
Dulesco
Dulkona
Dul-Tone
Dynacor

E
Enkona
Enka
Essevi
Evlan

F
Featheray
Fiber E
Fibrana
Fibranne
Fibrelta
Fibrenka
Fibro
Fibresilk
Flimba
Flisca
Flock
Flox

G
Glanzstoff

I
Iridex
Iridye
Ivorea

J
Jedmat
Jedsol
Jedsyl

K
Kasilga
Kelheim
Kikansei

Kirksyl
Kuralon

L
Lacisana
Lustrafil
M
Madame
Butterfly
Marimusume
Matapoint
Meryl B8

N
Nupron

O
Oplexmat

P
P.A.
Paramafil
Phrilan
Phrix STW
Pontova

R
Rainbow
Ratujal
Rayflex
Rayolanda
Rotwyla
Rovicella

S
Sarille
Sastiga
Shiro Diafil
Skenandoa
Sniafil
Spinstra
Superbreda
Supral
Supralan
Suprenka
Sviloza

T
Tendan
Tenkyo
Topel
Toyobo
Tropic
Tudenza
Tyrex
Tyron

V
Venus
Vibrem
Visada
Vistra XT
Vistralen

X
Xtra Dull

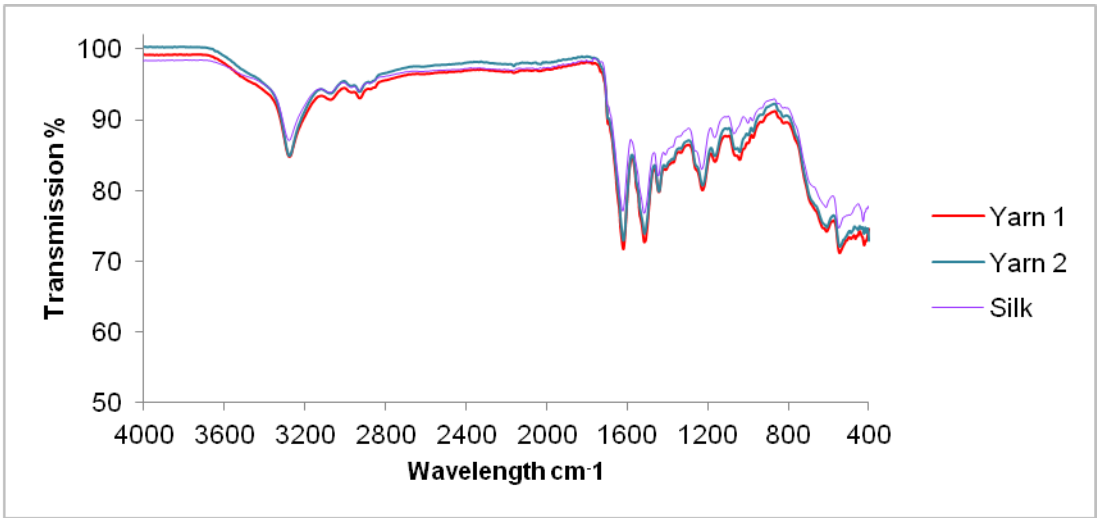
Z
Zehla
Zellwolle

Appendix 2 – ATR-FTIR Spectra

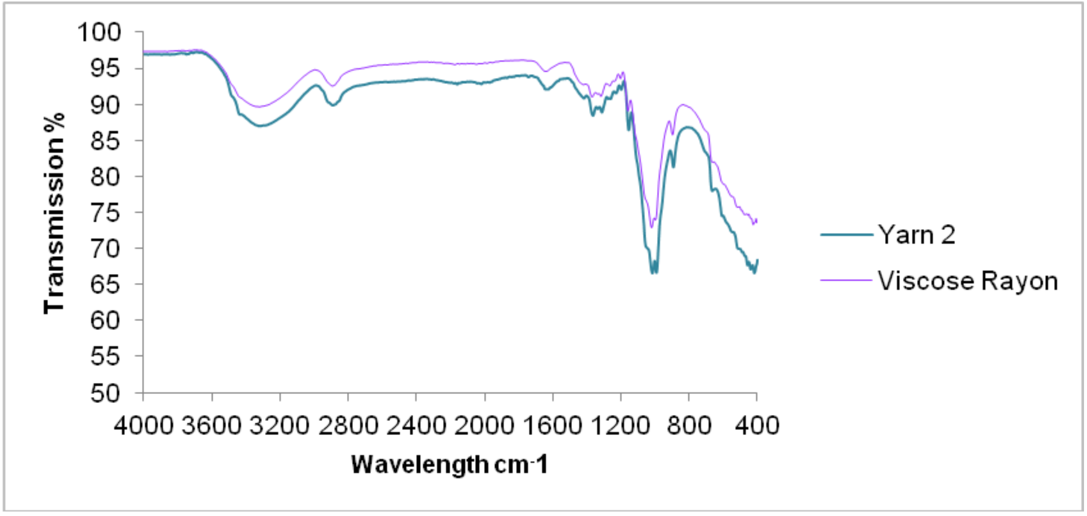
ATR-FTIR (Attenuated Total Reflectance-Fourier Transform Infrared) spectroscopy is used to identify organic materials. An infrared radiation beam is passed through a material sample into a detector. Different molecule structures absorb different infrared frequencies and this is information is then recorded as a spectrum on a graph for analysis.

ATR-FTIR spectra from objects analysed in Chapter 4 but not included in the main text are included here in Graphs 26-30 for reference. This includes spectra obtained from both yarn directions for Objects 1, 4 and 6, and, from just yarn 2 for Objects 2 and 9. A reference spectrum is included in each graph to show what fibre type the object was matched to.

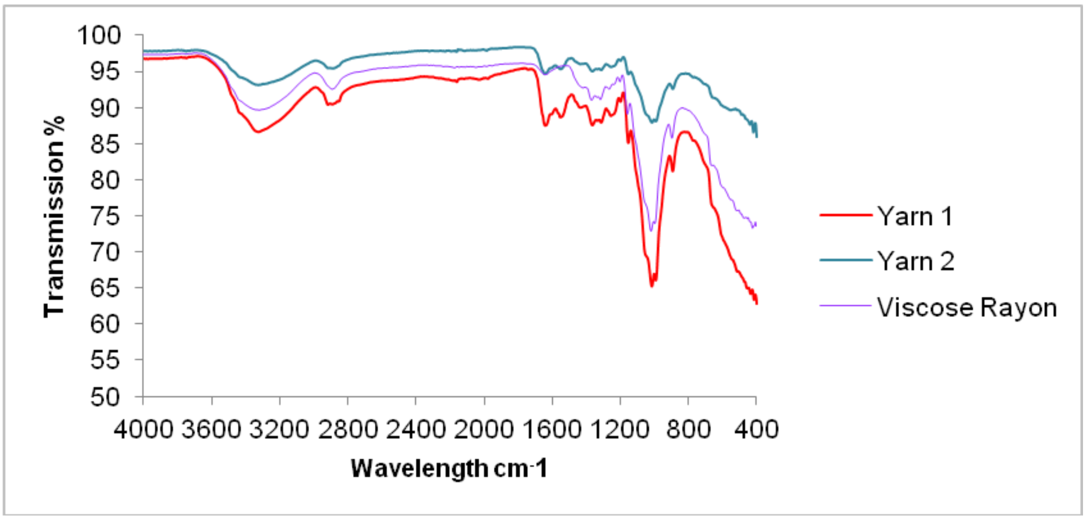
Graph 26 ATR-FTIR spectra, Object 1, both yarns



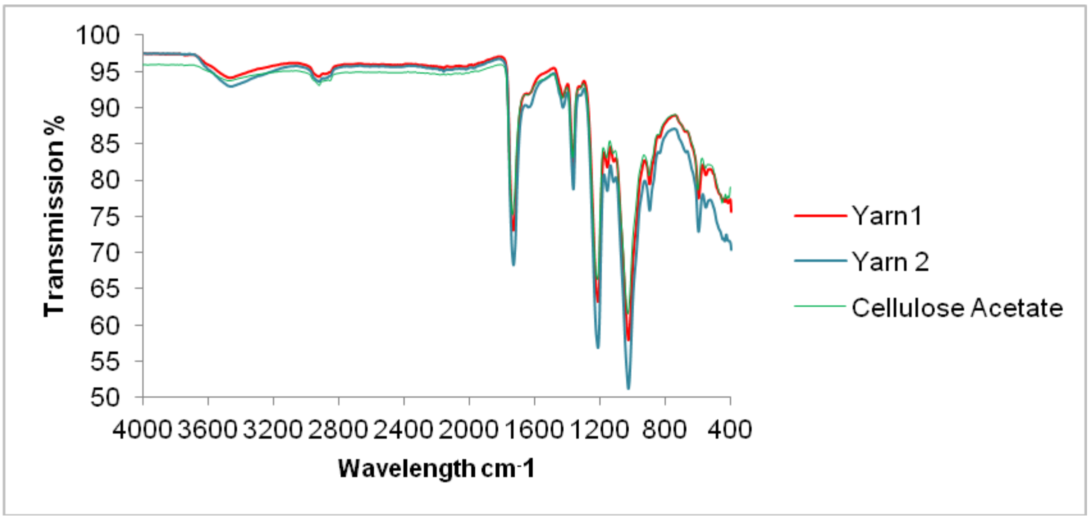
Graph 27 ATR-FTIR spectrum, Object 2, yarn 2: with reference of another regenerated cellulosic fibre



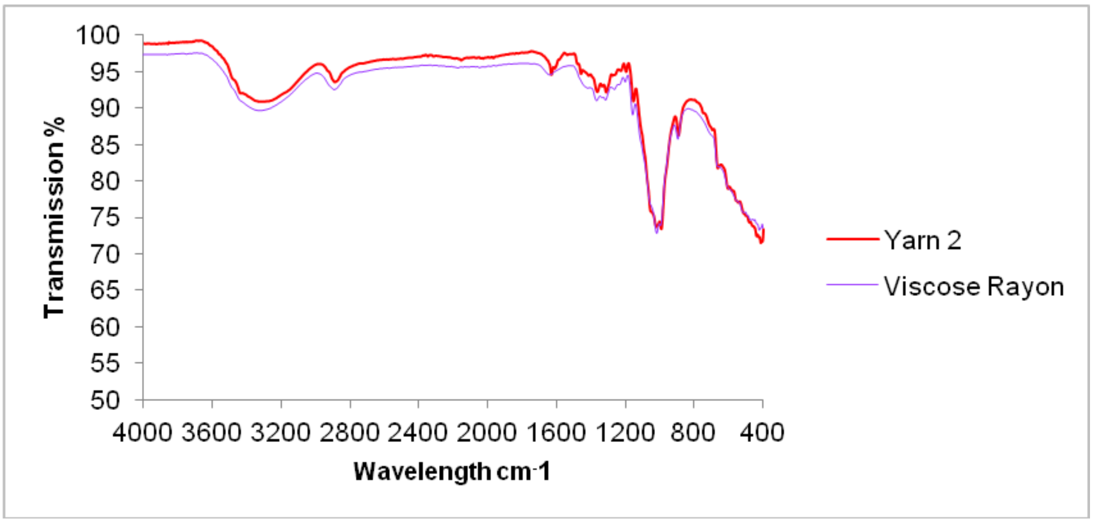
Graph 28 ATR-FTIR spectra, Object 6a, both yarns



Graph 29 ATR-FTIR spectra, Object 6b, both yarns



Graph 30 ATR-FTIR spectra, Object 9, yarn 2

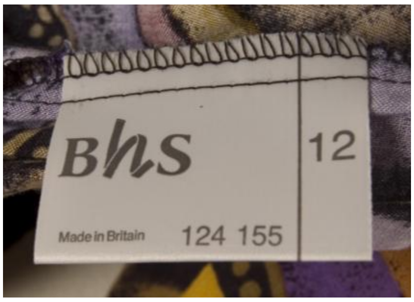


Appendix 3 – Test Object Samples and Dating (for Chapter 5)

Below is information on the dating assigned to test objects and a brief analysis of their fabric. All fabrics were identified as viscose rayon, warp and weft, following analysis in Chapter 4. A swatch of each test object fabric is attached for reference.

Fig. 57 Test Object A – *C.1980s/1990s* B*h*S *Trousers*

Garment Label Front



Garment Label Back

Dating:
Bright bold prints were typical of the 1980s as were harem-shaped trousers.[143]

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[143] Lisa Dyer (Ed.), Vintage Fashion: Collecting and Wearing Designer Classics, (London: Carlton Books Ltd, 2006), p. 196.

Fig. 58 Test Object B – *C.1960s Ricol Fabric*

Garment label

a ricol dress fabric

*100% Rayon
*Width 35/36" *Washable

PRODUCED IN WEST GERMANY

ricol
100% Rayon

Your sew-in label

Sewing points

thread | Sylko 50
needle | 14
stitch | 10-12
foot pressure on fabric: medium
Interlining where required:
Courtaulds Solena: D18
Lightning Zip:
dress: black or gold peck
skirt: red/black peck

Fabric care

HAND WASHING

4

water | hand hot 48°C
drying | cold rinse drip dry
do not bleach | do not wring

MACHINE WASHING

water | hand hot medium wash 48°C
drying | cold rinse short spin.
do not bleach | do not wring

IRONING

Medium hot on wrong side of damp fabric
• 1 COOL
• 2 WARM
• 3 MEDIUM HOT
• 4 HOT

P This fabric may be dry cleaned. Please draw your cleaners attention to the symbol "P" on the garment label.

General sewing hints

breaking of needle might be caused by:

1 incorrect size of needle for thread and material
2 needle blunt
3 pulling of material when stitching
4 needle striking improperly fastened presser foot or attachment

breaking of thread might be caused by:

1 a knot in thread
2 incorrect threading (see instruction book)
3 needle tension too tight
4 needle blunt or bent
5 thread too coarse for needle

skipping stitches might be caused by:

1 needle not inserted in needle clamp as far as it will go
2 needle threaded incorrectly (thread from long groove side to short groove side)
3 needle blunt or bent
4 needle too small for thread

puckering seams might be caused by:

1 tension too tight
2 stitch too long for material being sewn
3 incorrect thread used

<u>Dating</u>:
Psychedelic prints like this were fashionable during the 1960s.[144]

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[144] Dyer, (2006), pp. 116 and 131.

Fig. 59 Test Object C – *C.*1940s Dress

Seam Construction

Metal Press Stud Fastening

Dating:
Many clothes in 1940s were made of man-made materials due to shortages caused by World War II. Shortages also meant dresses were cut slim, contrasting coloured collars were also popular. 'Decorative rayon dresses in pretty colourful prints with draping and shirred fronts were worn in the daytime, sometimes with a soft crossover bodice […] particularly popular were dolman and three-quarter-length sleeves.'[145] The dress is also constructed of an enclosed seam and fastened with poppers, both typical methods employed in dressmaking in the 1940s.

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[145] Dyer, (2006), p. 65.

Appendix 4 – Detergent Wash Solution Calculations

All wash solutions were mixed with soft water from the tap. The soil anti-redesposition agent SCMC (Sodium Carboxymethyl Cellulose) was added to each wash solution at a concentration of 0.05g/Litre. The detergent concentration calculations are given below.

Anionic Detergent Solution

Orvus WA Paste ® (cost £10.90/454g)
Concentration:5 x cmc: % cmc = 0.29065
5 x % cmc = 1.4
Converted to grams = 14g/Litre

Non-ionic Detergent Solution

Dehypon LS45® (from Conservation by Design, cost £10.00/Litre)
Concentration:5 x cmc: % cmc = 0.05977
5 x % cmc = 0.29
Converted from % into g = 3g/Litre

Appendix 5 – Tensile Strength Testing Room Conditions

Room conditions in Table 12 were taken from a J S Holdings, Electronic Precision Thermo-Hygrograph (NAK REF TH-6, ISUZU CAT. NO. 3-3126).

Table 12 Room Conditions During Testing

Test Round	Temperature (°C)	Relative Humidity (%)
1-A	24	41
2-1-A	22	40
2-2-A	23	40
2-3-A	22.5	40
3-2-A	23	51
3-2-A	23.5	53
3-3-A	23.5	47
1-B	23	50
2-1-B	23	40
2-2-B	24	40
2-3-B	23	47
3-2-B	24	55
3-2-B	24	59
3-3-B	23.3	49
1-C	24	50.5
2-1-C	24	54
2-2-C	24	59
2-3-C	24	49
3-2-C	23.5	50
3-2-C	24	52
3-3-C	24.5	51

Appendix 6 - Dimensional Change Observations

Alongside tensile strength testing for Test Group 2, dimensional changes as a result of wet cleaning were measured to record any growth or shrinkage. In addition, the different wash solutions of non-ionic detergent, anionic detergent and soft water would show whether these variables affect dimensional change.

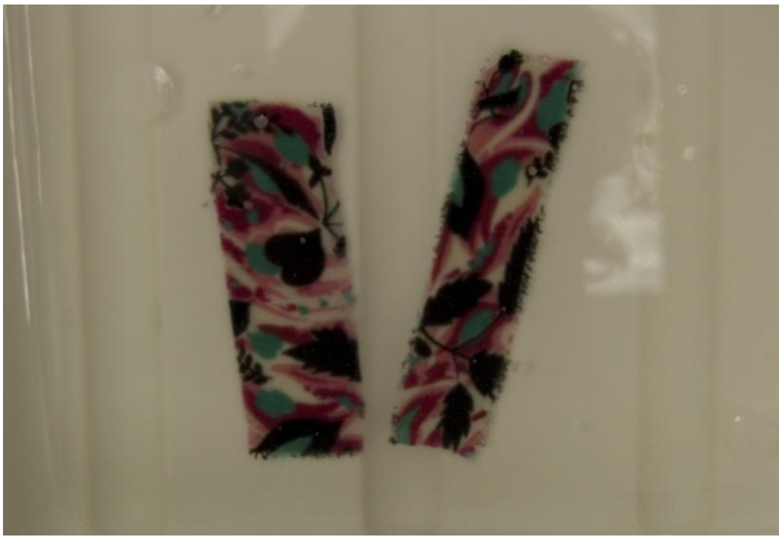
Two measurements were taken of specimens in Test Group 2: (1) dry measurements before wet cleaning treatment, (2) wet measurements immediately following wet cleaning treatment and before tensile strength testing. All four edges of the perimeter of test specimens were measured (Length/Width/Length/Width). However, only one measurement has been given for length and width in the analysis, as all dimensional changes occurred evenly on parallel sides.

Dimensional Change Observations during Wet Cleaning

It was noted that visible growth and shrinkage occurred on some specimens in the wash bath. Visible changes were most marked in Object C specimens, with visible dimensional changes in specimens from Objects A and B being far subtler. Warp specimens tended to shrink in length and, conversely, weft specimens tended to grow in length whilst decreasing in width (see fig. 60).

Specimens appeared to swell in density as a result of wetting, indicating that fibres were expanding in diameter in addition to any growth or shrinkage in length. Due to the fineness of density of the test specimens however, it was impossible to measure this with the resources available for this project.

Fig. 60 Visible dimensional change in a warp (left) and weft (right) specimen in the wash bath during Test 2-1-C



5.8.b Dimensional Change Test Results

The mean dry and wet measurements of specimens are presented for comparison in Tables 13-14. Percentage change in measurements are also included in the table, calculated using the formula below.[146]

$$\frac{\text{Mean wet length} - \text{Original length}}{\text{Original length}} \times 100 = \%\ \text{Dimensional Change}$$

Most weft specimens had shrinkage in width ranging between -4.5% to -8.8%, with the exception of Test 2-1-C which had growth in width of +4.5% and Test 2-1-B which had no change (see Table 13). Conversely, most weft specimens grew in length between +1% to +4.6%, with the exception of Test 2-2-B which had shrinkage of -0.2% (see Table 13).

Warp specimens showed less coherence in dimensional changes in width. Specimens from Object A had no changes in width apart for a small increase of +0.8% in Test 2-1-A. Object B specimens all incurred some shrinkage in width between -0.8% and -2.4%. Whereas Object C specimens had the most diversity in dimensional changes; ranging from -0.8% shrinkage for Test 2-2-C, to a +3.2% growth for Test 2-1-C and a +4% growth for Test 2-3-C (see Table 14). All warp specimens shrunk in length between -1.4% to -7.2%.

Results did not indicate that one wash solution had a greater impact than another (see Tables 13-14) and so it is concluded that neither Dehypon LS45® (non-ionic) nor Orvus WA® (anionic) detergent wash solutions affected dimensional change.

Dimensional Change testing showed growth and shrinkage in specimens from all test objects, indicating that older versions of viscose rayon do not undergo any significantly greater dimensional changes as a result of conservation wet cleaning. Weft specimens all grew in length with the exception of one which shrank (Test 2-2-B) and that warp specimens all shrank in length. In terms of wet cleaning viscose rayon objects, this may indicate a tendency for a certain degree of growth in the weft direction and shrinkage in the warp direction in examples of viscose rayon from different time periods. Further research needs to be done to show the significance percentage dimensional change has for viscose rayon in comparison with other common fibre types. In particular, it would be interesting to compare it with wool, which can felt[147] as a result of wet cleaning.[148]

It was noted after testing, that it would have been beneficial to measure whether any dimensional changes were reversed once specimens were air dried following wet cleaning. In hindsight it would have been better to conduct dimensional change testing on Test Group 3 specimens, which were all air dried following wet cleaning before being tensile strength tested.

In terms of conservation, conducting a wet cleaning treatment on viscose rayon textiles, dimensional changes could cause tension on any seams present and distort the original shape of objects to some extent. Viscose rayon objects should be supported during wet cleaning to reduce the likelihood of the added weight from water absorption stretching fibres and exacerbating dimensional changes. Viscose rayon objects should also be supported

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[146] Dimensional Change calculation taken from: Hatch, (1993), p. 19.

[147] Felting is an irreversible shrinkage that occurs in wool, caused by fibres being agitated in water. See, Gohl and Vilensky, (1981), p. 70.

[148] Gohl and Vilensky, (1981), p. 70.

when drying to reduce the risk of causing differential strain as the water (and added weight) evaporates, which could result in distortions. It should be noted that more research is required to indicate to what level any changes in dimensions revert upon drying.

Table 13 Dry and Wet specimen measurements taken before and after wet cleaning
Weft Specimens

Test Round	Original Dry Length (mm)	Mean Wet Length (mm)	% Change in Mean Length	Original Dry Width (mm)	Mean Wet Width (mm)	% Change in Width
2-1-A	100	102.4	+2.4%	25	23	-8%
2-2-A		101.4	+1.4%		23.4	-6.4%
2-3-A		101.4	+1.4%		23.8	-4.8%
2-1-B	100	101	+1%	25	25	0%
2-2-B		99.8	-0.2%		23.8	-4.8%
2-3-B		101.2	+1.2%		23.6	-5.6%
2-1-C	100	104.25	+4.25%	25	26	+4.5%
2-2-C		103.6	+3.6%		23.4	-6.4%
2-3-C		104.6	+4.6%		22.8	-8.8%

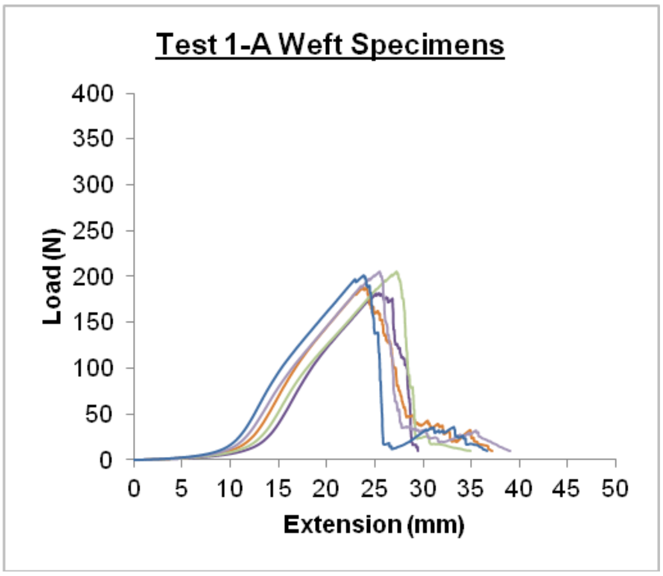
Table 14 Dry and Wet specimen measurements taken before and after wet cleaning
Warp Specimens

Test Round	Original Dry Length (mm)	Mean Wet Length (mm)	% Change in Mean Length	Original Dry Width (mm)	Mean Wet Width (mm)	% Change in Width
2-1-A	100	96	-4%	25	25.2	+0.8%
2-2-A		97	-3%		25	0%
2-3-A		97.2	-2.8%		25	0%
2-1-B	100	97.6	-2.4%	25	24.6	-1.6%
2-2-B		97.2	-2.8%		24.4	-2.4%
2-3-B		98.6	-1.4%		24.8	-0.8%
2-1-C	100	94.6	-5.4%	25	25.8	+3.2%
2-2-C		96.4	-3.6%		24.8	-0.8%
2-3-C		92.8	-7.2%		26	+4%

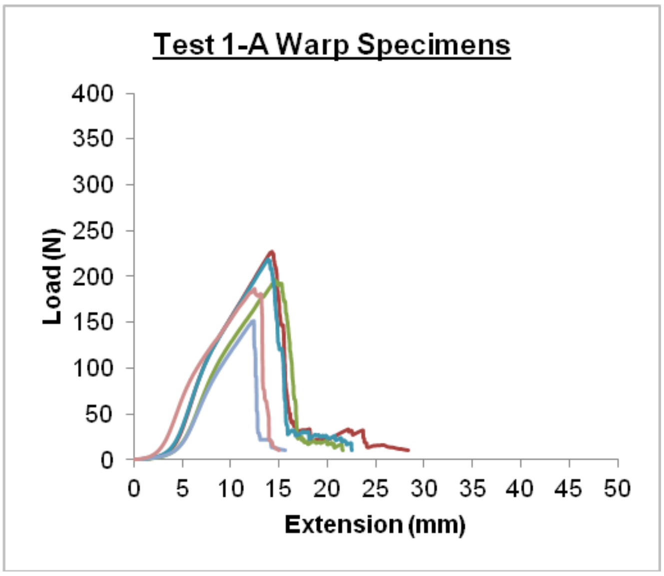
Appendix 7 – Tensile Strength Testing Load-Extension Graphs

All load-extension graphs from tensile strength testing are now given in this appendix for reference, including their mean maximum load and mean standard deviation rounded to two decimal places. All graphs were created in *Microsoft Excel 2007,* using data obtained from the *Instron* Tensile Strength Tester's *Bluehill* software.

Test Object A: *Test Group 1*

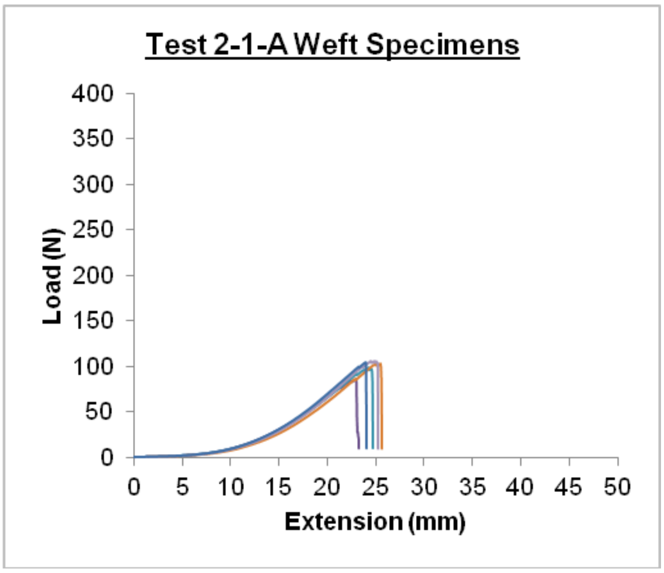


Mean maximum load= 196.58
Mean standard deviation = 11.06

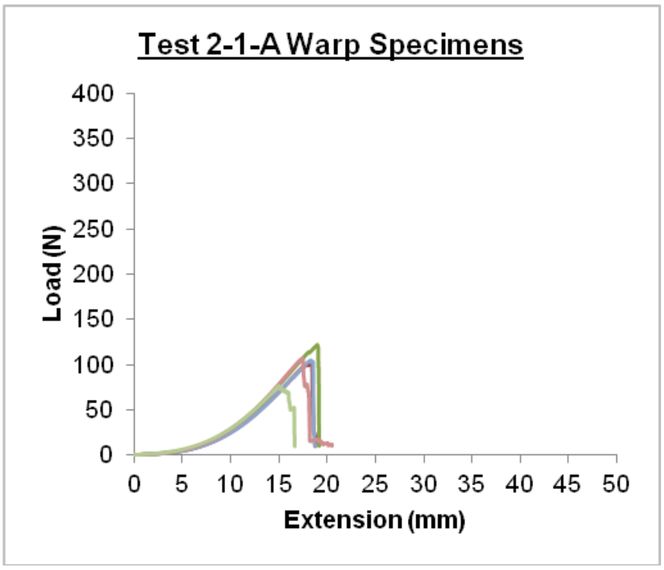


Mean maximum load= 195.94
Mean standard deviation = 29.81

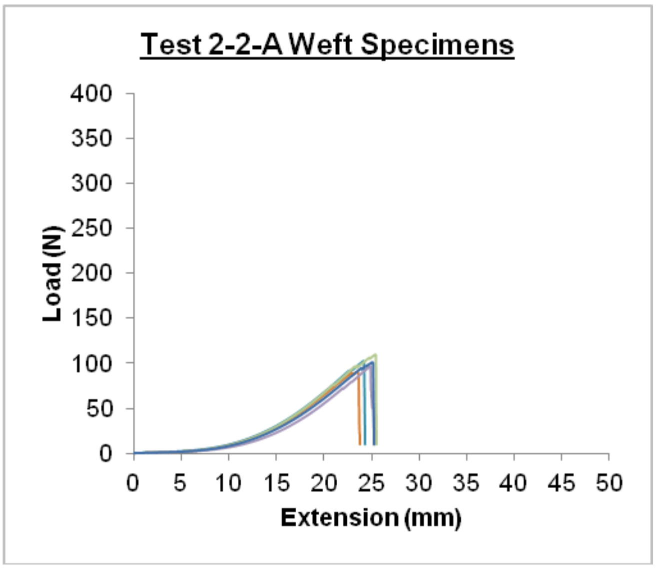
Test Object A: *Test Group 2*



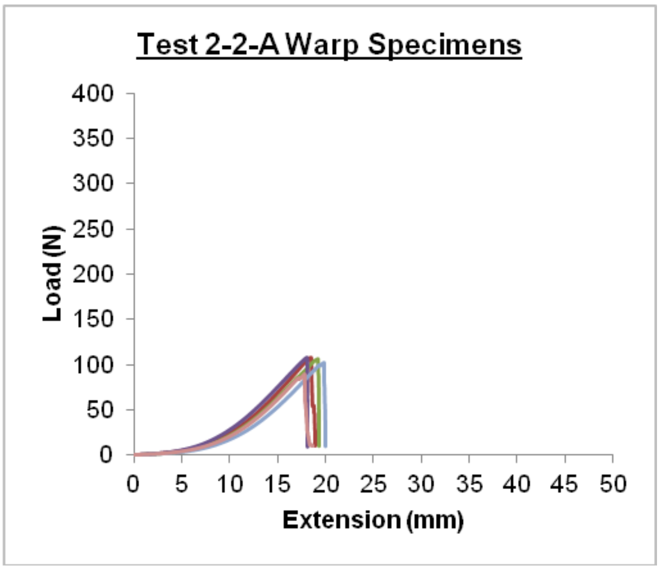
Mean maximum load= 99.92
Mean standard deviation = 8.10



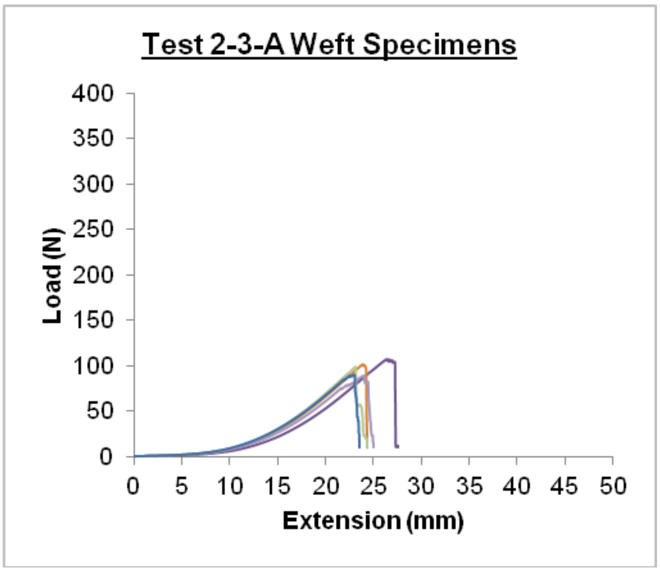
Mean maximum load= 102.61
Mean standard deviation = 16.41



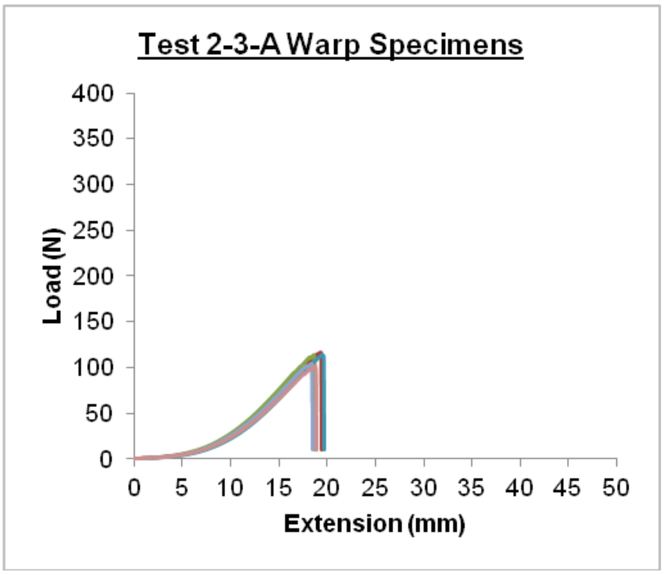
Mean maximum load= 100.34
Mean standard deviation = 6.96



Mean maximum load= 102.40
Mean standard deviation = 7.83

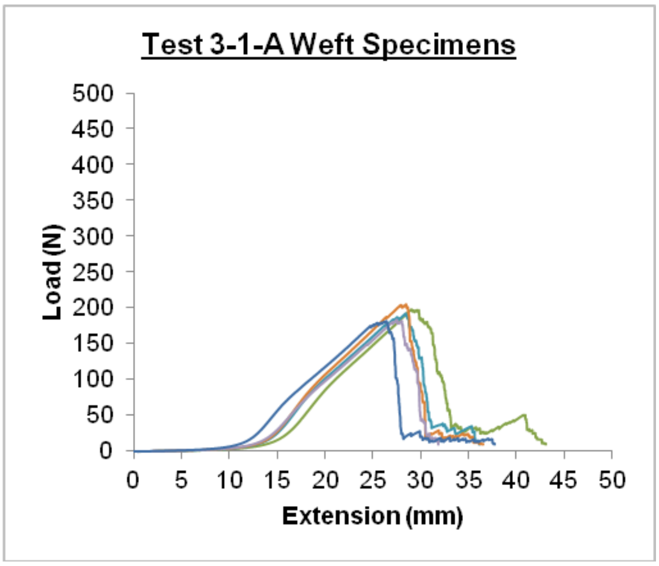


Mean maximum load= 98.85
Mean standard deviation = 10.09

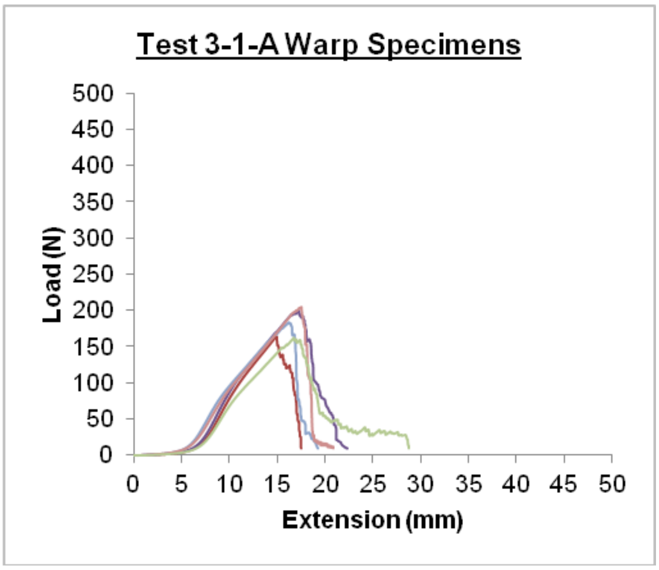


Mean maximum load= 108.25
Mean standard deviation = 6.70

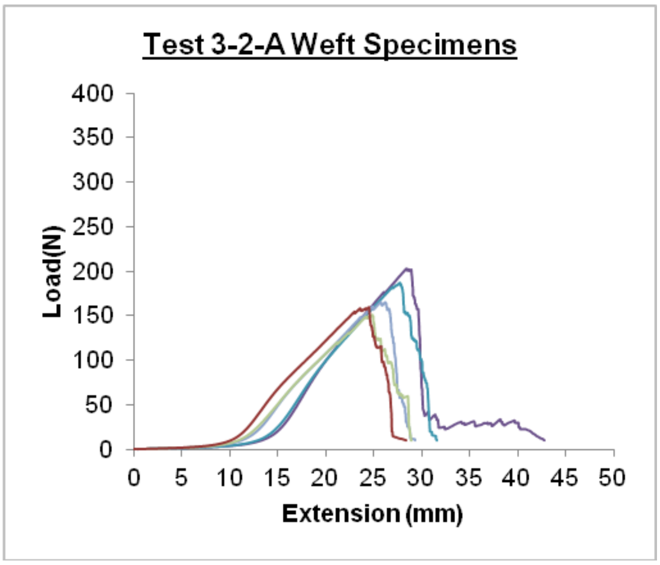
Test Object A: *Test Group 3*



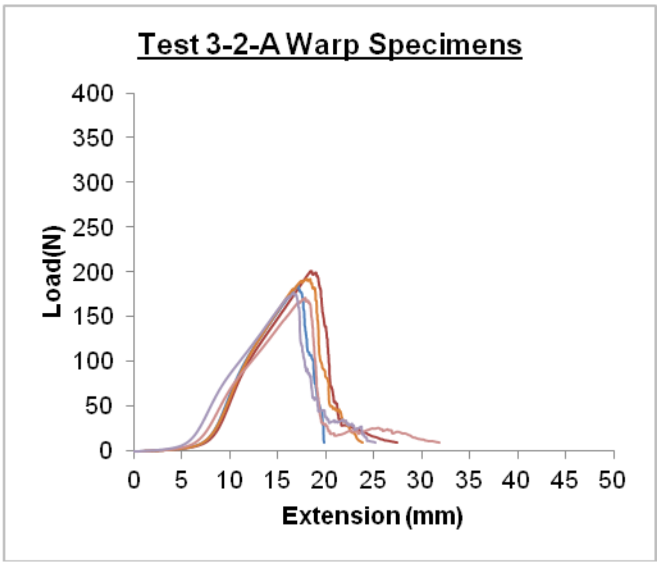
Mean maximum load= 192.41
Mean standard deviation = 9.63



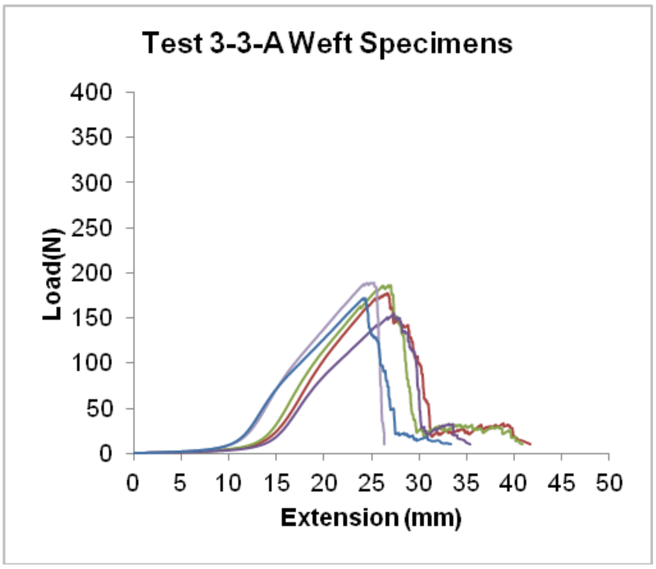
Mean maximum load= 182.67
Mean standard deviation = 19.73



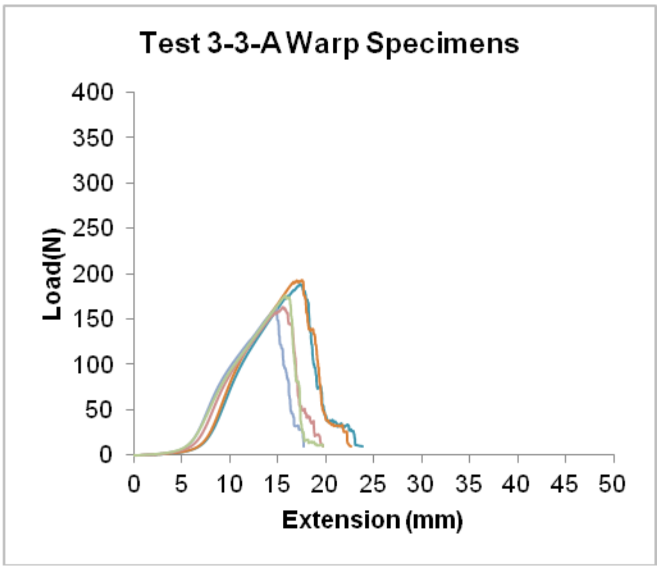
Mean maximum load= 173.18
Mean standard deviation = 21.03



Mean maximum load= 185.64
Mean standard deviation = 11.67

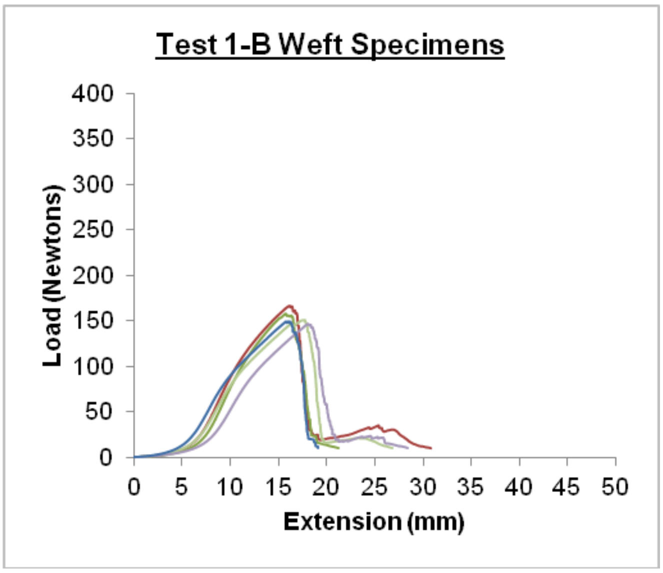


Mean maximum load= 176.24
Mean standard deviation = 13.90

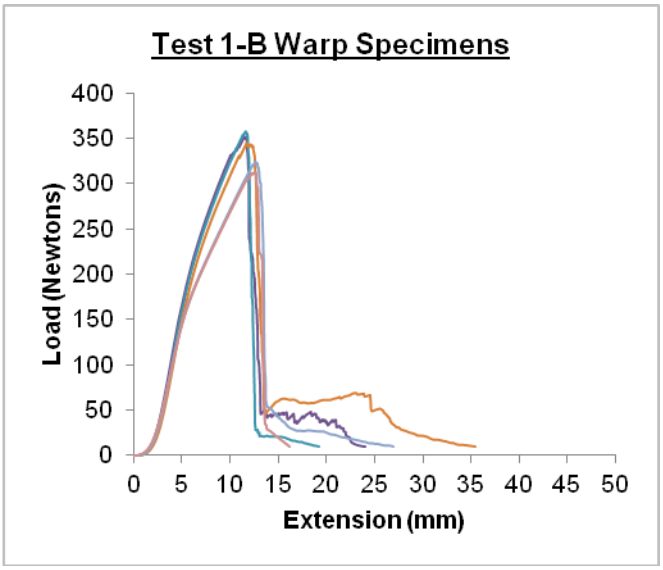


Mean maximum load= 177.08
Mean standard deviation = 13.91

Test Object B: *Test Group 1*

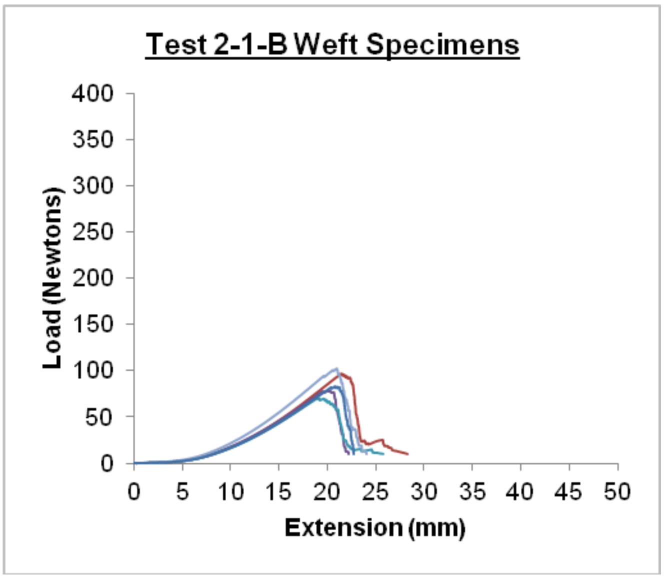


Mean maximum load= 154.17
Mean standard deviation = 7.77

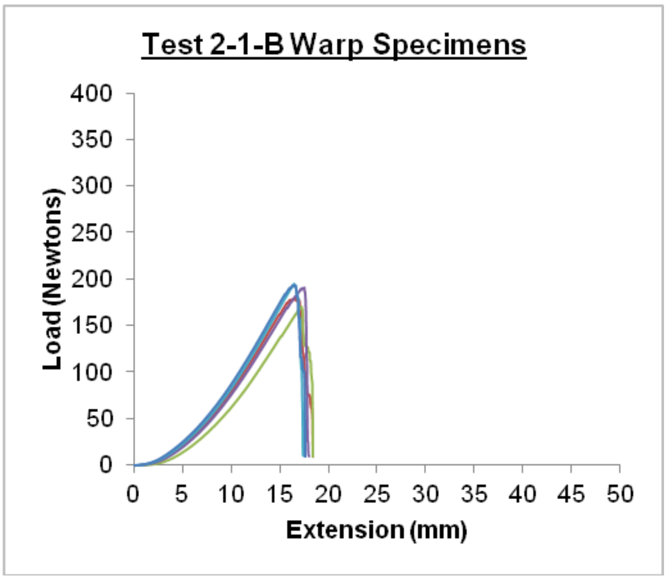


Mean maximum load= 338.01
Mean standard deviation = 19.45

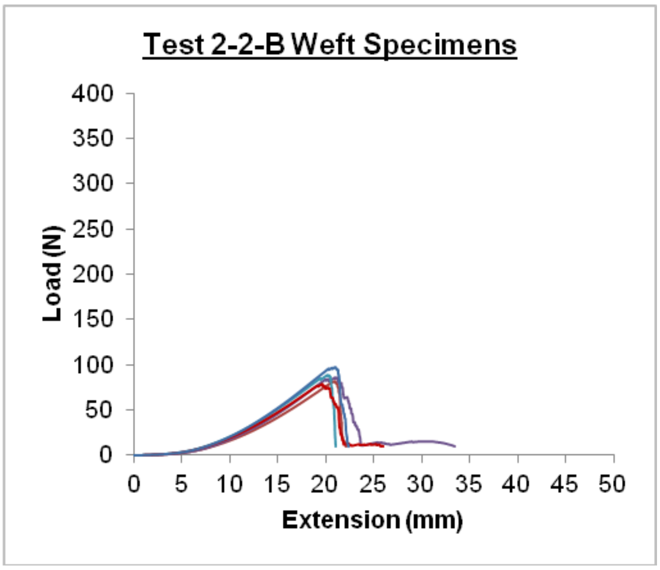
Test Object B: *Test Group 2*



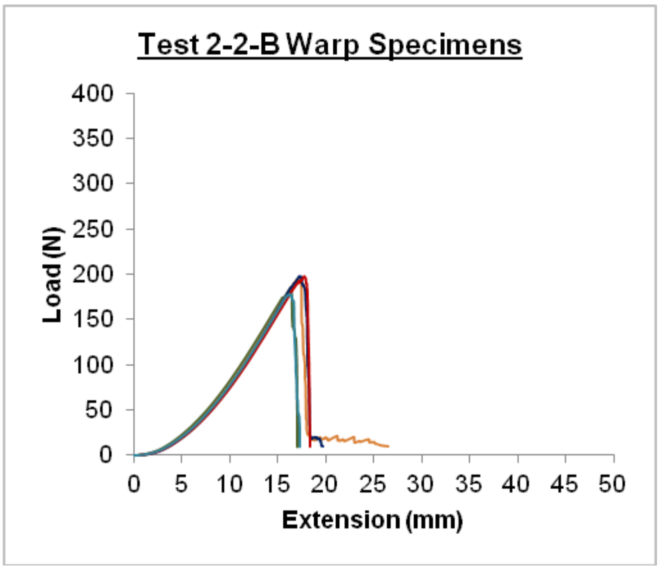
Mean maximum load= 86.34
Mean standard deviation = 13.22



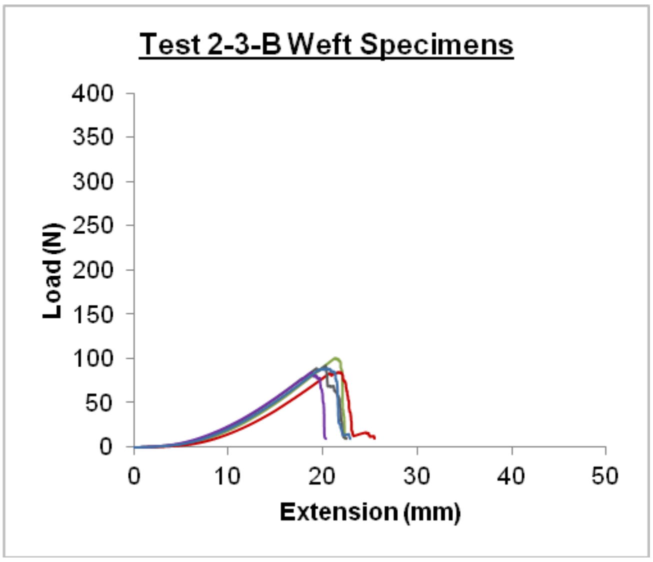
Mean maximum load= 185.77
Mean standard deviation = 10.53



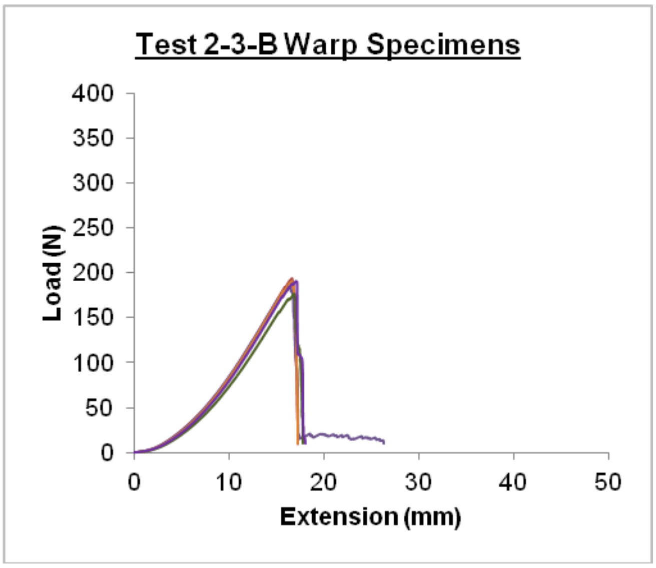
Mean maximum load= 86.66
Mean standard deviation = 7.39



Mean maximum load= 190.69
Mean standard deviation = 9.05

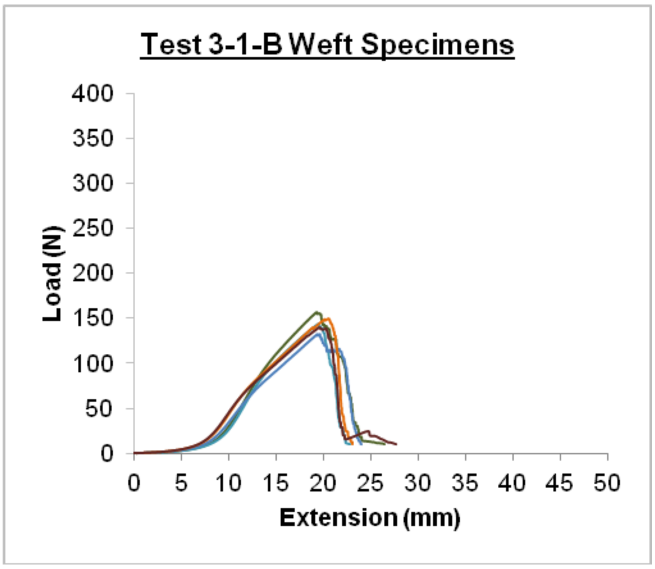


Mean maximum load= 90.32
Mean standard deviation = 6.90

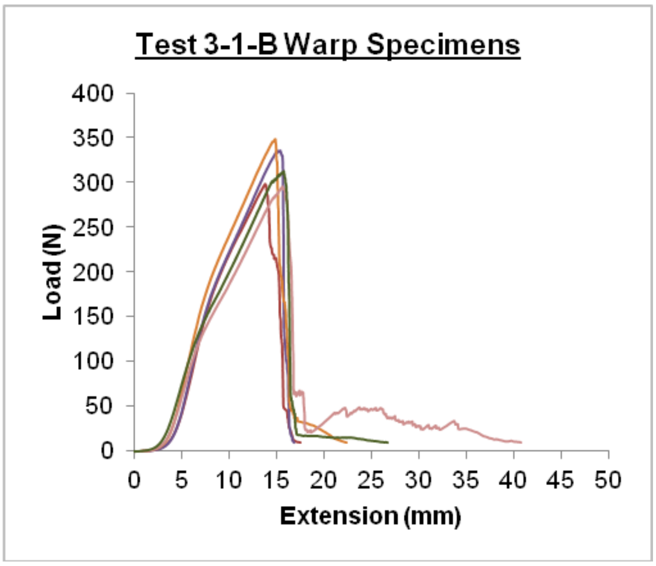


Mean maximum load= 187.66
Mean standard deviation = 7.20

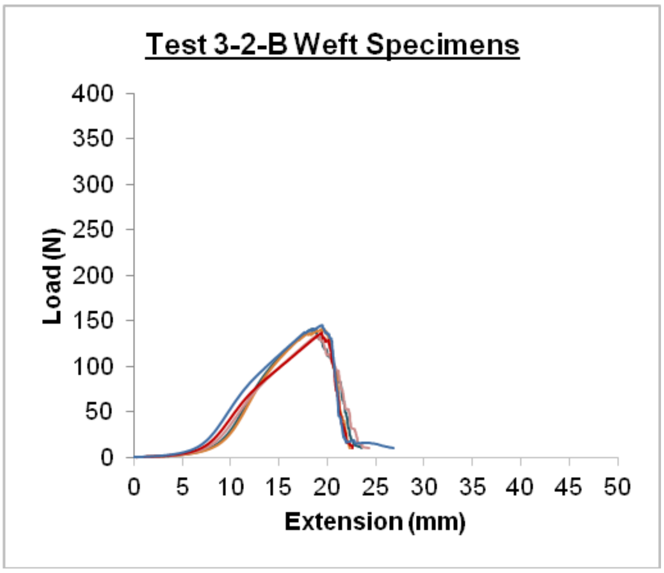
Test Object B: ***Test Group 3***



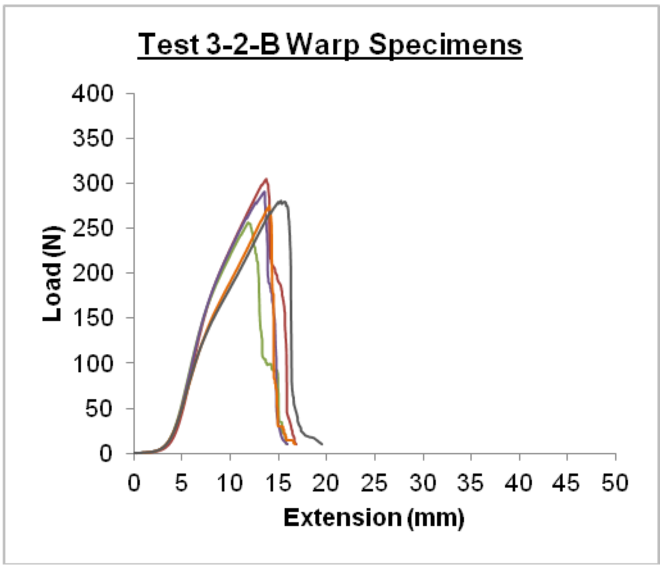
Mean maximum load= 144.11
Mean standard deviation = 9.33



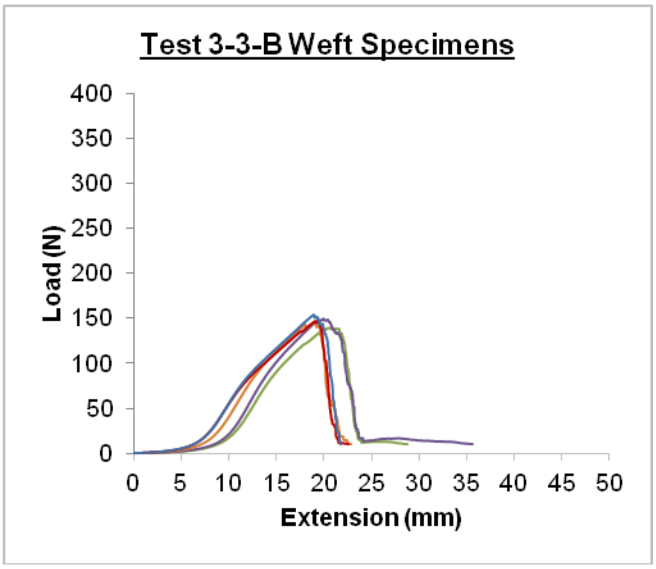
Mean maximum load= 318.20
Mean standard deviation = 22.95



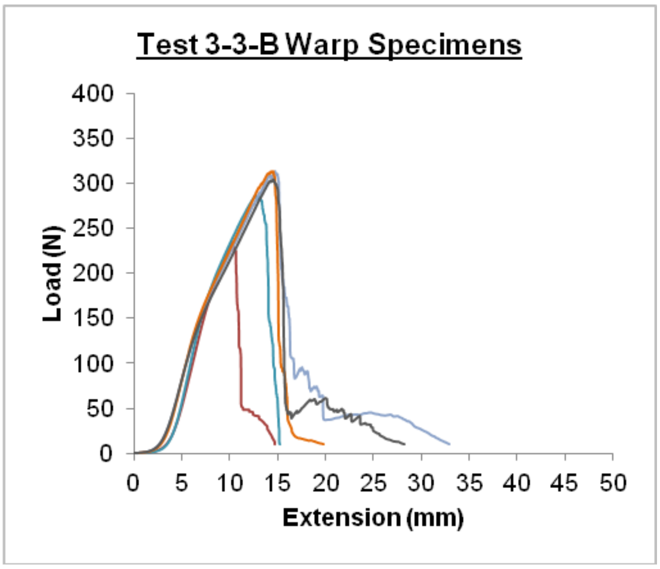
Mean maximum load= 139.58
Mean standard deviation = 4.11



Mean maximum load= 281.32
Mean standard deviation = 18.17

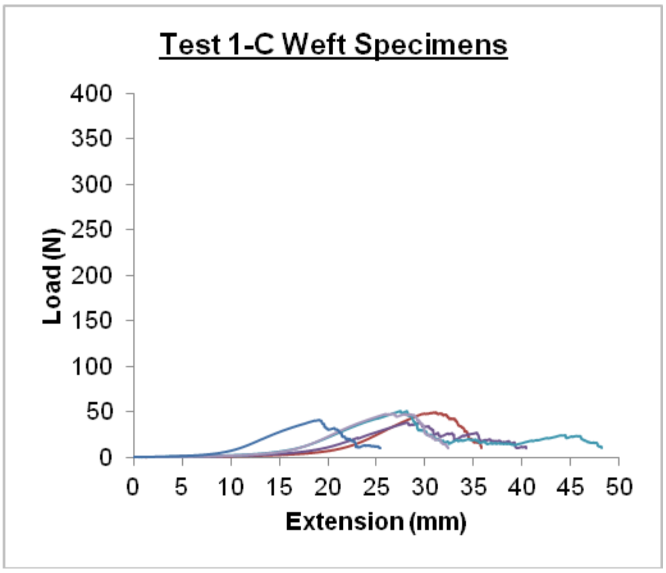


Mean maximum load= 147.72
Mean standard deviation = 5.00

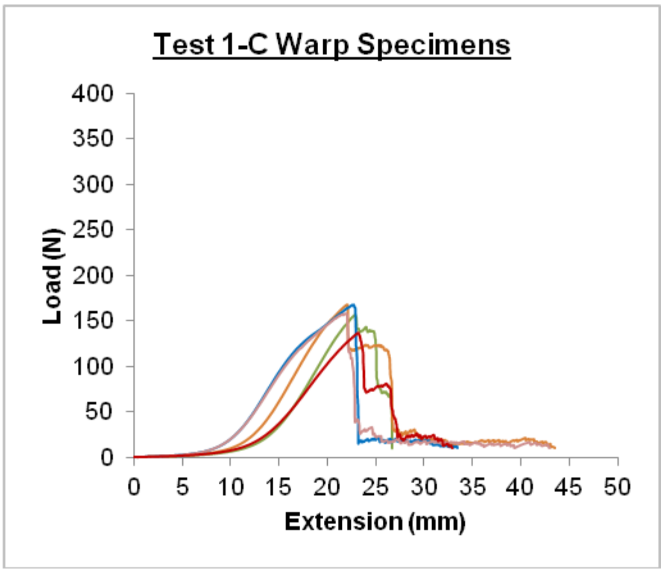


Mean maximum load= 290.57
Mean standard deviation = 32.93

Test Object C: *Test Group 1*

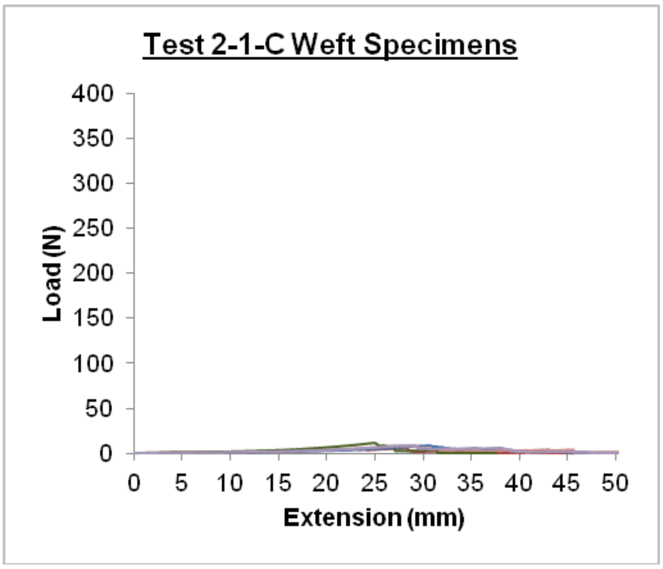

Test 1-C Weft Specimens

Mean maximum load= 45.77
Mean standard deviation = 5.21

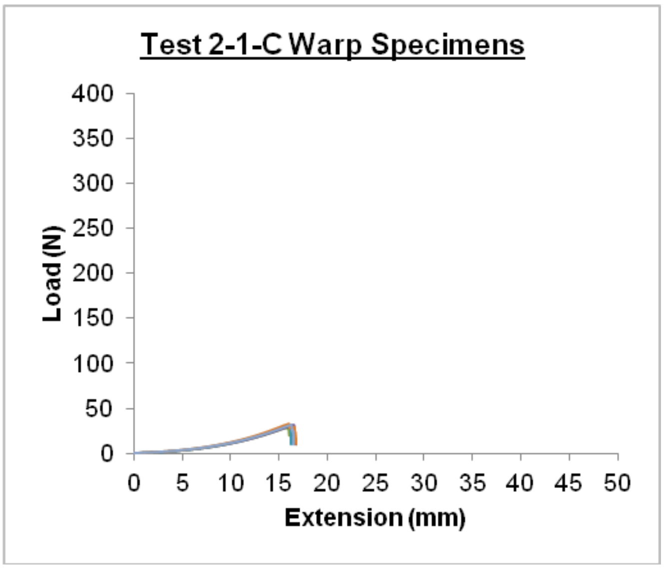

Test 1-C Warp Specimens

Mean maximum load= 157.23
Mean standard deviation = 12.70

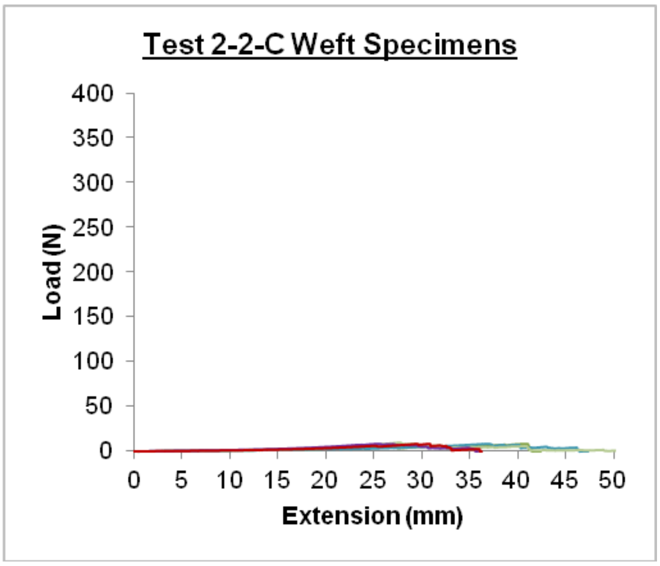
Test Object C: *Test Group 2*


Test 2-1-C Weft Specimens

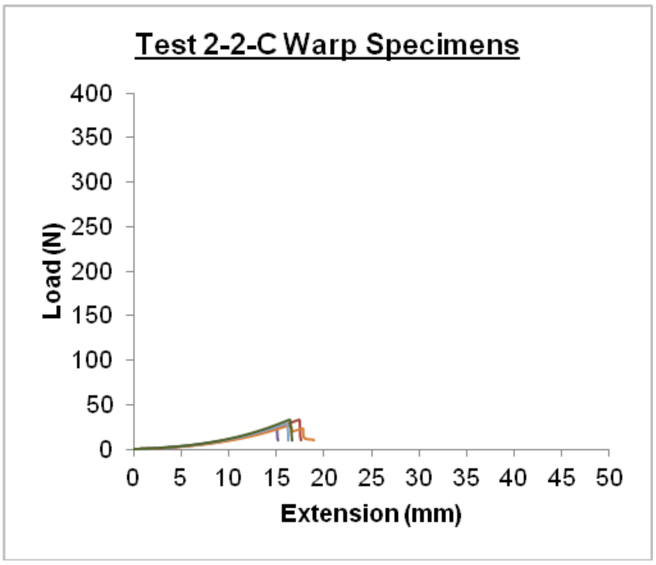
Mean maximum load= 8.86
Mean standard deviation = 2.11


Test 2-1-C Warp Specimens

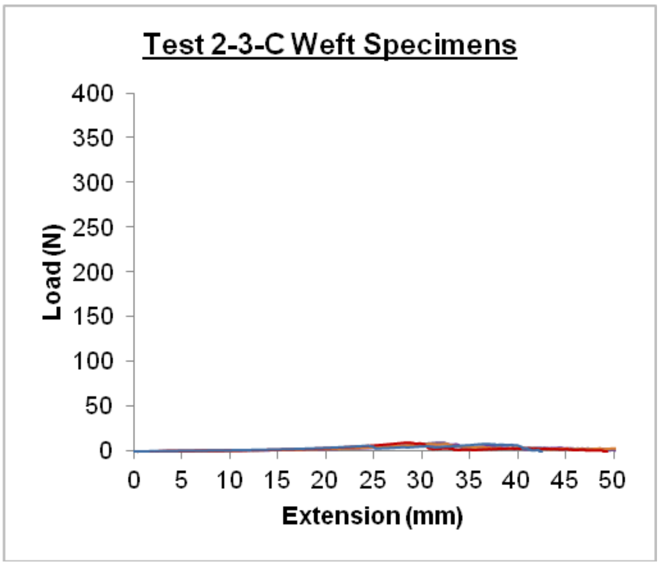
Mean maximum load= 31.10
Mean standard deviation = 1.58



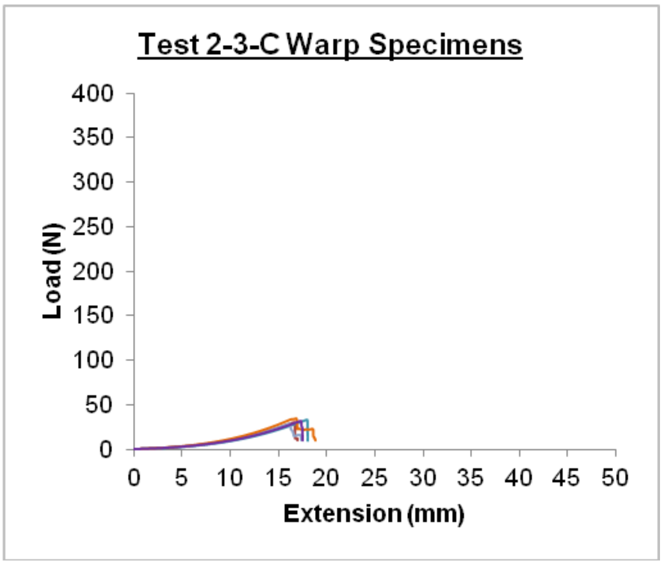
Mean maximum load= 8.62
Mean standard deviation = 0.91



Mean maximum load= 29.54
Mean standard deviation = 3.73

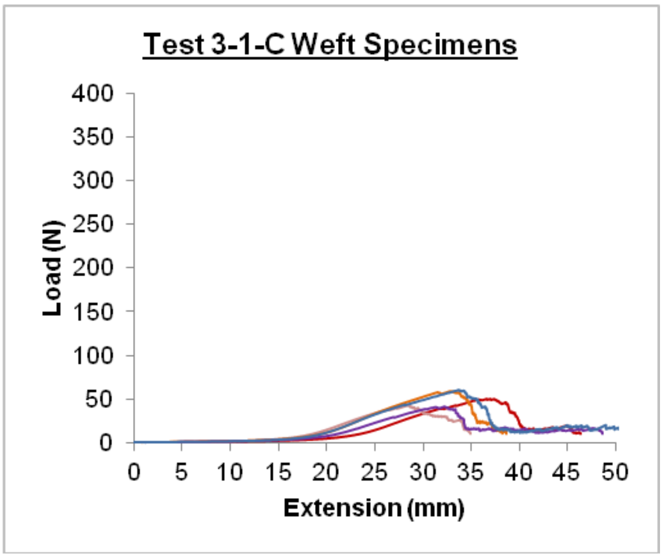


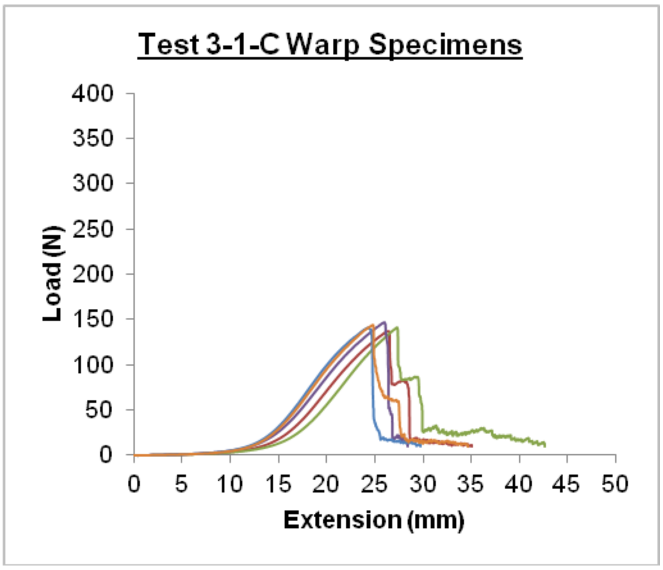
Mean maximum load= 8.61
Mean standard deviation = 0.52



Mean maximum load= 31.91
Mean standard deviation = 2.72

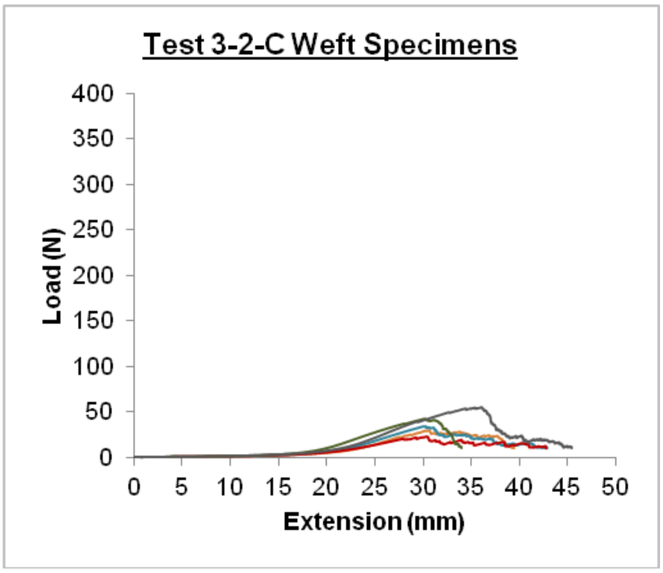
Test Object C: *Test Group 3*

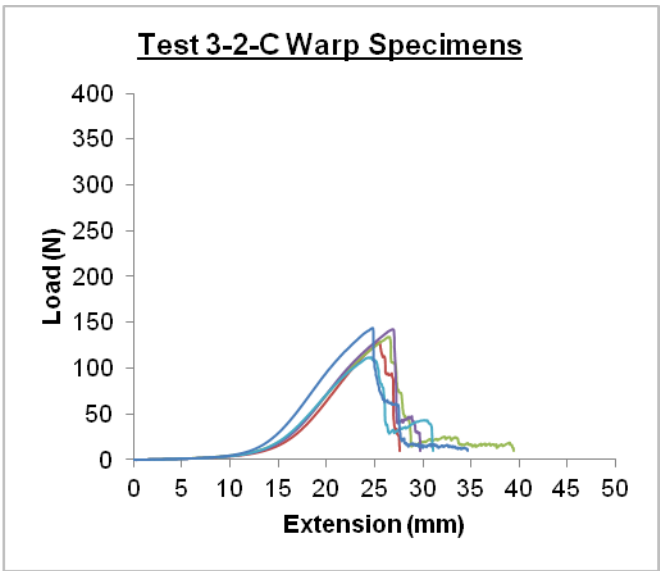




Mean maximum load= 51.01
Mean standard deviation = 8.99

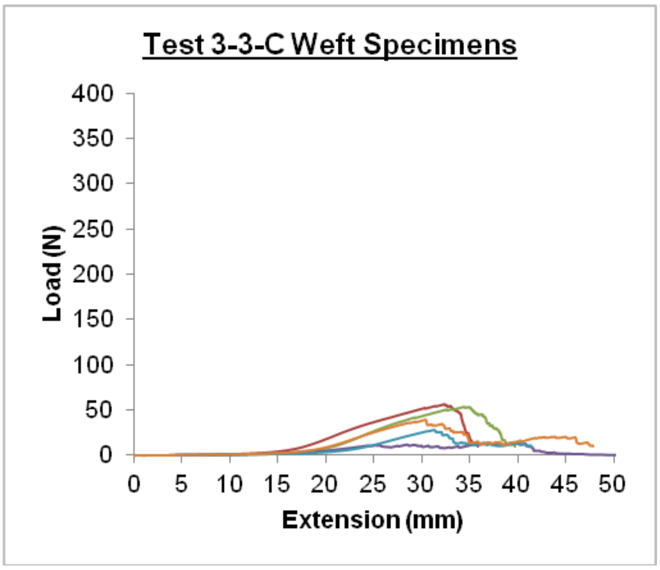
Mean maximum load= 142.04
Mean standard deviation = 3.38



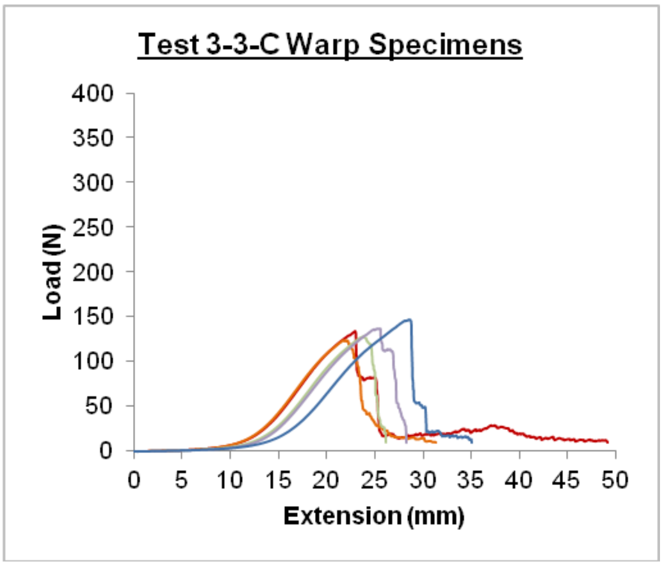


Mean maximum load= 36.99
Mean standard deviation = 12.43

Mean maximum load= 130.56
Mean standard deviation = 11.31



Mean maximum load= 38.65
Mean standard deviation = 17.76



Mean maximum load= 133.87
Mean standard deviation = 9.37

Appendix 8 – 'Student's t-test' Explanation and Calculations

'Student's t-test' Explanation[149]

Student's t-test is used to statistically assess the real differences which are present between the shapes of a pair of bell-shaped graphs, looking at the differences in mean and standard deviation.

The Student's t-test calculation gives a *p* (probability) value. This value is the probability that if the same tests were to be done again, that similar differences between the pair of results would be replicated. The lower the *p* value the more significant the result. If the *p* value falls below any of the following three numerical brackets, it is said to be statistically significant, having a probability ratio of replicating the same differences between the pair of results as outlined next to each:
<0.05 = 1:20
<0.01 = 1:100
<0.001 = 1:1000

If the *p* value is above 0.05, it is said to statistically show no significance, with a low probability that results would be replicated in future tests.

'Student's t-test' Calculations

Student's t-test calculations in Tables 15-18 have been taken using mean and standard deviation figures obtained from the *Instron* devices *Bluehill* software. Calculations have been done using the Student's t-test auto-sum function in *Microsoft Office Excel 2007* software.

Table 15 'Student's t-test', Test Group 1 versus Test Group 2 (dry untreated versus wet treated) Weft Specimens

Test Round Pair	*p* value	*p* =	ratio
Test 1-A / Test 2-1-A	0.0000019398	<0.001	1:1000
Test 1-A / Test 2-2-A	0.0000367153	<0.001	1:1000
Test 1-A / Test 2-3-A	0.0004244927	<0.001	1:1000
Test 1-B / Test 2-1-B	0.0000423986	<0.001	1:1000
Test 1-B / Test 2-2-B	0.0002783704	<0.001	1:1000
Test 1-B / Test 2-3-B	0.0000195268	<0.001	1:1000
Test 1-C / Test 2-1-C	0.0002360276	<0.001	1:1000
Test 1-C / Test 2-2-C	0.0000613994	<0.001	1:1000
Test 1-C / Test 2-3-C	0.0001183442	<0.001	1:1000

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[149] Student's t-test explanation based on personal correspondence with Professor Elizabeth Tanner, University of Glasgow.

Table 16 ‘Student’s t-test’, Test Group 1 versus Test Group 2 (dry untreated versus wet treated) Warp Specimens

Test Round Pair	***p* value**	***p* =**	**ratio**
Test 1-A / Test 2-1-A	0.00583828	<0.01	1:100
Test 1-A / Test 2-2-A	0.0015194662	<0.01	1:100
Test 1-A / Test 2-3-A	0.0016817959	<0.01	1:100
Test 1-B / Test 2-1-B	0.0000003346	<0.001	1:1000
Test 1-B / Test 2-2-B	0.0000467165	<0.001	1:1000
Test 1-B / Test 2-3-B	0.0000587594	<0.001	1:1000
Test 1-C / Test 2-1-C	0.0000274371	<0.001	1:1000
Test 1-C / Test 2-2-C	0.0000583243	<0.001	1:1000
Test 1-C / Test 2-3-C	0.0000286879	<0.001	1:1000

Table 17 ‘Student’s t-test’, Test Group 1 versus Test Group 3 (dry untreated versus dry treated) Weft Specimens

Weft Specimens	***p* value**	***p* =**	**ratio**
Test 1-A / Test 3-1-A	0.5964403710	>0.05	-
Test 1-A / Test 3-2-A	0.1755207707	>0.05	-
Test 1-A / Test 3-3-A	0.0890867817	>0.05	-
Test 1-B / Test 3-1-B	0.0428251673	<0.05	1:20
Test 1-B / Test 3-2-B	0.0138481485	<0.05	1:20
Test 1-B / Test 3-3-B	0.2893198743	>0.05	-
Test 1-C / Test 3-1-C	0.0782971810	>0.05	-
Test 1-C / Test 3-2-C	0.2358354324	>0.05	-
Test 1-C / Test 3-3-C	0.4702216017	>0.05	-

Table 18 Student’s t-test’, Test Group 1 versus Test Group 3 (dry untreated versus dry treated) Warp Specimens

Warp Specimens	***p* value**	***p* =**	**ratio**
Test 1-A / Test 3-1-A	0.534978443	>0.05	-
Test 1-A / Test 3-2-A	0.2901758266	>0.05	-
Test 1-A / Test 3-3-A	0.1960646250	>0.05	-
Test 1-B / Test 3-1-B	0.1305183690	>0.05	-
Test 1-B / Test 3-2-B	0.0085394765	<0.01	1:100
Test 1-B / Test 3-3-B	0.0850495440	>0.05	-
Test 1-C / Test 3-1-C	0.0647168001	>0.05	-
Test 1-C / Test 3-2-C	0.0194948590	<0.05	1:20
Test 1-C / Test 3-3-C	0.0753270717	>0.05	-