An Investigation into Ultrasonic Cleaning Historic Textiles

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Appendix A

Abstract

This dissertation investigated ultrasonic cleaning, an area of conservation research since the 1950s, with publications regarding textiles as recent as August 2018. This research brings together conservation literature, scientific research on sound and ultrasound, and experimental laboratory research with a bespoke ultrasonic device to characterise some of the effects and parameters of ultrasound in a textile wet cleaning environment. The findings of this research provide a body of evidence on the physical and chemical effects of ultrasound and variables that can impact those effects. Primary conclusions are that ultrasound creates a more complex cleaning environment than has previously been explored in publication, and that there are accessible means to characterise the cleaning action of any ultrasonic tool for use in conservation. As a result, this dissertation recommends further research take place in practice with collaborators in the field of ultrasonic cleaning, focusing on impact and control of key variables: frequency, power, cleaning solution, and barrier layers in textile wet cleaning environments. Following from the conclusions and future research suggestions, a case study was completed in Appendix A, comparing cleaning efficacy and damage between sponging techniques and ultrasonic cleaning on historical soiled linen.

Acknowledgements

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To Karen Thompson, I am grateful for her encouragement in pursuing my initial testing, and the introduction to her colleague Dr Mahesh Uttamlal in the Forensic Science Department at Glasgow Caledonian University who performed SEM-EDX analysis for a portion of this research. Valuable insight and imaging were provided by Dr Paul Prentice at the University of Glasgow Cavitation Laboratory which aided this research immensely. Thank you to Dr Margaret Smith for assistance with statistical design and interpretation. Special thanks to my dissertation supervisor Sarah Foskett, who provided insight and critique at all stages of my work, always with true honesty and humour.

Thank you to the Textile Conservation Foundation for the work done to support students and the CTC. To my fellow students at the CTC, I am grateful for the countless discussions and debates, both critical and encouraging. I would like to end these acknowledgments by thanking my husband Edward, who has maintained an endless enthusiasm for both my personal and professional interests, without which the opportunity to take on this research would not have come to pass.

1 Introduction

1.1 Conservation Wet Cleaning

In textile conservation, wet cleaning is presently seen as an interventive treatment, to be undertaken after ethical and contextual consideration of the object and its soiling. Sometimes it is preferable to not wet clean to preserve aspects of the object's current state, such as soiling from use, or creasing indicative of purposeful folds. At other times, a textile is too structurally degraded for wet cleaning, or too damaged to withstand sponging or other mechanical action. However, some objects are determined to be structurally stable and of appropriate context for wet cleaning. This is often a collaborative decision-making process, with curators or owners working with conservators to identify the level of cleaning desired to present the object as it was used or seen at a specific point in an object's biography.[1]

When wet cleaning is determined to be beneficial to both the object's preservation and its interpretation, wash baths are formulated according to the type and level of soiling and staining, as well as the fibre types, dyes, degradation issues, and other materials present.[2] The solvents, surfactants, or other additives for wash baths must be tailored to remove only the "matter out of place," and strive leave all other evidence and materials undisturbed by the process.[3] The threshold between suitably clean and over-clean is different for every object and context. An object may be left less-clean than desired to preserve structural integrity or long-term preservation, and the process is continually monitored to avoid over-cleaning. Typically, mechanical wet cleaning options have been limited to various natural and synthetic sponges, brushes, or gentle agitation of shallow wash baths.

The introduction of ultrasound as an option for use in aqueous or organic solvent cleaning systems has been repeatedly investigated across conservations disciplines (*Chapter 3*). However, characterisation of the actions of ultrasonic cleaning, and how they might be controlled within a conservation context has not been explored. Related information is available in the context of industrial and medical cleaning literature, which is not easily translated to conservation. This technical literature requires significant background in the physics and chemistry of sound, which are not generally areas conservators have experience in. Without a deep understanding of the physical and chemical mechanisms and actions of

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[1] Frances Lennard and Patricia Ewer, "Remedial Conservation," Chapter 4 in *Textile Conservation: Advances in Practice*, ed. Frances Lennard and Patricia Ewer (Oxford: Butterworth-Heinemann, 2010), 141-143.
[2] Ágnes Tímár-Balázsy and Dinah Eastop, *Chemical Principles of Textile Conservation* (Oxford: Butterworth-Heinemann, 1998), 194-241.
[3] Mary M. Brooks and Dinah Eastop, "Matter out of Place: Paradigms for Analyzing Textile Cleaning," *Journal of the American Institute for Conservation (JAIC)* 45, no. 3 (2006): 171–81, accessed 17 June 2018, doi:10.2307/40026689.

ultrasonic cleaning, it has not been a simple task for conservators to practically or ethically evaluate when or under what conditions ultrasonic cleaning could be safely applied.

1.2 Research Aims

The aim of this research was to provide data on the physical and chemical effects of ultrasonic cleaning as related to textile conservation wet cleaning. Evaluating the conservation literature and summarizing current industrial and scientific literature on ultrasonic cleaning aimed to deepen the understanding of ultrasonic cleaning for conservators. The experimental portion of the research aimed to characterise and evaluate the actions of a specific ultrasonic cleaning probe using methods that would allow for evaluation of other ultrasonic devices in practice. The experimental research was completed in two phases:

- The aim of Phase 1 was to provide both quantitative statistically valid data, and visualisation of the effects of ultrasound and the impact of the cleaning environment. A series of experiments aimed to characterise physical and chemical effects of ultrasound with variables of amplitude and cleaning solution. This included temperature and pH monitoring, high-speed high-magnification imaging of the probe, and cavitation detection.
- The aim of Phase 2 was to provide quantitative statistically valid data with reproducible results on the effects of ultrasonic cleaning in a simulated wet cleaning environment to evaluate the impact of amplitude and cleaning solutions on cleaning efficacy and physical damage of a textile.

The research ends with a conclusion identifying key results, and avenues for future research. A series of appendices are included to provide additional relevant information, such as a glossary, health and safety parameters, additional imaging, full data sets, and experimental samples. Further research on applying the data gathered in this dissertation to a historical textile was performed (*Appendix A)* but was outside the scope of the focused research aims of this dissertation.

2 Research Methods

2.1 Research Strategy

This dissertation began with questions regarding the basic science and control of ultrasound:

- How does ultrasonic cleaning work?
- What is ultrasound doing in the cleaning solution, and what impact might that have on historic textiles?
- What level of control can be exerted by a conservator using ultrasound in practice?

Research started with an analysis of the current literature in conservation on ultrasonic cleaning, with a focus on textiles. Extended research was then conducted on the physicochemical nature of sound and current technical literature on ultrasonic cleaning. This secondary research informed the experimental design and was summarised to provide necessary background information for conservators.

Phase 1 of the research included extensive characterisation of the experimental ultrasonic device. Phase 2 evaluated the effects of the ultrasonic device on standard soiled textile samples to provide statistical data set on potential cleaning efficacy and physical damage in a conservation wet cleaning environment.

2.2 Data Collection

Conservation literature research was done from text books, peer-reviewed journals, and other conservation publications and conferences since the 1950s. Literature on the science and technology of ultrasound was limited to the last 30 years of publications, focusing on text books and peer-reviewed journals.

Experimental Phases 1 and 2 used primarily standardized samples, randomized into testing groups of five replicates each. Quantitative and qualitative data and observations were recorded during each test. Detailed methodology and sampling are provided for each experiment.

2.3 Data Analysis

Data was analysed through summary statistics such as counts, means, and standard deviations. Analysis of Variance (ANOVA) was used for one-factor and two-factor statistical analysis of variables. Data visualisation and interpretation was done with graphics such charts with calculated standard deviations, annotated diagrams, photographs, and microscopy with calibrated scale.

2.4 Limitations

Limitations of this study included subject-specific expertise, as the author has no formal training nor specialised knowledge of physics or chemistry of sound, nor ultrasonic cleaning. Where possible, oversight was sought from those working in these fields. Experimental testing was usually limited to standard lab equipment, with small sample numbers, and only utilised one ultrasonic device with certain fixed parameters. Only English-language publications were researched due to language limitations of the author.

3 Literature Review

3.1 Introduction

The purpose of the literature review was to provide an understanding of conservation publications on ultrasonic cleaning of cultural heritage objects. Extended discussion was given to research on textiles and feathers, although materials and contexts outside this scope were included, such as inorganic and organic archaeological material, adhesives, enzymes, and twentieth century art.

Research on ultrasonic cleaning systems was limited to the use of ultrasonic baths and hand-held probes. Not included were ultrasonic dental scalers, humidification, welding, imaging, or other analysis using ultrasound. Scientific background was not covered in this chapter as the large scope of the subject covered a separate body of literature. A summary on the relevant science of sound can be found in *Chapter 4*.

3.2 Early Exploration and Publication

Publications on ultrasonic cleaning cultural heritage objects began in the late 1950s. Articles by conservation science researchers E.T Hall at the Museum of Fine Arts Boston in 1959[4] and R.M. Organ at the British Museum in London, 1960[5] were short introductions to the subject. These early articles focused on archaeological objects, with limited descriptions of cleaning experiments on metal, ceramic, fossils, and bones. The frequency of the ultrasonic bath (20 kHz and 40 kHz respectively), and the cleaning solutions were reported, but technical descriptions of cleaning that are crucial for understanding the process are not. Missing details included power or amplitude, volumes or distances used in ultrasonic baths, cleaning times and temperatures, and detailed descriptions of soiling and object condition.

Understandably, in the convergence of two fledgling fields – conservation science and ultrasonic cleaning, Hall and Organ started with the basics, explaining how ultrasound works, and giving brief notes that focus on the science and engineering of the equipment. Understanding of the mechanism of ultrasonic cleaning through baths or probes were not as well defined, or as understood as they are today. These studies do not provide scientific methodology nor detailed, statistically valid results. However, they did lay the basic groundwork for future research in conservation. The primary findings were that ultrasonic cleaning can result in remarkably cleaner objects, particularly those with heavy particulate

[4] E.T. Hall, "Some Uses of Physics in Archaeology," in *Application of Science in Examination of Works of Art. Proceedings of the Seminar: September 15-18, 1958* (Boston: Museum of Fine Arts, 1959), 179–93.
[5] R.M. Organ, "Treatment Using Ultra-Sonic Vibrations," *Studies in Conservation* 4, no. 1 (1959): 35–38, accessed 21 December 2017, http://www.jstor.org/stable/1504993.

accretions, however damage to surfaces was also identified. Both articles highlighted that the material properties and condition of objects were important factors in ultrasonic cleaning of historic objects.

Research throughout the 1960s was sparse but did include research on both ultrasonic baths and probes, published in a variety of academic, subject-specific publications. Publications continued to be from the archaeological field, regarding objects with heavy soiling accretion, but they did not add significant knowledge to the understanding of the cleaning process or variables. The speed and efficiency of ultrasonic cleaning remained key points in articles by Stevens, Jones, and Todd,[6] and Spier.[7] At the same time, the cleaning mechanism was not further investigated, and in some cases, it was attributed simply to vibration. The experimental design of most articles relied on single case studies, without controls or scientific methodologies, and with little technical analysis or imaging of results.

In the palaeontology department of the British Museum, Macadie experimented with cleaning fossils and bones with an ultrasonic probe (48 kHz).[8] Descriptions given of cavitation were more nuanced, and Macadie notes that optimal conditions occurred with the probe positioned just under the surface of cleaning liquids with best cleaning results in close range to the object, but with no explanations of why. Despite brief articles providing improvements on technique, experimentation remained scattershot in this decade, with little cohesion between research, and little discussion of why positive or negative outcomes occurred. Many more conservators, archaeologists, and conservation scientists continued to publish similar investigations of ultrasound, but the research was widely similar, and not well linked throughout the 1970s.

3.3 Feathers

In the 1980s, several authors experimented with ultrasonic cleaning feathers. These publications highlighted issues of experimentation with ultrasonic cleaning without a nuanced understanding on the science surrounding it. As conservation became more professionalised through the mid-twentieth century, the importance of chemistry took a primary place in conservation education in ways that physics, particularly in terms of sound,

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[6] Calvin Stevens, Daniel Jones, and Robert Todd, "Ultrasonic Vibrations as a Cleaning Agent for Fossils," *Journal of Paleontology* 34, no. 4 (1960): 727–30, accessed 28 March 2017, http://www.jstor.org/stable/3555339.
[7] R.F.G. Spier, "Ultrasonic Cleaning of Artifacts: A Preliminary Consideration," *American Antiquity* 26, no. 3 (1961): 410–14, accessed 28 March 2017, http://www.jstor.org/stable/277407.
[8] C.I. Macadie, "Ultrasonic Probes in Palaeontology," *Journal of the Linnaean Society (Zoology)1* 47, no. 311 (1967): 251–53, accessed 2 June 2018, doi:10.1111/j.1096-3642.1967.tb01408.x.

did not.[9] As in early decades, the research on feathers made use of case studies of historic objects without controls or replicate samples. In this scenario, the complexity of ultrasonic cleaning seemed to be a barrier to deeper research, making it difficult for conservators to analyse results and move forward.

Danish conservators Petersen and Somer-Larsen presented a case study of cleaning feather garments with an ultrasonic device at the 1984 International Council of Museums symposium.[10] The reason for choosing ultrasound was not described, and only a few details were given on the conditions under which ultrasound was used. Terminology such as resonance, frequency, and decibels were used without discussion, and relationships given between these terms were unclear. The ultrasonic device was not well described other than frequency (23 kHz), and the post-print lacked parameters of use that could allow other conservators to obtain similar results. There was no treatment evaluation describing what the ultrasound did that was different from other mechanical action. The description of successful cleaning may have sparked interest in further exploration, but the publication contributed little to understanding why or how ultrasonic cleaning worked, or to what level cleaning and damage were evaluated to determine success.

In 1986 New Zealand conservators Barton and Weik published on cleaning historic feather objects in an ultrasonic bath.[11] This study contained focused research questions and included examination of cleaning efficacy and damage with scanning electron microscopy (SEM). They provided significantly more background and rationale for why ultrasound was tested, and the level of description and detail of times, temperatures, cleaning environment and solutions were an improvement in the literature. The frequency of the device (40-60 kHz) was discussed as non-important variable,[12] and damage seen in the microscopy images was not discussed in terms of the many variables in the study. Despite lack of discussion of the variables and SEM damage, the findings concluded that ultrasound was safe and effective for the cleaning of historic feather garments in ultrasonic baths.

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[9] "Careers in Conservation, Prerequisites," American Institute of Conservation, accessed 27 June 2018, https://www.conservation-us.org/jobs/become-a-conservator/graduate#.W1smg7gVg2w.
[10] K.S. Petersen and A. Sommer-Larsen, "Cleaning of Early Feather Garments from South America and Hawaii," in *ICOM Committee for Conservation 7th Triennial Meeting Copenhagen 10-14 September 1984 Postprints*, ed. Diana de Froment (Copenhagen: ICOM in association with the J. Paul Getty Trust, 1984), 84.3.13-84.3.16.
[11] Gerry Barton and Sabine Weik, "Ultrasonic Cleaning of Ethnographic Featherwork in Aqueous Solutions," *Studies in Conservation* 31, no. 3 (1986): 125–32, accessed 12 March 2018, doi:10.2307/1506258.
[12] Ibid., 126.

Barton and Weik's findings were contradicted in the same year by CCI conservation scientist Gregory Young.[13] Young performed the SEM analysis for Barton and Weik and used the same data and images to support his conclusion that the damage seen on the feathers did not outweigh the benefits of cleaning. A potential relationship between the varied detergent solutions and ultrasound was suggested, and he highlighted the need for research with variables of frequency, depth, and distance but referenced no scientific literature. Ultrasonic cleaning was described as "inadvisable" and "unpredictable"[14] based on his interpretation of the damage seen through SEM. Shortly after, Allyson Rae, conservator at the British Museum, published on cleaning protocols for feathers, specifically stating that ultrasound was not used for feathers, without further comment.[15] This flurry of related experimentation and publication ended there, perhaps in part due to an inability to evaluate the variables and mechanisms in ultrasonic cleaning in ways that allowed for controlled, safe application to historic objects.

Outcomes of both positive cleaning and negative damage were published with careful thought by conservators, and scientists, all looking to inform decision-making on the time-intensive issues of cleaning fragile historic feathers. Yet, the field of conservation was left without any further knowledge of what factors in the ultrasonic cleaning process were most likely to have caused the damage, and with few references to scientific literature to move forward. No discussion or experimentation was done to see if suggested factors could be controlled, and discussion of why or how the ultrasonic cleaning caused damage was scant. This, combined with lack of study controls to compare damage to traditional cleaning methods resulted in a difficulty analysing acceptable levels of damage or change from ultrasonic cleaning.

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[13] Gregory Young, "Disruption of Feather Structure by Ultrasonic Cleaning in Aqueous Detergent Baths," in *Symposium 86: The Care and Preservation of Ethnological Materials*, ed. JC McCawley and T. Stone. Edited by R. Barclay, M. Gilber (Ottowa: Canadian Conservation Institute (CCI), 1986), 37–43.
[14] Ibid., 42.
[15] Allyson Rae, "Cleaning of Featherwork," in *Recent Advances in the Conservation and Analysis of Artifacts, Jubilee Conservation Conference University of London Institute of Archaeology*, ed. James Black (London: Summer Schools Press, 1987), 243–48.

3.4 Textiles

Until new publications in August 2018, only one study was published in English on ultrasonic cleaning historical textiles using an ultrasonic bath or probe. In 1989, textile scientist William Cooke and V&A head conservator Linda Hillyer[16] published preliminary research with historic linen tapestry backing fabrics. Cleaning efficacy and damage of an ultrasonic bath was explored using several types of analyses. The interdisciplinary knowledge base of the two authors provided a promising platform for research. However, without specialised knowledge or oversight on the science of sound, some information was misleading. The cavitation phenomenon of ultrasound was described as occurring only at the "liquid/solid interface,"[17] and the relationships given between ultrasonic frequency to harmonics and decibels were unclear as no ultrasonic device parameters or analytical measurements of the cleaning system were provided.[18] Scientific literature on ultrasound was mentioned, but without specific references, leaving readers with little idea of where to search for more literature. This continued to underscore the complexity of the science of sound, and lack of fluency in the subject.

Overall, Cooke's study would have benefitted from more controls. There was no comparison of cleaning or damage to traditional sponging techniques, or to compare to fibre swelling in aqueous solutions without additional mechanical action. Common controls used in conservation were not used, such as temperature (reported range from 20-40 °C). Loose fibres and yarns were not secured or prevented from unravelling as would be done before any conservation cleaning. The results of the study did not include consideration of the ultrasonic device variables such as frequency (40 kHz) on cleaning efficacy or damage. Given this, Cooke's finding that ultrasound should not be used to clean historic cellulosic textiles in aqueous solutions was not fully substantiated by the data provided.[19] Much like the research on feathers with similar findings of both high cleaning efficacy and physical damage, the research was not pushed further to understand and evaluate the variables at play.

3.4.1 Current Research

Two case studies published by the V&A just prior to this dissertation provided new insight into ultrasonic cleaning historic textiles. The inclusion of probe distance to the object, and direct comparisons to sponging treatment were

[16] W. D. Cooke, "A Pilot Study in the Use of Ultrasonic Cleaning in Textile Conservation," *The Conservator* 13, no. 1 (1989): 41–48, accessed 17 November 2017, doi:10.1080/01410096.1989.9995046.
[17] Ibid., 41.
[18] Ibid., 47.
[19] Ibid., 47-48.

particularly valuable for the conservation literature.[20] Collaboration with conservation graduate students at UCL provided SEM analysis of ultrasonic cleaning on a variety of textiles. The research explored proteinaceous as well as cellulosic fibres and dyed textiles, and methods of controlling textile movement and ultrasonic cleaning using barriers,[21] much of which covered new ground in the literature with short, focused publications of case studies with some controls and comparisons of different treatments.

3.5 Cumulative Research

Investigation into refining ultrasonic cleaning and controlling effects has remained limited, with some recent developments. Brief mention of ultrasonic cleaning continued to crop up in conservation case studies, often for archaeological materials, through the twenty-first century. The use of an ultrasonic scaler with a liquid coupling media to treat marine archaeological rubber,[22] and general use of ultrasonic baths for marine archaeological cleaning[23] showed that the technology is still in use to some extent in the present day, but perhaps not widely used or accepted within conservation. This pattern of use for heavy particulate accretion is likely to continue as time-efficiency and high level of cleaning seen remains alluring for objects with difficult cleaning issues.

Archaeologist and conservator Niccolo Caldararo's publications were an exception to the one-off case study pattern seen in ultrasonic research over time. Caldararo has researched ultrasound over several decades and has collaborated editorially with early researcher R.M. Organ. The extensive bibliography and nuanced descriptions of scientific concepts provided by Caldararo for archaeological and paper conservation were unique resources in the literature. These publications were paired with discussions of conservation

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[20] Joanne Hackett, "Application of Ultrasonic Cleaning for Historic Textiles: Initial Trials and Treatments," Victoria & Albert Museum Conservation Journal 65 (2018), accessed 9 August 2018, https://www.vam.ac.uk/blog/caring-for-our-collections/application-of-ultrasonic-cleaning-for-historic-textiles-initial-trials-and-treatments.

[21] Gabrielle Crowther, Stefani Cavazos, and Netanya Schiff, "Application of Ultrasonic Cleaning for Historic Textiles: Micro-Analysis of Potential Damage to Textile Fibres Using Scanning Electron Microscopy." Victoria & Albert Museum Conservation Journal 65 (2018), accessed 9 August 2018, https://www.vam.ac.uk/blog/caring-for-our-collections/application-of-ultrasonic-cleaning-for-historic-textiles-micro-analysis-of-potential-damage-to-textile-fibres-using-scanning-electron-microscopy.

[22] Susanne Grieve, "The Excavation, Conservation, Storage, and Display of Rubber Artifacts Recovered from the USS Monitor (1862)," *JAIC* 47, no. 2 (2008): 139–48, accessed 8 March 2018, http://www.jstor.org/stable/27784633.

[23] D. Hamilton, *Methods of Conserving Underwater Archaeological Material Culture,* (Washington DC: Texas A&M University and the U.S. Department of Defense, 1999), 43, accessed 20 February 2018, http://nautarch.tamu.edu/CRL/conservationmanual/ConservationManual.pdf.

cleaning ethics, and collegial discussions with conservators and archaeologists from 1992[24] to 2005,[25] which provided useful resources focusing ultrasonic cleaning on modern conservation practice. The effects of Caldararo's work, where understanding the details on the actions and consequences of ultrasound were key, may be urging new research, particularly in paper.

Conservation scientist Bartl,[26] and conservators Hummert and Pataki-Hundt[27] both published articles on ultrasonic cleaning of paper over the last decade. Bartl examined the use of ultrasound to improve enzyme treatments on paper. This study utilised standardized samples, instead of the common historical-object case study seen in the literature. Drawing extensively on conservation and industrial literature on the subjects, Bartl improved on past articles by using controls and replicate samples, with more descriptive parameters of the cleaning environment that would allow for some reproduction of the testing parameters by others.

Hummert and Pataki-Hundt used the case study model but performed initial cleaning tests on a near-ruined work of art on paper before proceeding to treatment on a larger collection of items. This process determined detailed parameters of treatment with ultrasound for a collection of badly soiled and stained works on paper for which standard cleaning approaches were unsuccessful. Like Bartl, this article did not deeply explore the cleaning mechanisms of ultrasound but did provide significantly more focused research with well-described cleaning issues, tests, descriptions of environment, and largely substantiated, reproducible results and findings.

Research into ultrasound as a tool in conservation labs has also started to be published with similar rigour, adding to the understanding of ultrasonic effects on materials. Chemist Hems, and archival conservator Curtis published a controlled, well-cited article with substantiated findings on using an ultrasonic bath to reduce yellowing in the preparation of isinglass adhesive. This interdisciplinary work provided relevant insight into the action of

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[24] Niccolo Caldararo, "Some Effects of the Use of Ultrasonic Devices in Conservation and the Question of Standards for Cleaning Objects," *North American Archaeologist* 14, no. 4 (1993): 289–302.
[25] Niccolo Caldararo, "Effects of Cleaning and Regard for Cleaning Goals: Eleven Years Later," in *AIC Objects Specialty Group Postprints*, ed. Virginia Greene and Patricia Griffin, vol. 12 (Portland, Oregon: The American Institute for Conservation of Historic and Artistic Works, 2005), 126–53.
[26] Benjamin Bartl et al., "Application of α-Amylase in Combination with Ultrasound to Remove Starch Based Adhesives from Paper," *Restaurator* 31, no. 2 (2010): 60–80, accessed 17 January 2018, doi:10.1515/rest.2010.005.
[27] Eva Hummert and Andrea Pataki-Hundt, "Ultrasonic Cleaning of Mud Encrustations from Flood Damaged Woodcuts," *Restaurator* 31, no. 1 (2010): 65–74, accessed 3 June 2018, doi:10.1515/rest.2010.004.

ultrasound on fats, proteins, and yellow compounds in different solutions,[28] which is of interest to object treatment as well.

3.6 Conclusion

Overall the conservation literature showed a limited understanding of the variables that impact the physicochemical action of ultrasound that cause cleaning or damage. This indicated that conservators needed a better understanding of the science of sound to pursue more refined research. Common understanding of key concepts and critical parameters of ultrasonic cleaning would allow for more robust analysis of the results of ultrasonic cleaning experiments.

Case studies and experimental research provided numerous preliminary looks at ultrasonic cleaning in conservation but were limited for textiles. The beginnings of cumulative research seen in paper conservation are a promising area for shared research with textile conservation. Future experiments that provide details of the cleaning environment and device parameters using widely understood terminology will add significantly to the literature. Research that deepens the understanding of the physical and chemical forces at play within an ultrasonic cleaning environment is needed. Combining both historic case studies and controlled experiments with statistically valid data sets will further contribute to the understanding of how, why, and when ultrasonic cleaning is beneficial to remove soiling from historic textiles, and how to prevent overcleaning or damage.

[28] Edward Hems and Antoinette Curtis, "Decolourising Isinglass Derived from Aqua-Farmed Sturgeon by Sonication," *Journal of the Institute of Conservation* 38, no. 2 (2015): 188–99, accessed 8 June 2018, doi:10.1080/19455224.2015.1068199.

4 Background: Ultrasonic Cleaning

4.1 Introduction

As discussed in *Chapter 3*, it was clear that conservators were not deeply familiar with the terminology and science of sound, and this may have hampered research. To improve understanding and increase research into conservation applications of ultrasound, more information about the physicochemical actions of ultrasound aimed at conservators was necessary. A short, illustrated introduction to sound as it relates to ultrasonic cleaning is found in this section. A glossary of terms can be found in *Appendix B*.

4.2 Overview of the Units and Properties of Sound

Sound is capable of travel through solid, liquid, and gaseous media. Sound moves as a wave, of which there are two main types: transverse and longitudinal (*Figure 1*). Both types of waves can travel through solids, but generally longitudinal waves travel through liquids and gases. Sound is often depicted as a transverse wave, where the distance between one peak and the next peak of the wave gives the unit measurement of wavelength. In longitudinal waves, wavelength is similarly measured, but through measuring the length of one pressure cycle.[29]

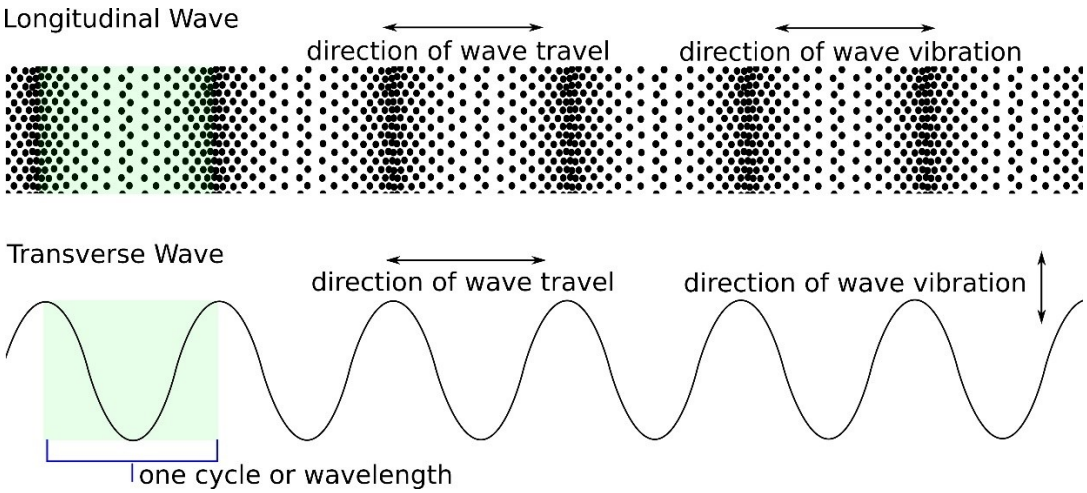

Figure 1 Diagram of a longitudinal wave and a transverse wave showing the difference between direction of travel and direction of vibration.

[29] Thomas Rossing, F. Richard Moore, and Paul Wheeler, *The Science of Sound* (London: Addison Wesley, 2002), 40-41.

The number of wavelengths per second is known as the frequency, given in Hertz (Hz). The human ear can hear frequencies within 20 Hz to 20,000 Hz (20 kHz). Ultrasound is a range of sound frequency above human hearing, starting at 20 kHz. Ultrasound in the range of 20 kHz to 150 kHz is inaudible to humans, but audible to some animals, such as bats and porpoises. Sound frequencies below the audible human hearing 20 Hz are known as infrasound and are utilized for communication by elephants and giraffes[30] (*Figure 2*).

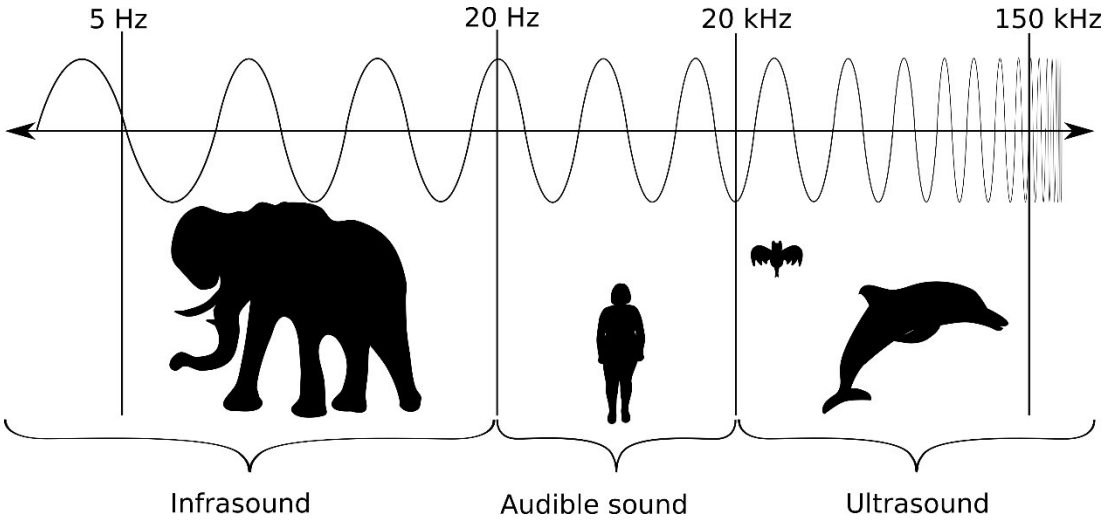

Figure 2 Diagram of frequency ranges of audible sound for humans and some animals.

High-pressure areas of a wave are known as compression, and low-pressure areas are known as rarefaction. This is heard in the human auditory range as sound and can also be felt as vibration. Ultrasonic waves are inaudible to humans, but the vibration and pressure still effect the media they pass through, including liquids, solids, and gasses. The alternating high and low pressure in the range of ultrasound are the primary source of the physical and chemical effects that are utilized in cleaning, usually in the frequency range of 20-60 kHz (*Figure 3*). Medical applications of ultrasound are generally used at much higher frequencies, starting around 1 megahertz (MHz).[31]

[30] Ibid., 3-59.
[31] Ibid., 12-14, 18.

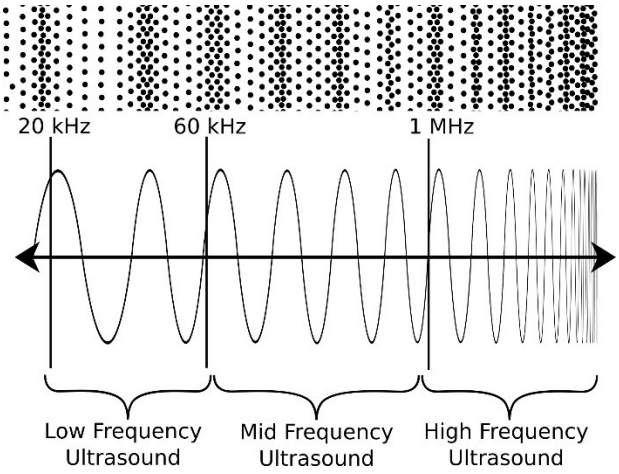

Figure 3 Diagram of frequency ranges of ultrasound, depicted as longitudinal and transverse waves.

In addition to frequency and wavelength, sound can be measured in terms of amplitude, power, speed, and other variables. These units are not related to frequency, but also contribute to the physical and chemical effects of ultrasound. The density and flexibility of the solid, liquid or gas through which the sound wave moves determines the speed of sound, measured in units of distance over time (meters/second), and can also affect the amplitude of the wave. The amplitude of the sound wave is measured in units of distance, measuring the maximum displacement of a substance by the wave, which is related to the power of the wave. A high amplitude transverse or longitudinal wave has higher energy to move a substance a farther distance[32] (*Figure 4*).

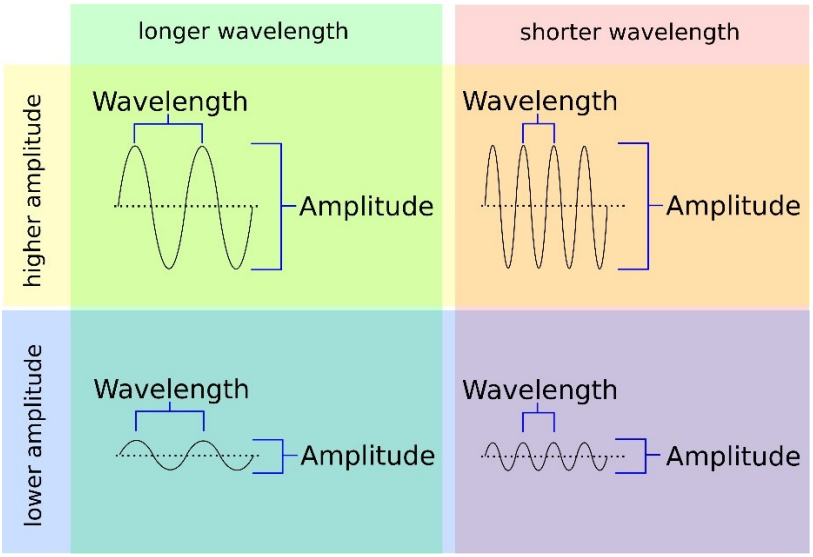

Figure 4 Diagram of transverse sound waves at different amplitudes and wavelengths.

[32] Ibid., 10-12, 15-18, 24.

Sound is highly directional, often moving in a spherical or conical shape, continuing in the direction of the wave propagation until it is reflected, transmitted, or diffracted by a change in medium, such as an object or from liquid to solid. Sound also loses energy or amplitude as it travels farther from its source. The rate of change in amplitude, and loss of energy are dependent on the properties of the media it travels through.[33]

4.3 The Physical and Chemical Effects of Sound

Within conservation and collections care, the physical vibratory effects of sound are known to be an agent of potential damage. Vibration is well understood for objects requiring padding to dampen vibration during travel, or for storage or display in earthquake prone areas. There is widespread understanding that inflexible materials like embrittled glass are more prone to damage from vibration compared to more flexible materials made of supple organic polymers.[34] However, a deeper understanding of sound is required for using ultrasound to clean an object.

Sound can have physical and chemical effects on the medium it flows through. The physical and chemical effects of ultrasound are complex, interrelated, and continually researched within scientific literature. In the field of ultrasound, understanding and harnessing physicochemical effects are the subject of dedicated journals for research[35] where ultrasonic baths and probes are used to create physical changes, and catalyse chemical and biological reactions. The capabilities of ultrasound as a cleaning agent in liquid media rely on the frequency, amplitude, power, speed, volumes, and distances of the ultrasonic energy involved. The source of much of the cleaning action delivered by ultrasound is a phenomenon known as acoustic cavitation.[36]

4.4 Cavitation

Cavitation occurs due to the alternating pressure cycles of compression and rarefaction in longitudinal ultrasonic waves as they pass through liquid media. These pressure cycles can cause the nucleation of small cavitation bubbles of water vapour and gasses. As the pressure cycles continue, bubbles grow during periods of rarefaction (low

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[33] Shigemi Saito, "Ultrasound Field and Bubbles," in *Sonochemistry and the Acoustic Bubble,* ed. Franz Grieser et al., (Oxford: Elsevier Inc., 2015), 11-29, accessed 13 March 2018, doi:10.1016/B978-0-12-801530-8.00002-5.

[34] Paul Marcon and CCI, "Physical Forces," *10 Agents of Deterioration*, accessed 28 July 2018, https://www.canada.ca/en/conservation-institute/services/agents-deterioration/physical-forces.html#vibration4.

[35] *Ultrasonics*, https://www.journals.elsevier.com/ultrasonics; *Ultrasonics Sonochemistry*, https://www.journals.elsevier.com/ultrasonics-sonochemistry.

[36] F.J. Fuchs, "Ultrasonic Cleaning and Washing of Surfaces," in *Power Ultrasonics* ed. Juan Gallego-Juárez and Karl Graff (Elsevier, 2015), 581–586, accessed 22 June 2018, doi:10.1016/B978-1-78242-028-6.00019-3.

pressure) and are forced to a smaller size during compression (high pressure). When bubbles grow to a size at which they can no longer stand the pressure changes, they can violently implode (*Figure 5*). This release of energy can result in localized temperatures of up to 10,000°C, and pressures that exceed 500 bar of atmospheric pressure. When cavitation bubbles collapse, pressure or shock waves are generated, and microjets of liquid stream from the collapse, carrying particles and solutes at high speeds.[37]

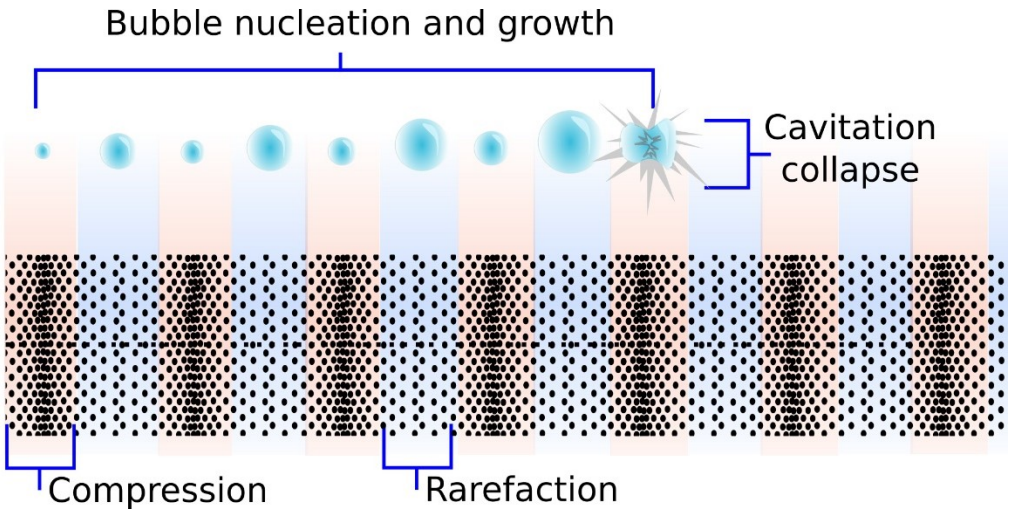

Figure 5 Diagram outlining bubble nucleation, and growth through cycles of high and low pressure, leading to cavitation collapse.

Cavitation occurs at a minute scale – a few hundred nanometres for a fraction of a second. However, it also happens continually when ultrasound flows through liquids with the correct conditions. The size, distribution, and movement of cavitation bubbles, and the rate at which they are formed, grow, and collapse is determined by many factors including:

- frequency and amplitude of the ultrasound
- properties of the liquid medium such as viscosity, density, and vapour pressure
- quantity of favourable particles or molecules within the solution for nucleation of cavitation bubbles to occur[38]

The extreme forces of cavitation collapse provide a physical source of cleaning through rapid changes in pressure, movement, and temperature within the liquid media. The material properties of a historic textile must be able to withstand these strong forces to undergo cleaning with ultrasound. Properties such as wet strength, tensile strength,

[37] Christopher E. Brennen, *Cavitation and Bubble Dynamics* (New York, NY: Cambridge University Press, 2014). 59-73.
[38] Ibid., 15-20.

flexibility, yarn and weave structure, surface finishing, and other factors must be evaluated. The physical impact of the liberation and high-speed movement of particulate and solute material during the cleaning process must also be considered.

In cleaning applications, ultrasound is often applied at the lower-end of the ultrasonic range (20-60 kHz), although up to several hundred kHz is also used.[39] Historical objects are often less robust and structurally stable than similarly constructed new textiles, thus it is important to understand the relationship between cavitation, frequency, potential energy, and physical force. The relationship between frequency and cavitation is inverse: as frequency increases, the forces of cavitation collapse decrease.[40] Frequencies closer to 20 kHz are characterised by larger size of individual bubbles (but lower numbers of bubbles), which creates higher potential energy during collapse, leading to stronger collapse forces per bubble. Comparatively, higher frequencies are characterised by smaller size bubbles (but higher numbers of bubbles). The small size of each bubble results in lower-energy cavitation collapse per bubble. This relationship generally holds true in low-frequency ultrasound used in cleaning applications, although cavitation is greatly influenced by physical factors in the specific liquid environment and vessel.[41]

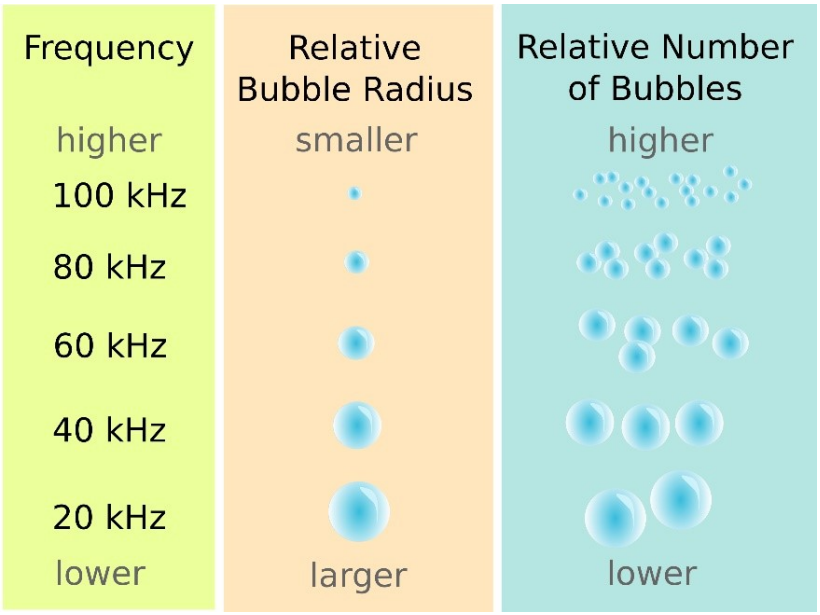

Figure 6 Diagram of the relative relationship of frequency, cavitation bubble radius, and number of bubbles. No scale or direct numerical relationships are displayed.

[39] Fuchs, "Ultrasonic Cleaning," 581.
[40] Ibid., 577–609.
[41] Ibid., 583.; Saito, *Ultrasound Field and Bubbles,* 11-29.

4.5 Sonochemical Reactions in Water

Cavitation collapse can catalyse or accelerate chemical reactions.[42] The pressure threshold for cavitation collapse, and initiation of chemical reactions is similar in distilled water at low-frequency-range ultrasound.[43] The presence and effects of cavitation collapse have been recorded with both pulsed and continuous ultrasonic frequency application at a variety of frequencies (20-100 kHz) and power levels, in both ultrasonic baths and probes.[44]

High energy levels released in cavitation collapse can cause hydrolysis of water, liberating hydrogen (H^+) and hydroxyl (OH^-) free radicals. Hydroxyl radicals can further react to form hydrogen peroxide (H_2O_2), a strong bleaching agent that initiates oxidative reactions.[45] Other reaction products have also been observed or theorised in ultrasonic baths of water alone (*Figure 7*).[46] Sonochemical reactions are the subject of extensive current research. Frequency, power, amplitude, liquid volume, and other parameters of the cleaning environment are currently understood to play a part in sonochemistry.[47] No investigations into the prevalence or detection of sonochemical activity in conservation applications were found in the literature, but potential free radicals and bleaching agents represent the potential for significant, uncontrolled conditions for historic textiles.

$$H_2O \longleftrightarrow H^+ + OH^-$$
$$H^+ + H^+ \longleftrightarrow H_2$$
$$OH^- + OH^- \longleftrightarrow H_2O_2$$
$$H^+ + O_2 \longleftrightarrow HO_2^+$$
$$H^+ + HO_2 \longleftrightarrow H_2O_2$$
$$HO_2^+ + HO_2^+ \longleftrightarrow H_2O_2 + O_2$$
$$H_2O^+ + OH^- \longleftrightarrow H_2O_2 + H^+$$

Figure 7 Chemical reactions that have been observed or theorised to occur in ultrasonic baths with water.

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[42] Georges Chahine et al., "Modeling of Surface Cleaning by Cavitation Bubble Dynamics and Collapse," *Ultrasonics Sonochemistry* 29 (2016): 528–49, accessed 7 April 2018, doi:10.1016/j.ultsonch.2015.04.026.
[43] Tam Thanh Nguyen et al., "Dependence of Cavitation, Chemical Effect, and Mechanical Effect Thresholds on Ultrasonic Frequency," *Ultrasonics Sonochemistry* 39 (1 November 2017): 301–6, accessed 24 July 2018, doi:10.1016/J.ULTSONCH.2017.04.037.
[44] A. Henglein, "Chemical Effects of Continuous and Pulsed Ultrasound in Aqueous Solutions," *Ultrasonics Sonochemistry* 2, no. 2 (1995): 115–21, accessed 12 March 2018, doi:10.1016/1350-4177(95)00022-X.
[45] N.H. Ince et al., "Ultrasound as a Catalyzer of Aqueous Reaction Systems: The State of the Art and Environmental Applications," *Applied Catalysis B: Environmental* 29 (2001): 167–76, accessed 12 March 2018, doi:10.1016/S0926-3373(00)00224-1.
[46] Richard James Wood, Judy Lee, and Madeleine Bussemaker, "A Parametric Review of Sonochemistry: Control and Augmentation of Sonochemical Activity in Aqueous Solutions," *Ultrasonics Sonochemistry* 38 (2017): 354, accessed 27 July 2018, doi:10.1016/J.ULTSONCH.2017.03.030.
[47] Ibid., 351–70.

The high energy of cavitation collapse can also catalyse reactions with solutes in the water, including gasses, salts, acids, dyes, or other soluble materials.[48] The addition of buffers, chelating agents, enzymes, or even traditional surfactants in wet-cleaning formulations create complex chemical solutions to react with ultrasound. The textile and the soiling will also bring further chemical complexity as acids, salts, lipids, and other molecules move into the solution. Therefore, the purity of the water, chemical composition of cleaning solutions, and types of soiling released during the cleaning process are critical for understanding the chemical processes possible in ultrasonic cleaning.

4.6 Conclusion

Ultrasonic cleaning is rooted in the basic science of sound. The relationships between terminology and concepts used in discussing sound are crucial to understanding the impact of ultrasound on gases, liquids, and solids. This knowledge is required to begin understanding the complex variables of ultrasonic cleaning equipment, and the reciprocal impact between the equipment and the cleaning environment. With this research into current scientific research of ultrasound, several parameters appeared crucial to understanding and controlling ultrasonic cleaning:

- frequency
- amplitude and power
- volumes, depths, and distances of the cleaning environment
- temperature, pressure, and other physical properties of cleaning liquids
- cavitation and sonochemical potential of the cleaning system

These parameters were seen to be poorly reported, or not highlighted as significant factors in the results of cleaning or damage when the conservation literature was analysed. Thus, the two experimental laboratory phases of this dissertation focused on recording, quantifying, controlling, and analysing these factors. Experiments were generally restricted to equipment and scenarios common in textile conservation to provide maximum impact for future research in practice and explored only the variables of a single bespoke ultrasonic probe.

[48] Yogesh Karnjkar et al., "Degradation of Magenta Dye Using Different Approaches Based on Ultrasonic and Ultraviolet Irradiations: Comparison of Effectiveness and Effect of Additives for Intensification," *Ultrasonics Sonochemistry* 27 (2015): 117–24, accessed 12 March 2018, doi:10.1016/j.ultsonch.2015.05.011.

5 The Ultrasonic Device

5.1 Introduction

The experimental ultrasonic device used in this dissertation was developed at the British engineering company HDS Ultrasonics Ltd., London by Harry Singh. The device was loaned by V&A for this research (*Figure 8*).

Figure 8 Ultrasonic device loaned from the V&A, designed and produced by HDS Ultrasonics Ltd.

5.2 Ultrasonic Device Parameters

The device consists of two components. A control unit contains the electromechanical components of the device (*Figure 9*), and a hand-held probe contains a piezoelectric transducer (*Figure 10*). Ultrasonic probes are also referred to as sonotrodes or horns. This probe can be used by hand, or on a fixed stand. The probe was provided with a round 5 mm diameter stainless steel, flat-bottom tip, which can be changed to other sizes and shapes by the manufacturer. The device is controlled and operated with a front panel interface. Additional features and parameters are outlined below.[49]

[49] Device parameters provided through personal communication with Harry Singh, email and phone communications, May-June 2018.

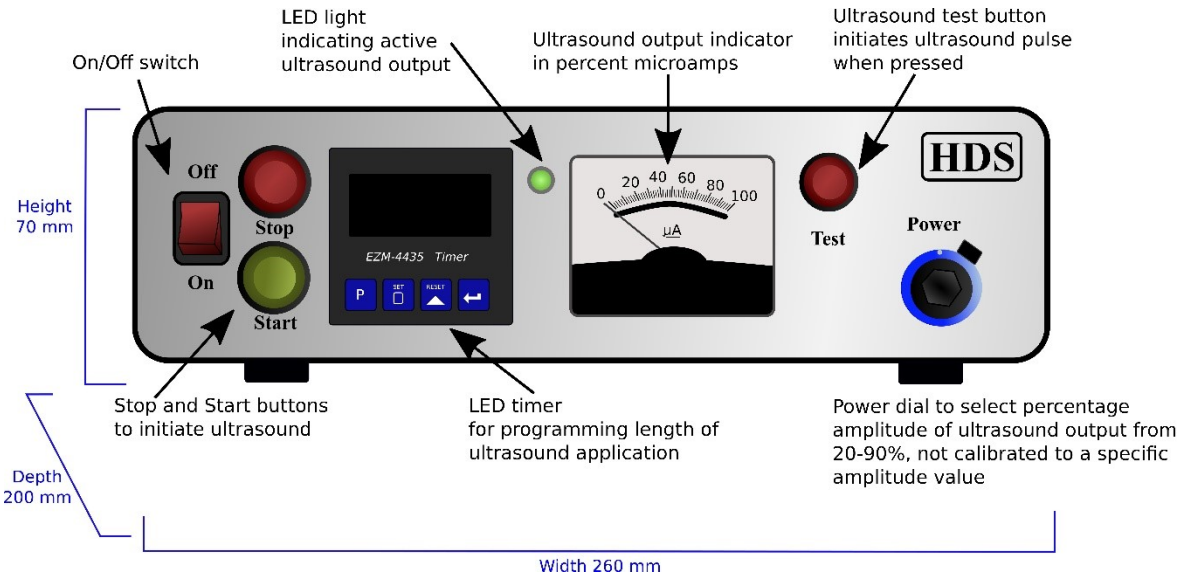

Figure 9 Diagram of ultrasonic tool control unit with annotated features, not to exact scale.

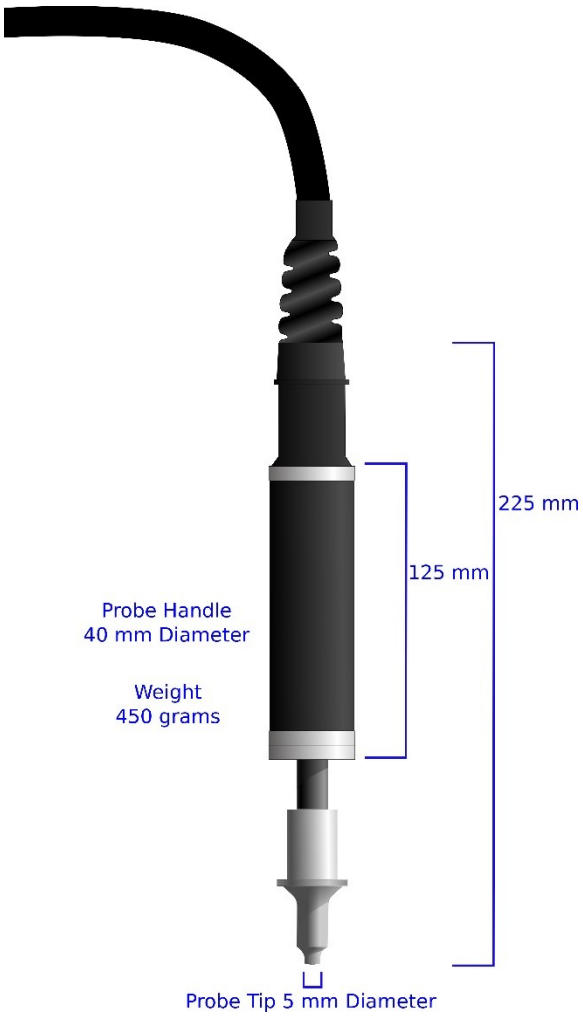

Figure 10 Diagram of hand held ultrasonic probe, with the piezoelectric transducer located in the black barrel-shaped handle. Not to exact scale.

5.3 Device Parameters

5.3.1 Frequency

The device works at a fixed frequency of 40 kHz, which is generated by the piezoelectric transducer in the probe.

5.3.2 Power Dial

The power dial can be changed by the operator from 20% to 90%, which is described as a percentage of total amplitude of the device. Total amplitude is affected by many variables, such as the electrical power input, the size and shape of the probe tip, and the liquid media the ultrasound flows through. A numeric value of amplitude has not been calibrated for this device configuration.

5.3.3 Wattage

Operational wattage (W) capacity for this unit is 50 W. This is the wattage capacity of the electromechanical components of the device, which in turn affects the power capability of the device. Wattage for commercial ultrasonic baths is often given as a wattage density of 50 or 100 W per gallon of the ultrasonic bath.[50] The HDS ultrasonic probe does not have a calculated wattage density or wattage output.

5.3.4 Timer

The device is further regulated by limiting the time of the ultrasonic pulse, from a tenth of a second up to 60 seconds. There is no mechanism to leave the ultrasound engaged for longer intervals.

5.4 Health and Safety

A review was done of general health and safety recommendations for working with ultrasound cleaning equipment. Health and safety recommendations generally fell into two categories: potential damage to human tissue from physical contact with an ultrasonic transducer or the liquid media, or damage from ambient noise of ultrasonic equipment.

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[50] Rachel Kohn, "10 Things You Need to Know Before Selecting an Ultrasonic Cleaner", Ultrasonic Cleaners (blog), *Tova Tech*, 15 May 2012, https://www.tovatech.com/blog/11021/ultrasonic-cleaner/how-to-pick-an-ultrasonic-cleaner.

5.4.1 Physical Contact

Risks of tissue contact with a transducer can result in transmission of thermal energy, which can cause burns. Contact with ultrasound through a liquid medium can cause surface damage to tissue. Reports of damage to blood and internal tissue in vivo and in vitro have been reported in mammals across a range of frequencies (20-100 kHz), decibels (75-155 dB), and time exposures (one minute to one day).[51]

5.4.2 Exposure to Ultrasound Transmitted Through Air

Hearing damage caused by airborne ultrasound is also possible and is highly dependent on the decibel level. Generally, over 110 dB can lead to damage to the inner ear, leading to shifts in sound perception or hearing loss. Self-reported symptoms of headache, nausea, and other effects from those working around airborne ultrasound have been reported in industrial usage, at a wide variety of frequency ranges, dB levels, and time exposure.[52]

5.4.3 Health and Safety Recommendations

Ultrasonic device parameters and their potential risks must be assessed before beginning any work. Health and safety recommendations for commercial ultrasonic cleaning devices for industrial or home use that operate at lower-range frequencies (20-100 kHz) warn users of potential damage to human tissue from touching the interior tank, transducer, or the liquid bath when ultrasound is on.[53] Health and safety guidelines followed for the experimental phases can be found in *Appendix C*.

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[51] Farzaneh Ahmadi et al., "Bio-Effects and Safety of Low-Intensity, Low-Frequency Ultrasonic Exposure," *Progress in Biophysics and Molecular Biology* 108, no. 3 (2012): 119–38, accessed 20 February 2018, doi:10.1016/j.pbiomolbio.2012.01.004.
[52] Ibid., 119-38.
[53] Elma GmbH & Co KG, "Operating Instructions for Elmasonic P Ultrasonic Cleaning Units" (Elma GmbH & Co KG), accessed 24 July 2018, http://imlab.be/imlab_nl/elma/Pdf/Elmasonic_P/Elmasonic_P_Operating_Instructions_ENG_Imlab.pdf; Branson Ultrasonics Corp., "Operators Manual," accessed 24 July 2018, https://www.nist.gov/sites/default/files/documents/ncnr/UltrasonicCleaner_Branson_1510.pdf; Allendale Ultrasonics, "Ultrasonic Cleaner User Manual 2013," accessed 24 July 2018, https://www.allendale-ultrasonics.co.uk/docs/ultra/ultrasonics-v1.pdf.

6 Phase 1: Characterising Cavitation Activity of the Ultrasonic Probe

6.1 Introduction

As discussed in *section 4.4,* the cleaning effect of ultrasound is largely a function of cavitation collapse. There are several ways to detect and characterise cavitation or bubble activity without specialized equipment: visually, aurally, and through an aluminium foil test.[54] Visually, bubbles that result in collapse and cleaning are not usually seen by the naked eye, as the bubbles are very small, and exist for only fractions of a second. However, ultrasound can create larger, stable bubbles from the degassing of liquids, and the formation and collapse of bubbles from liquid agitation.[55] Aurally, white noise is a known phenomenon of cavitation collapse,[56] but does not necessarily give an indication of strength or location of cavitation collapse. Aluminium foil easily becomes dented, pitted and mechanically damaged to the naked eye during cavitation collapse.[57] The formation of bubbles in the liquid visible to the naked eye, the audible white noise of cavitation, and damage to aluminium foil were all assessed in this experiment to characterise the cavitation collapse and therefore cleaning and damage potential of the probe using non-specialised equipment.

Within any conservation cleaning environment, the cleaning solutions are integral to the success of the treatment. Cavitation is also affected by the physical properties of liquids, and thus cavitation collapse potential is changed due to a complex relationship of pressure, density, surface tension, and other factors. The chemical components of common aqueous solutions in textile conservation present opportunities for unique sonochemical reactions to occur.[58] As such, six different pure-aqueous and dilute aqueous solutions were tested.

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[54] Fuchs, "Ultrasonic Cleaning," 591-592.
[55] Madeleine Bussemaker and Dongke Zhang, "A Phenomenological Investigation into the Opposing Effects of Fluid Flow on Sonochemical Activity at Different Frequency and Power Settings," *Ultrasonics Sonochemistry* 21, no. 1 (2014): 436–45, accessed 27 January 2018, doi:10.1016/j.ultsonch.2013.07.002.
[56] Nicolas Segebarth et al., "Correlation between Acoustic Cavitation Noise, Bubble Population, and Sonochemistry," *Journal of Physical Chemistry B* 106, no. 35 (2002): 9181–90, accessed 22 June 2018, doi:10.1021/jp0146566.
[57] Bram Verhaagen and David Fernández Rivas, "Measuring Cavitation and Its Cleaning Effect," *Ultrasonics Sonochemistry* 29 (2016): 619–28, accessed 10 March 2018, doi:10.1016/j.ultsonch.2015.03.009.
[58] Bogdan Niemczewski, "A Comparison of Ultrasonic Cavitation Intensity in Liquids," *Ultrasonics* 18, no. 3 (1980): 107–10, accessed 10 March 2018, doi:10.1016/0041-624X(80)90021-9.

6.2 Aims

The aim of this experiment was to characterise and relate the action of the ultrasonic device in a controlled cleaning environment through visible and audible detection, and the physical effects on aluminium foil. A secondary aim was to evaluate the impact of six different aqueous solutions and three different amplitude settings on the visible, audible, and aluminium foil detection methods.

6.3 Methodology

Experiments were carried out at fixed distances in 100 mL of solution in a stainless-steel beaker (*Figure 11*). A 30 mm circle of aluminium foil[59] was placed at the bottom the beaker, and then a solution was added (*Figure 12)*. Ultrasound was applied for 60 seconds, and the foil was removed, labelled and evaluated. Testing parameters are outlined in *Table 1*. All testing was done at ambient conditions (18-28 °C, 30-55% relative humidity [RH]).

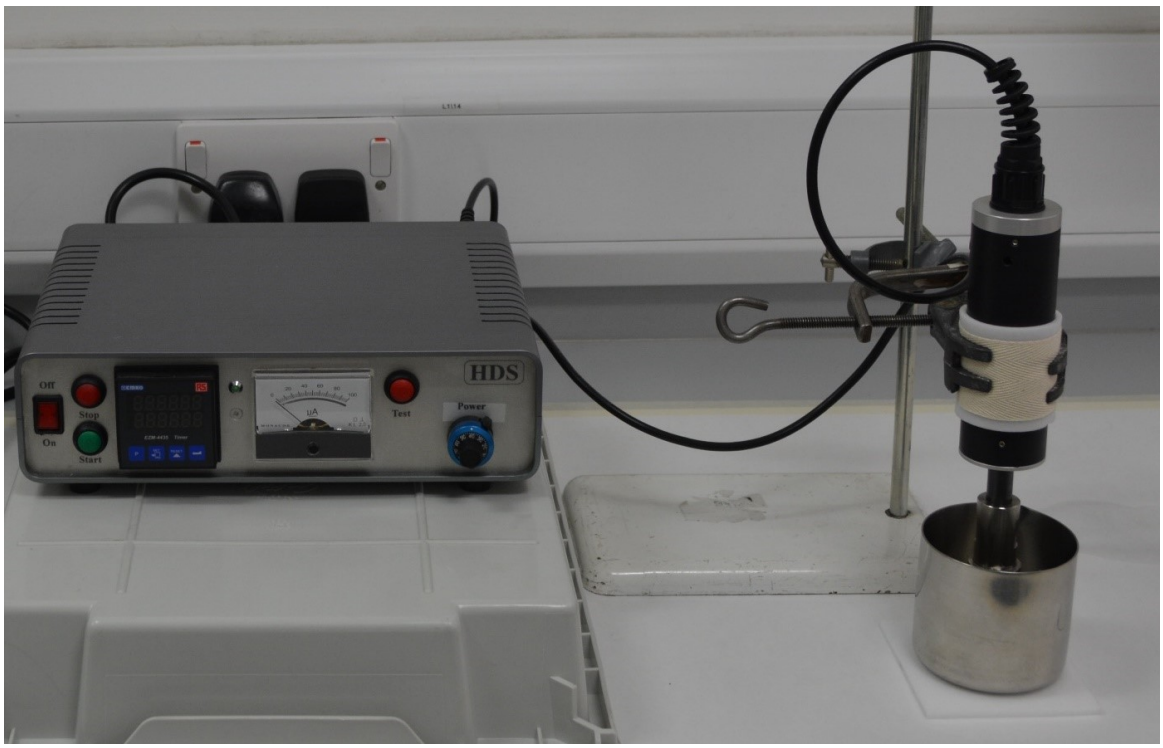

Figure 11 Experimental testing set up.

[59] Standard kitchen-supply aluminium foil was used.

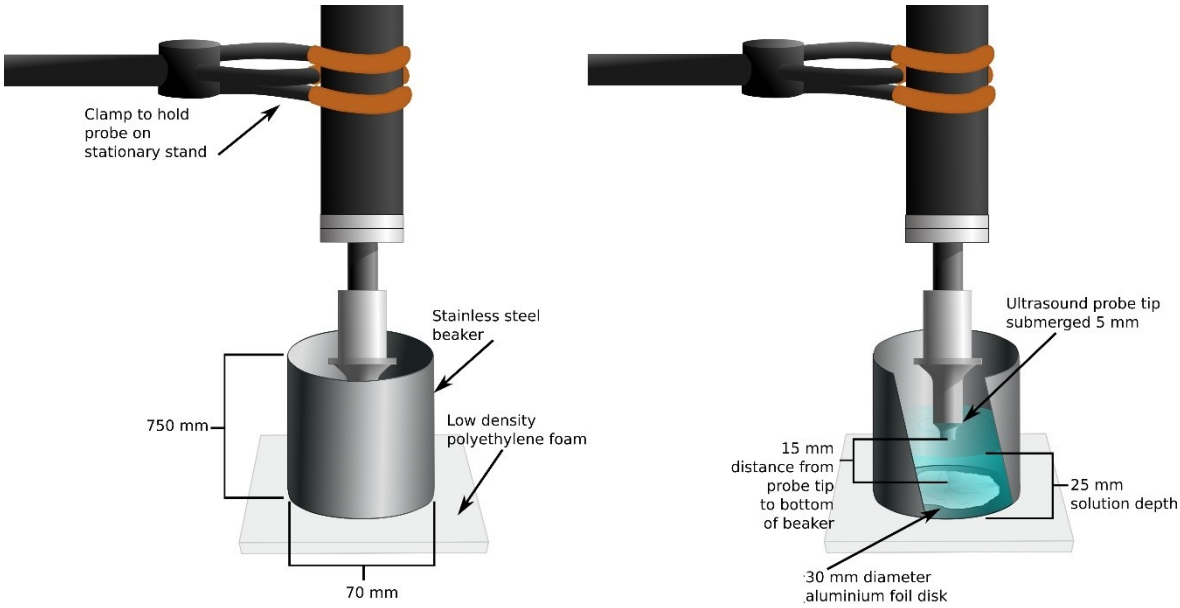

Figure 12 Diagram of the testing environment for the detection of cavitation by visual, aural, and aluminium foil detection, not to exact scale.

Solution	Amplitude %		
	40	**60**	**80**
Degassed water	Group 1	Group 2	Group 3
Deionised water	Group 4	Group 5	Group 6
Soft water	Group 7	Group 8	Group 9
0.3% w/v Dehypon® LS54 in deionised water	Group 10	Group 11	Group 12
0.5 g/L Trisodium citrate in deionised water	Group 13	Group 14	Group 15
0.05 g/L SCMC in deionised water	Group 16	Group 17	Group 18

Table 1 Test groups for cavitation characterisation on variables of solution and amplitude, resulting in 18 test groups of 5 replicate samples each for a total of 90 samples.

6.3.1 Cleaning Solutions

Six test solutions were used to represent a variety of chemical compositions, densities, viscosities, vapour pressures, and other qualities. Three purities of water were selected for the different ion and gas content of each type (*Table 2*). Three dilute cleaning solutions were made with deionised water. These solutions were chosen as they are common components of wet cleaning solutions for textile conservation, either alone or in combination with distinctly different molecular size, structure, and cleaning mechanisms (*Table 3*). Each water purity and dilute solution represented an opportunity to observe different cavitation effects based on the properties of the liquid.

Water Type	Relative Purity	Use	Preparation
Degassed	Very low ionic concentration and very low gas content, slightly acidic pH (around 6)	Sonochemistry[60]	Repeat boiling of deionised water, followed by sealing in a container under a vacuum
Deionised	Very low ionic concentration, slightly acidic pH (around 6)	Conservation solutions[61]	Reverse osmosis filtration of tap water
Soft	Scottish water has a low ion concentration with a neutral pH (around 7)[62]	Wet cleaning historic textiles[63]	Used directly from the tap

Table 2 Three purities of water selected for experimentation.

Additive	Molecular Weight (g/mol)	Description	Action on Aqueous Solution			
			Density	Viscosity	Surface Tension	Speed of Sound
3% w/v Dehypon® LS54	638.914	Non-ionic surfactant, long, narrow shape, hydrophilic head, hydrophobic tail[64]	Increase	Increase	Decrease	Suspected Increase
0.5g/L Trisodium Citrate	258.06	Salt, pH buffer, compact molecular size and shape[65]	Increase	Increase	Increase	Increase
0.05g/L SCMC	262.19	Emulsifier, cellulose ether, compact molecular size and shape[66]	Increase	Increase	Decrease	Suspected Increase

Table 3 Properties of additives selected for cleaning. The suspected increase on the speed of sound for Dehypon® LS54 and SCMC is based on the relationships with the other listed parameters.[67]

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[60] Bogdan Niemczewski, "Cavitation Intensity of Water under Practical Ultrasonic Cleaning Conditions," *Ultrasonics Sonochemistry* 21, no. 1 (2014): 354–59, accessed 12/28/2017 doi:10.1016/j.ultsonch.2013.07.003.
[61] Tímár-Balázsy and Eastop, *Chemical Principles*, 185-190.
[62] Scottish Water, *Water Register Regulation Zone=Milngavie M3* (Scotland, 2018), accessed 12 June 2018, https://www.scottishwater.co.uk.
[63] Tímár-Balázsy and Eastop, *Chemical Principles*, 185-190.
[64] Moe Sato, "An Experimental Evaluation of Non-Ionic Surfactant Dehypon® LS54," (master's dissertation, University of Glasgow, 2014), 5-12; Supplied by BASF Chemical Co, Ludwigshaven Germany.
[65] Pub Chem, "Trisodium Citrate Dihydrate," accessed 27 July 2018 https://pubchem.ncbi.nlm.nih.gov/compound/Trisodium_citrate_dihydrate#section=Top.; Reagent grade, Fisher Chemical, Leicestershire, England.
[66] Ibid., "Sodium Carboxymethylcellulose," accessed 27 July 2018 https://pubchem.ncbi.nlm.nih.gov/compound/23706213#section=Computed-Properties; Research and development grade, Sigma-Aldrich Co. St. Louis, Missouri, USA.
[67] Diego Gómez-Díaz, José M. Navaza, and Begoña Sanjurjo, "Density, Kinematic Viscosity, Speed of Sound, and Surface Tension of Hexyl, Octyl, and Decyl Trimethyl Ammonium Bromide Aqueous Solutions," *Journal of Chemical and Engineering Data* 52, no. 3 (2007): 889–91, accessed 31 July 2018, doi:10.1021/je060486k.

6.4 Results

Cavitation or bubble formation was visibly seen, and cavitation collapse was audibly heard in the liquid for all 90 tests, covering all amplitudes for all six solutions tested. Cavitation collapse was detected on the aluminium foil of 43 samples (*Figure 13*).

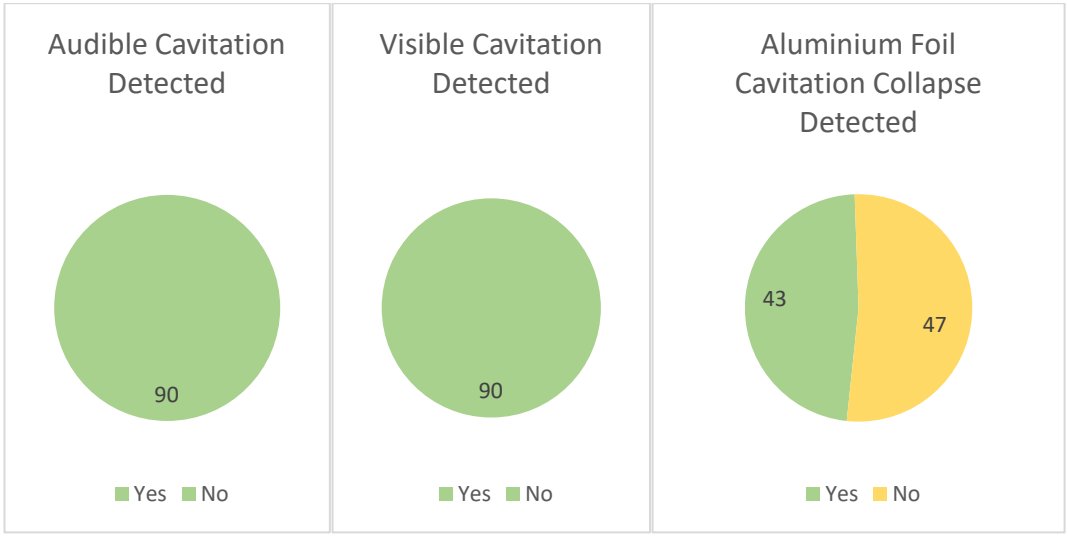

Figure 13 Chart summarizing the overall cavitation detection results audible, visible, and aluminium foil cavitation results.

6.4.1 Visual Detection

Visual detection of bubble formation in the liquid was analysed as a yes or no presence for each test. There was visible bubble formation and agitation of liquid for 100% of samples. The amount of visible bubble formation and agitation within the liquid varied, as did the size, shape, and location of bubbles (*Figure 14*). The lowest tested amplitude of 40% had the least visible bubble activity and liquid agitation. At 80% amplitude, agitation and bubble activity were so high that they resulted in water droplets being thrown from the solution, with vapour rising from the solution of all tests at 80% amplitude. In surfactant solution of Dehypon® LS54, very little foam was produced, although large stable bubbles were prevalent, even at 80% amplitude.

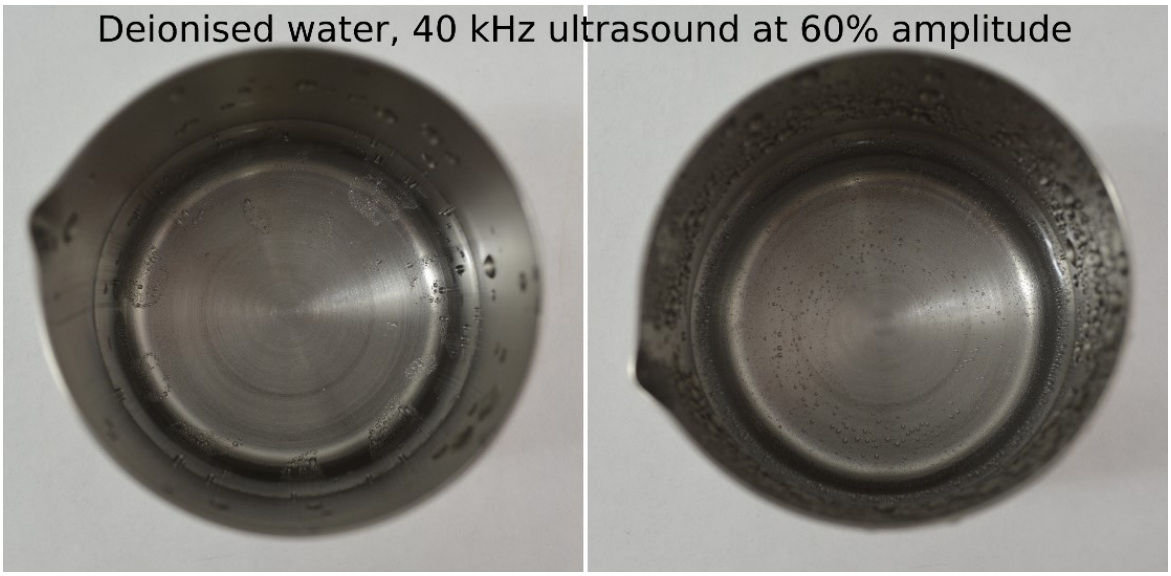

Figure 14 Stable bubble formation seen after 10 minutes ultrasonic pulse. Localised bubble clusters seen on the left, and bubbles dispersed across the bottom of the beaker on the right.

6.4.2 Aural Detection

The white noise of cavitation was heard in all tests. The loudness of the audible cavitation signal varied. The lowest amplitude of 40% was generally quieter with the least white noise. Amplitudes of 60% and 80% created similar levels of white noise across all solutions. The sound sometimes started with the initiation of ultrasound, while at other times there was a delay in the onset of the white noise after initiation of ultrasound. Occasionally loud, high-pitched resonance was heard at several amplitudes. Decibel (dB) level was intermittently measured for health and safety with the SoundMeter[68] app (accurate within ±2dB)[69] giving a range of 70-95dB across the amplitudes tested (background ambient noise level was 60dB) but results were not systematically recorded for analysis.

6.4.3 Cavitation Collapse Detection by Aluminium Foil

Cavitation collapse was detected on 43 of the 90 aluminium foil samples tested. All samples were evaluated into three categories: high, low, and no, representing the relative amount of cavitation collapse detected (*Figure 15)*.

[68] LQH Developers application version 1.0.2, using iPhone 5S integrated microphone, measuring only frequencies audible to the human ear.
[69] Chucri Kardous and Peter Shaw, "Evaluation of Smartphone Sound Measurement Applications," *The Journal of the Acoustical Society of America* 135, no. 4 (2014): EL186-EL192, accessed 12 May 2018, doi:10.1121/1.4865269.

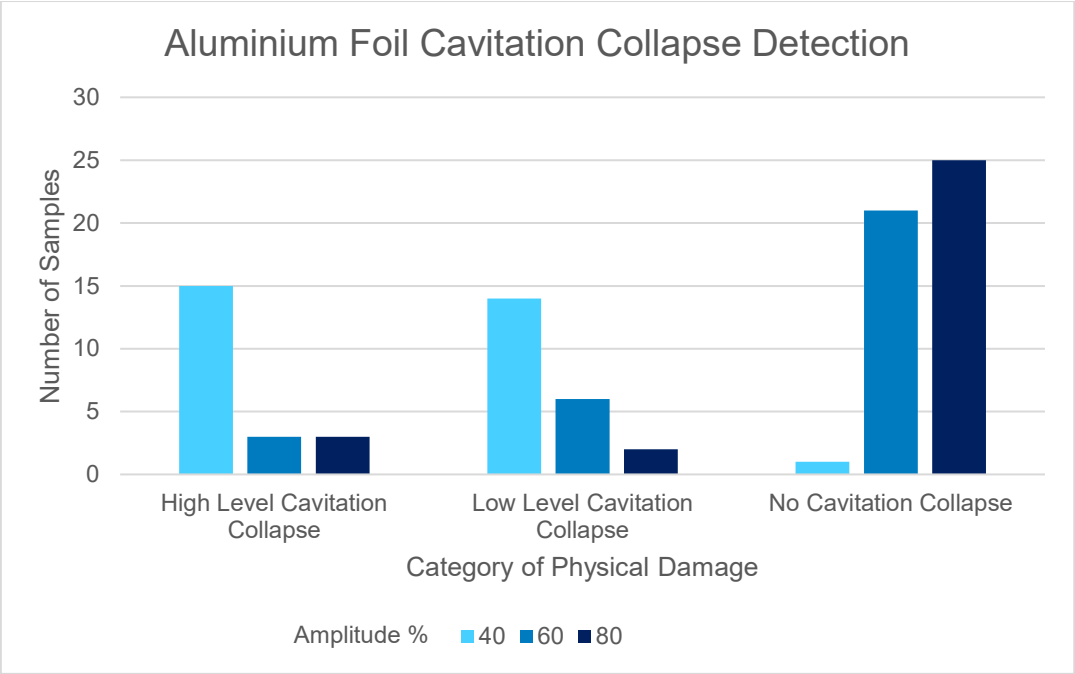

Figure 15 Chart showing overall detection of physical damage using aluminium foil according to amplitude for all six test solutions.

6.4.3.1 High-Level Cavitation Collapse

High-level cavitation collapse was defined as widespread pitting and damage across the aluminium foil, or cavitation that created holes though the aluminium in one or more locations. High-level cavitation collapse was identified in 21 samples across the six solutions (*Figure 16*). It was seen most frequently at 40% amplitude, and less frequently detected at 60% and 80% amplitude for all solutions. Solutions of degassed deionised water, and soft water, followed by Dehypon® LS54 had more samples with high-level cavitation collapse at 40% amplitude compared to other solutions (*Figures 17, 18, 19*).

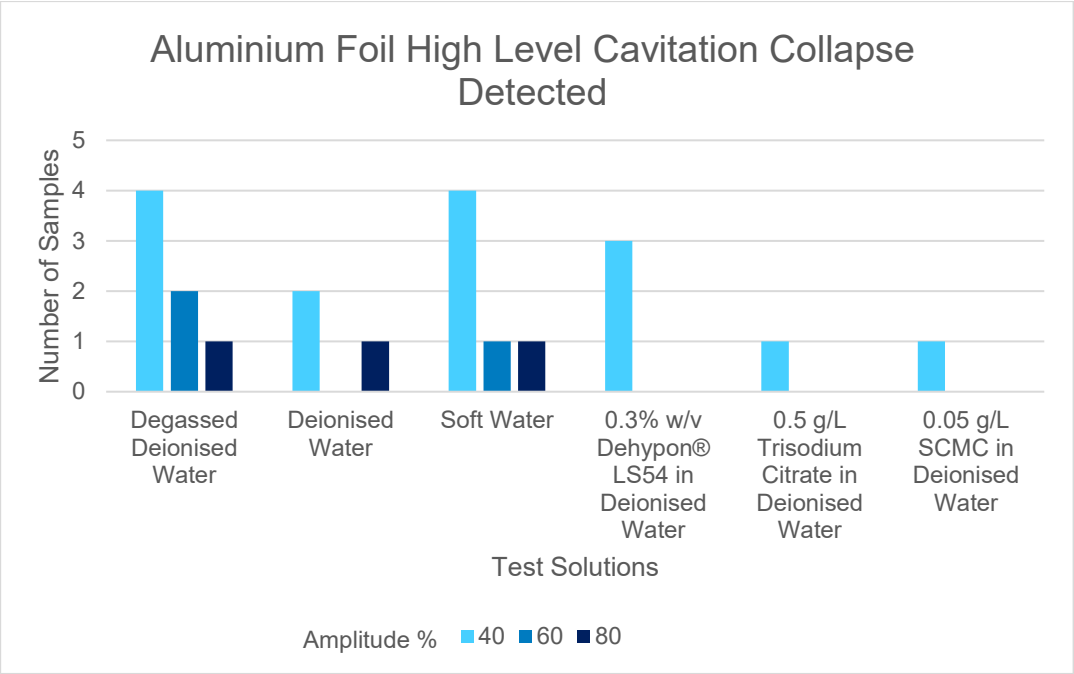

Figure 16 Chart of aluminium foil high-level cavitation collapse by solution and amplitude.

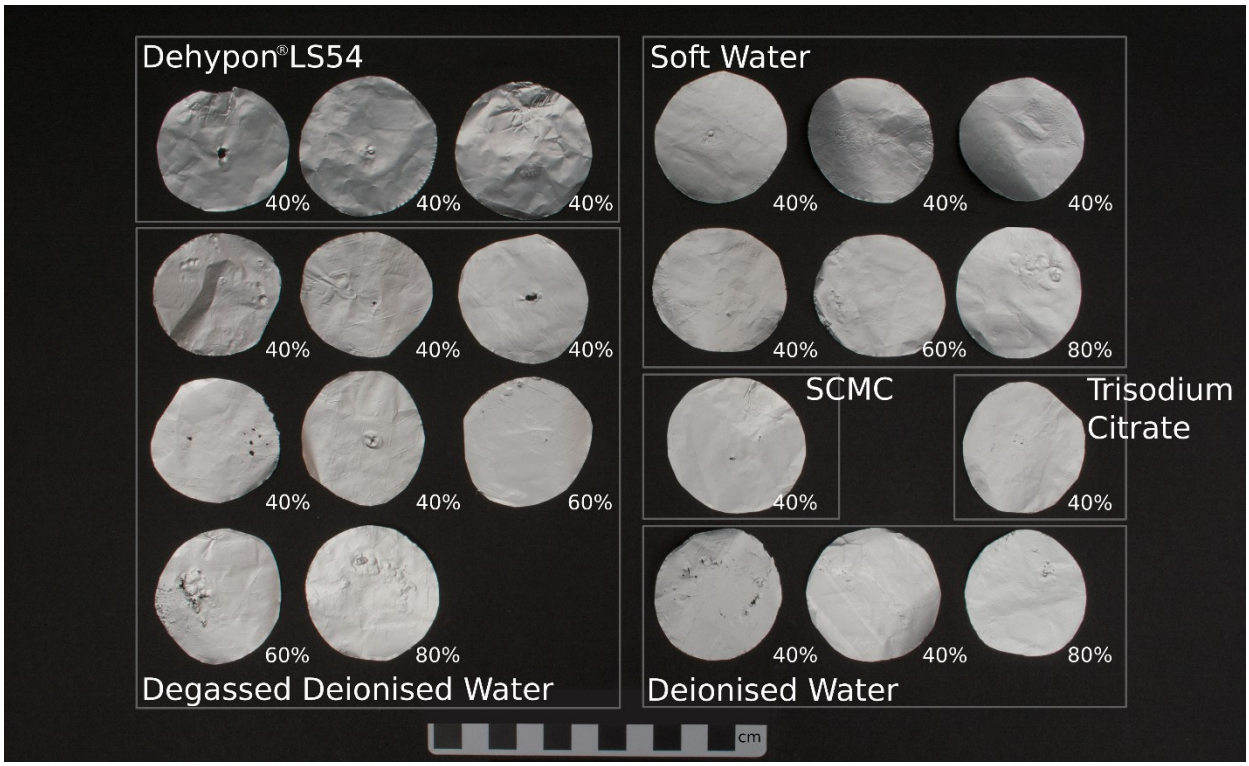

Figure 17 All samples with high-level cavitation damage.

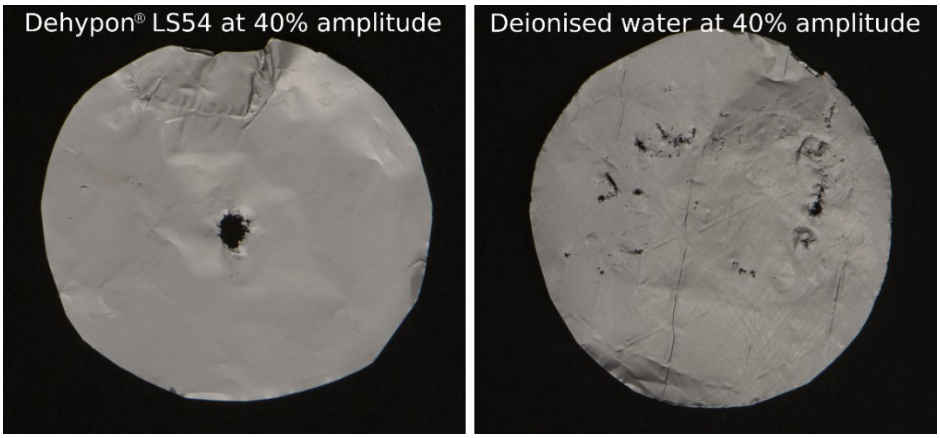

Figure 18 Detail of high-level cavitation collapse on aluminium foil. Dehypon® LS54 at 40%, and deionised water at 40%.

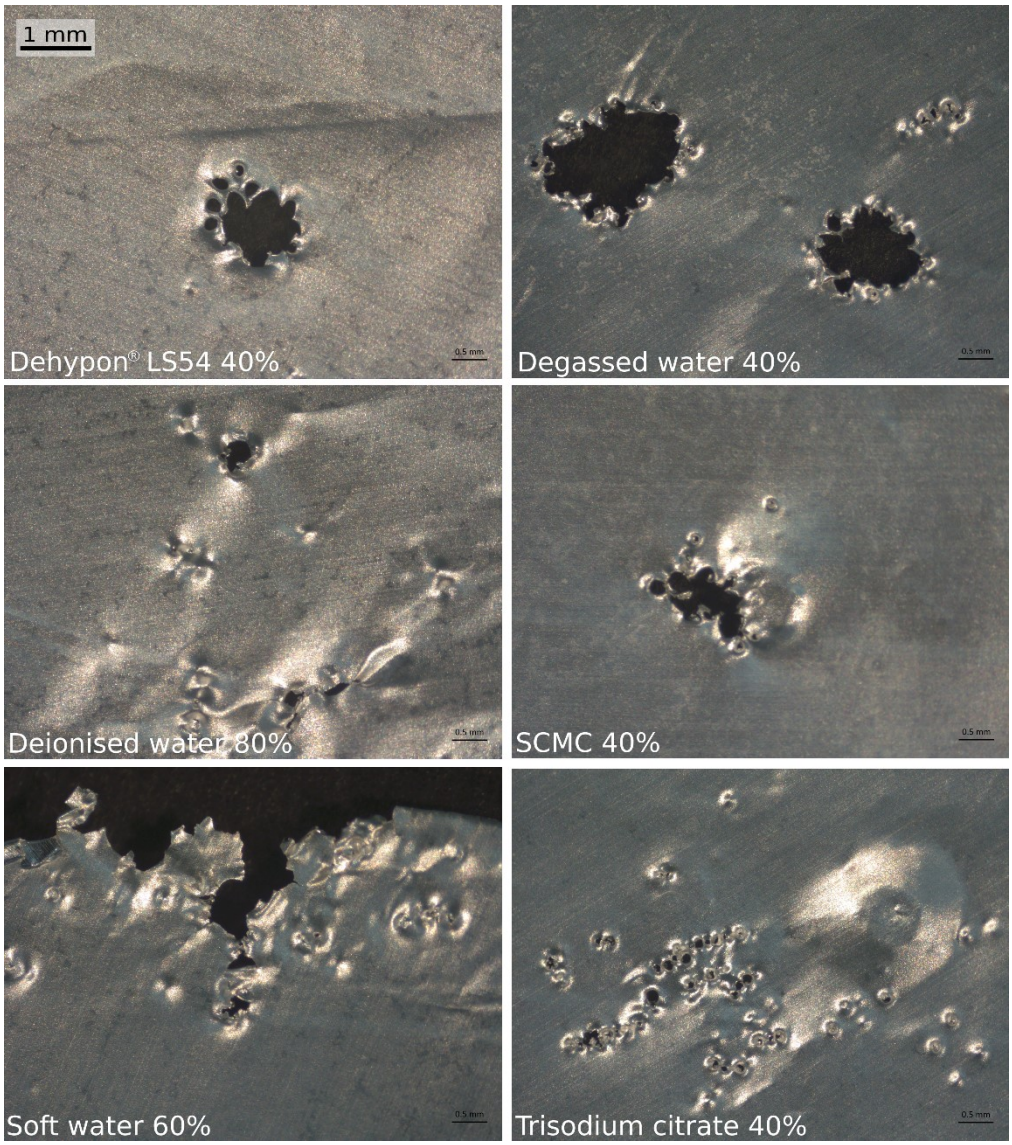

Figure 19 Detail of physical damage such as pitting and holes in aluminium foil from cavitation collapse annotated with test solution and percent amplitude ultrasound.

6.4.3.2 Low-Level Cavitation Collapse

Low-level cavitation collapse was identified in 22 samples across the six test solutions (*Figure* 20*)*. This was defined as localised small areas of pitting, but with no holes in the aluminium (*Figures 21, 22, 23)*. Within the testing environment, low-level physical damage was seen most frequently at 60% amplitude tested for deionised water, effecting all five samples, followed by trisodium citrate and SCMC with four of five samples affected.

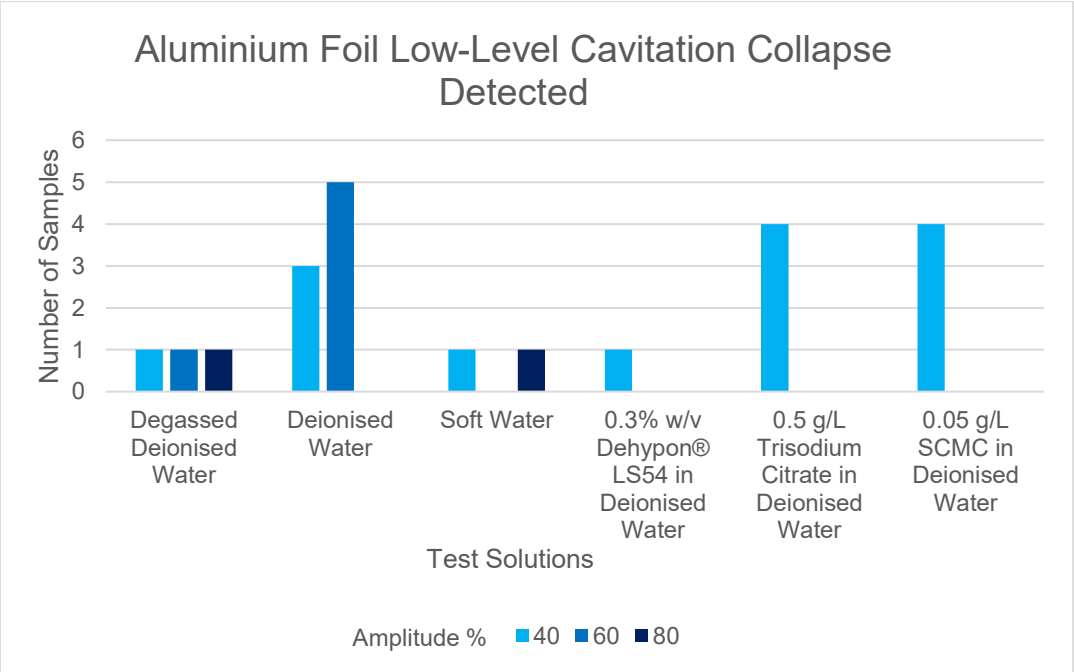

Figure 20 Chart of aluminium foil low-level cavitation collapse by solution and amplitude.

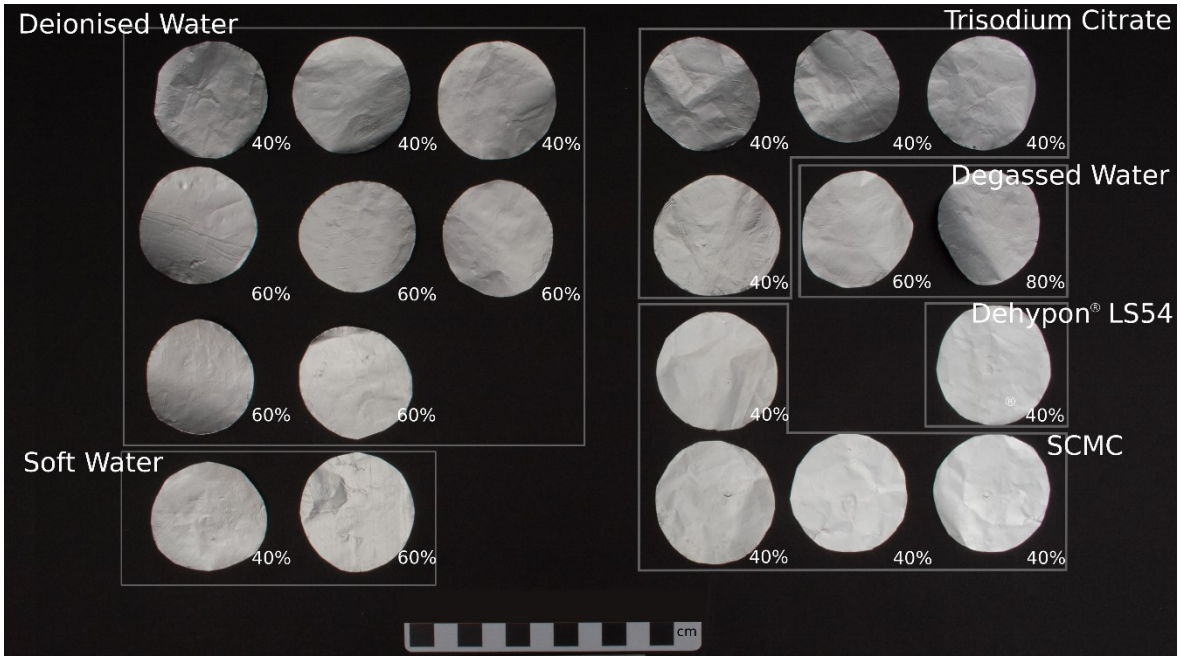

Figure 21 Low-level cavitation collapse on aluminium foil samples.

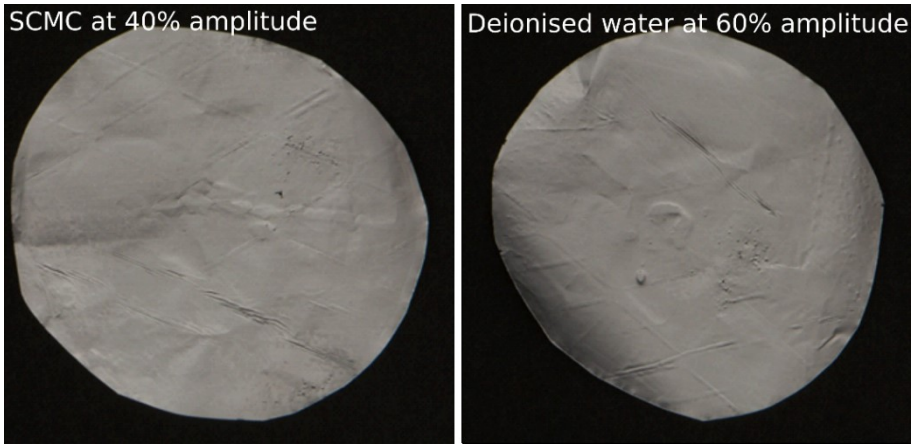

Figure 22 Examples of low-level cavitation collapse.

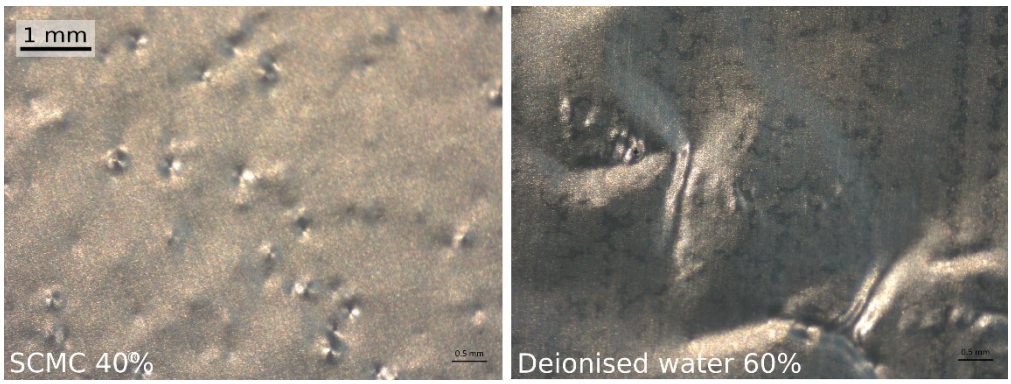

Figure 23 Examples of low-level cavitation collapse seen with low-powered microscopy.

6.4.3.3 No Cavitation Collapse

No cavitation collapse damage was defined by complete lack of pitting or surface damage due to cavitation (*Figure 24*). This was visible in 47 samples across the six solutions (*Figure 25)*. Within the testing environment, no cavitation collapse was seen most frequently at 80 and 60% amplitude for five of the solutions. Deionised water was an exception to this, as discussed above.

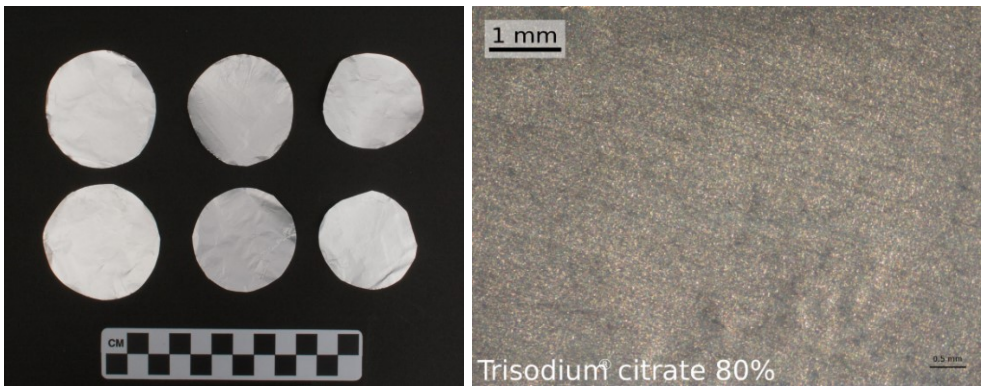

Figure 24 One example of no cavitation collapse detected for each of the solutions tested on the left, and one example under low-powered microscopy on the right.

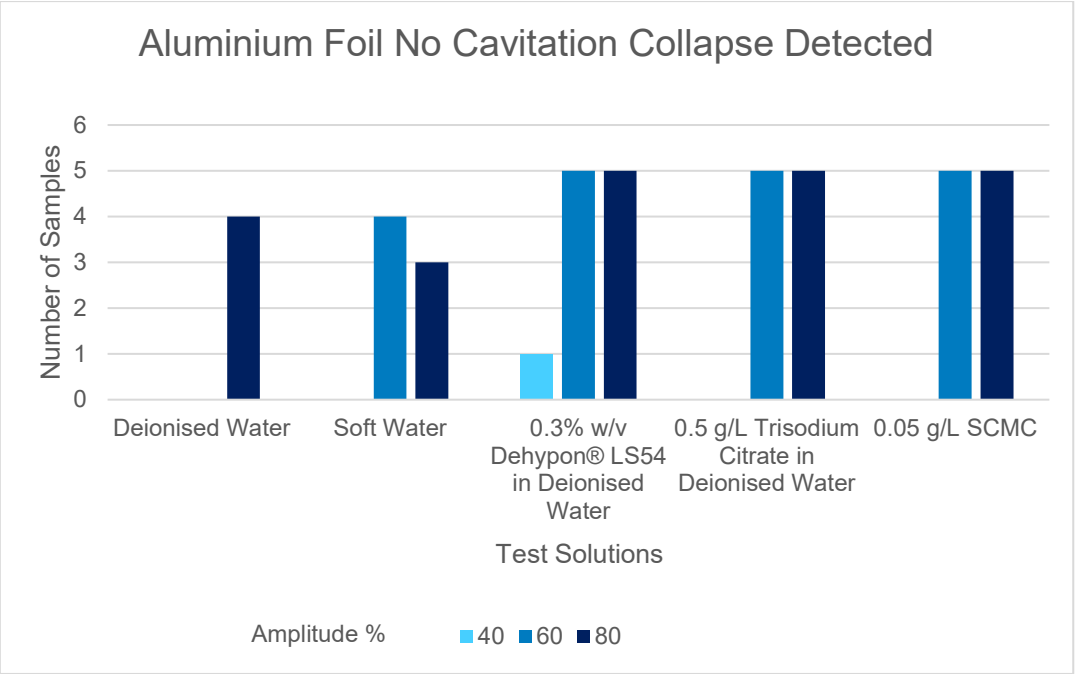

Figure 25 Chart of aluminium foil with no cavitation collapse detected by solution and amplitude.

6.5 Discussion

In this experiment, inexpensive, practical methods of detecting cavitation for any ultrasonic cleaning system and environment were explored. The impacts of six solutions and three amplitudes were also explored within an otherwise fixed, stationary testing environment. Dehypon® LS54 provided an interesting example of how ultrasonic cleaning differs from sponging. The ultrasonic device did not create stable white foam, as occurs with the surfactant with sponging. This indicates that the mechanical action provided by ultrasound is distinct from traditional sponging techniques.

In reviewing the results, it is important to remember that aluminium foil, as a thin, soft metal easily becomes pitted and damaged during cavitation collapse. This makes it a useful indicator of the location and relative force of cavitation collapse, but it is not indicative of intrinsic damage to a textile. The material properties of thin aluminium foil and textile fibre types are significantly different, including tensile and shear strength, flexibility, density, and porosity. However, within ultrasonic cleaning systems for historic textiles, low-level cavitation collapse strength may be preferable to high-level collapse strength to moderate the strong physical and chemical forces acting on the textile.

6.5.1 Health and Safety

During testing, amplitudes of 80% resulted in vapour rising from the cleaning solutions. Wash bath solutions that are acidic, basic, or have specific risk and toxicity to eyes, respiratory systems, or skin are used in conservation, creating additional risks if the solution is vaporised by ultrasound. In conservation practice, soiling is also solubilised into the wash bath, which can include pesticides, and other materials that pose a risk to health and safety, such as historical moth treatments (naphthalene).[70]

The resonance of the cylindrical metal vessel in this experiment resulted in unpleasant, loud, high-pitched noise that required hearing protection. This resonance is not expected for all cleaning environments but hearing protection should be available to those working with or near ultrasound as indicated in *section 5.4*.

6.6 Conclusion

6.6.1 Visual Cavitation Detection

Visual assessment of the liquid in not recommended to understand cavitation, as the type of bubbles that are visible may not be contributing to cleaning. It is important to not correlate cavitation with degassing, foaming, agitation, or other movement within the liquid. At 40% amplitude, relatively little cavitation was visible as bubble formation in the liquid when the ultrasonic frequency was applied, which directly contradicts the damage to aluminium foil. Visual assessment is not a reliable method for determining cavitation collapse location or strength.

6.6.2 Aural Cavitation Collapse Detection

Cavitation by auditory assessment was a clear indicator of cavitation collapse. The sound can be described as a fizzing, static, or white noise, and the correlation of sound and the presence of cavitation is well known and described in ultrasonic literature on cavitation.[71] However the sound did not always correlate with physical effects of cavitation collapse seen on the aluminium foil tests.

[70] Tímár-Balázsy and Eastop, Chemical Principles, 292-300.
[71] Segebarth et al., "Correlation between Acoustic Cavitation Noise, Bubble Population, and Sonochemistry," 9181-9190.

6.6.3 Aluminium Foil Cavitation Collapse Detection

Damage to aluminium foil was a useful indicator of the location and strength of cavitation collapse in a given wet cleaning environment. The impact of amplitude became clear in this experiment. Cavitation collapse damage of aluminium foil was most consistently seen at 40% amplitude. This was the lowest amplitude tested, with very low visibility of activity in the wash bath, but did have reliable, quiet white noise.

Higher amplitude did not result in more cavitation collapse. The highest amplitude tested of 80% resulted in the least amount of strong cavitation collapse on a parallel surface 15 mm from the ultrasound tip. This was possibly due to the higher convection of liquid that was noticed at 80% amplitude, moving the liquid solution around with great force. Cavitation may be occurring, but the location and directionality of collapse may not have been able to interact with the surface of the aluminium due to the high agitation in the wash bath.

The results indicated that strong cavitation collapse on a near (15 mm) parallel surface, at low amplitudes was possible. The distance of the probe to the aluminium was representative of low wash-bath depths used in textile conservation, indicating that strong cavitation on a textile could also occur in practical applications using a similar depth of water.

The six solutions had some impact on the level of cavitation collapse seen on the aluminium foil. Trisodium citrate and sodium carboxymethyl cellulose both resulted in damage to the aluminium foil at 40% amplitude for all five samples tested, with four out of 5 showing low-level damage. Dehypon® LS54 showed damage to the aluminium foil at 40% for all five samples tested, however four had high-level damage. This would support a conclusion that the solution properties (density, vapour pressure, surface tension and others) had significant impact on the liquid environment which could increase or decrease cavitation activity and strength, as seen in the literature.[72]

[72] Niemczewski, "A Comparison of Ultrasonic Cavitation Intensity in Liquids," 107-110.;
Bogdan Niemczewski, "Influence of Concentration of Substances Used in Ultrasonic Cleaning in Alkaline Solutions on Cavitation Intensity," *Ultrasonics Sonochemistry* 16, no. 3 (1 March 2009): 402-407, accessed 23 July 2018, doi:10.1016/J.ULTSONCH.2008.09.002.

7 Phase 1: Cavitation Characterisation by High Speed Imaging

7.1 Introduction

Cavitation occurs at small sizes and high speeds, which means it is not always possible to directly visualise or analyse cavitation activity with the naked eye. As discussed in *Chapter 4*, cavitation activity is also greatly impacted by the cleaning environment, with respect to volumes, distances, and material properties involved. Observing cavitation activity of liquids with varied parameters has resulted in improved understanding of cavitation activity in a given environment.[73]

7.2 Aims

The aim of this experiment was to characterise the cavitation activity of the probe showing the nucleation, growth, and collapse of cavitation evolving at a very small scale and high speed. A secondary aim was to examine how depth of probe submergence, and the proximity of a surface impacts cavitation at the tip of the probe in deionised water at different amplitudes.

7.3 Methodology

High speed imaging was performed by Dr. Paul Prentice at the Cavitation Research Laboratory[74] using a Shimadzu HPV-X2 high speed camera.[75] Cavitation was detected using the Shimadzu instrument via a live feed to image capture software (*Figure 27*). Experiments were performed with the ultrasonic probe submerged in deionised water using a custom plastic tank (*Figure 26*). This tank is of a large enough size and volume to allow for imaging of acoustic cavitation with limited effects from acoustic field reflection. All experiments were performed at ambient conditions (24 °C, 50% RH).

This experiment used variables of submerged depth of probe, and proximity to a flat surface (*Table* 4). The introduced surface was a 3 mm thick aluminium plate, used to approximate the bottom of a wash tank during conservation wet cleaning. Images were taken at 500,000 frames per second (fps). Three image capture sequences were done at each of the tested variables.

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[73] Tzanakis et al., "Characterizing the Cavitation Development and Acoustic Spectrum in Various Liquids," *Ultrasonics Sonochemistry* 34 (January 2017): 651–62, accessed 22 June 2018, doi:10.1016/j.ultsonch.2016.06.034.
[74] University of Glasgow, European Research Council, http://cavlab.co.uk/.
[75] Shimadzu Corporation, https://www.shimadzu.com/an/test/hpv/hpv-x2/.

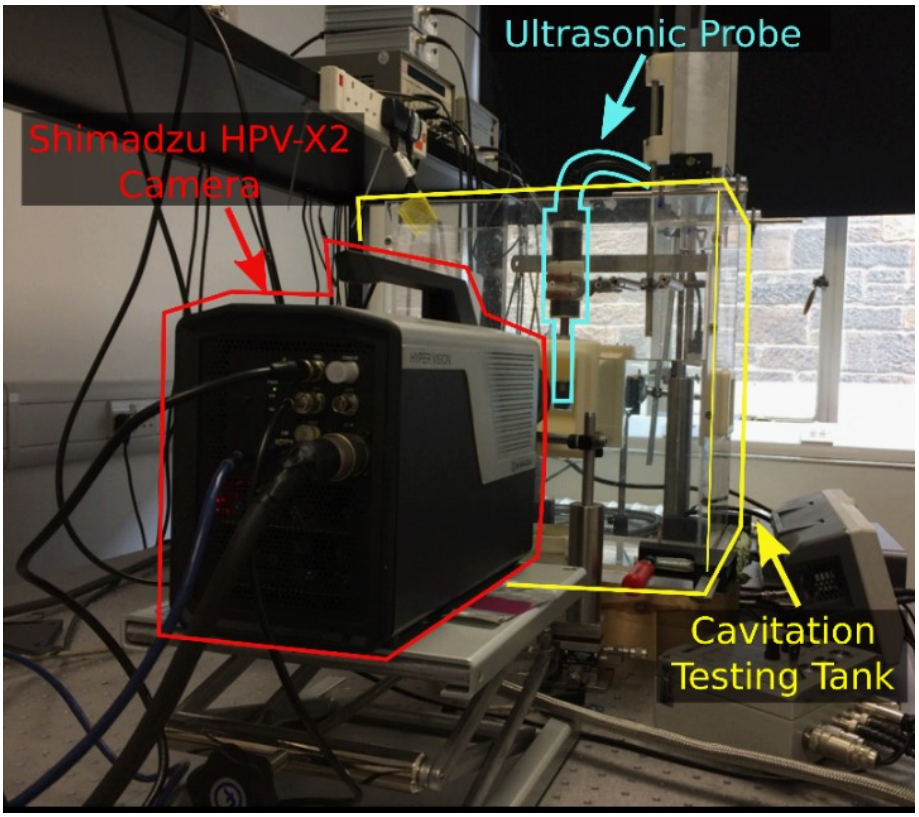

Figure 26 Cavitation lab experimental set up.

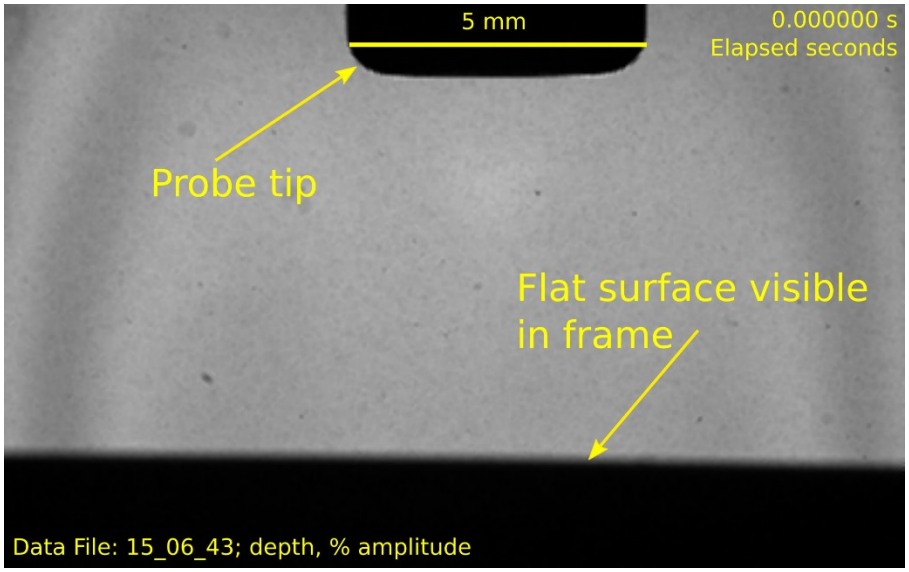

Figure 27 Diagram of Shimadzu imaging frame with annotations. The high-contrast, back-lit images show the probe and a flat surface as opaque black shapes. The deionised water of the testing environment appears as shaded grey background. Any bubble activity or wave forms will appear as black and grey back-lit shapes. At the top right, each image is annotated with the number of elapsed seconds of each imaging sequence. The data file annotation is displayed with probe depth, and the percent amplitude at the bottom left.

Depth of Probe Tip in Solution	Distance from Probe Tip to Surface	Amplitude %
5 mm	≥ 200 mm	40
		60
		80
20 mm	≥ 200 mm	40
		60
		80
	≥ 15 mm	40
		60
		80
	≥ 10 mm	40
		60
		80

Table 4 Experiment parameters of submerged probe depth, near surface distance, and amplitude examined with high-speed imaging.

7.4 Results: Effects of Probe Submergence Depth

With the tip of the probe submerged to a depth of 20 mm, cavitation was only detected at 80% amplitude. With the tip of the probe submerged to a depth of 5 mm, cavitation was detected at all amplitudes tested (*Table 5*). The change in cavitation activity was as visibly different for each image frame in the test, as it was between test variables.

Depth of Probe Tip in Solution	Distance from Probe Tip to Surface	Amplitude %	Cavitation Detected
5 mm	≥ 200 mm	40	Yes
		60	Yes
		80	Yes
20 mm		40	No
		60	No
		80	Yes

Table 5 Cavitation detection by imaging under magnification, according to depth of probe, distance to surface, and percent amplitude.

7.4.1 Probe Depth of 20 mm

With the probe tip submerged 20 mm into at 80% amplitude, (*Figure 28)* cavitation activity is seen evolving at a minute scale, and at incredible speeds. The elapsed time from first to last image is only 512 µseconds ($5.12x10^{-4}$ seconds). Cavitation did not occur at 40% or 60% amplitude.

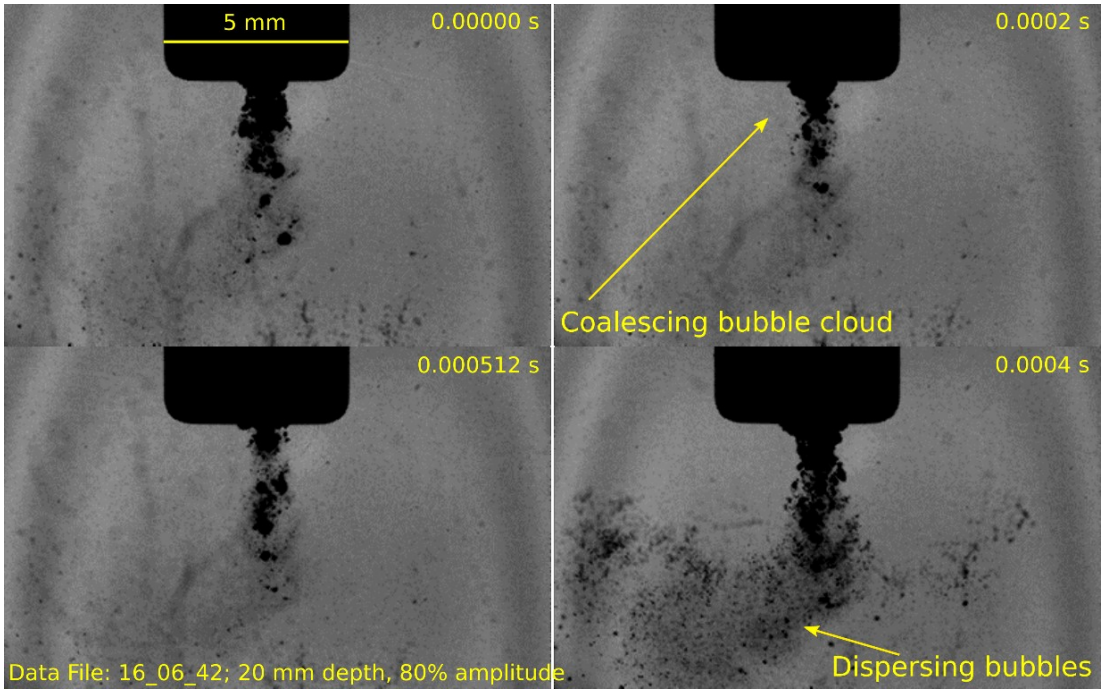

Figure 28 Cavitation activity of the probe over 512 µseconds, while submerged at 20 mm, with no near surfaces, operating at 80% amplitude.

7.4.2 Probe Depth of 5 mm

At 5 mm depth, cavitation was detected at all amplitudes (*Figures 29, 30, 31)*, with the same time elapse of 512 µseconds between the first and last image frame. In the images below, the circular ripples between frames show a shockwave moving rapidly away from a cavitation collapse.

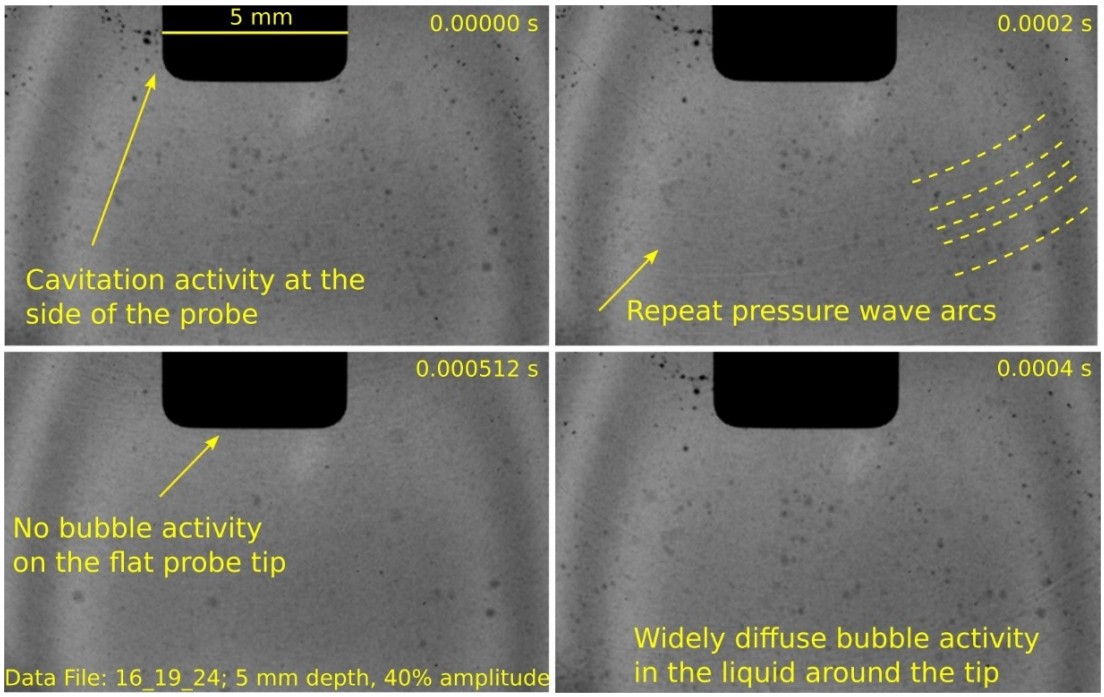

Figure 29 Cavitation activity of the probe, submerged at 5 mm, operating at 40% amplitude.

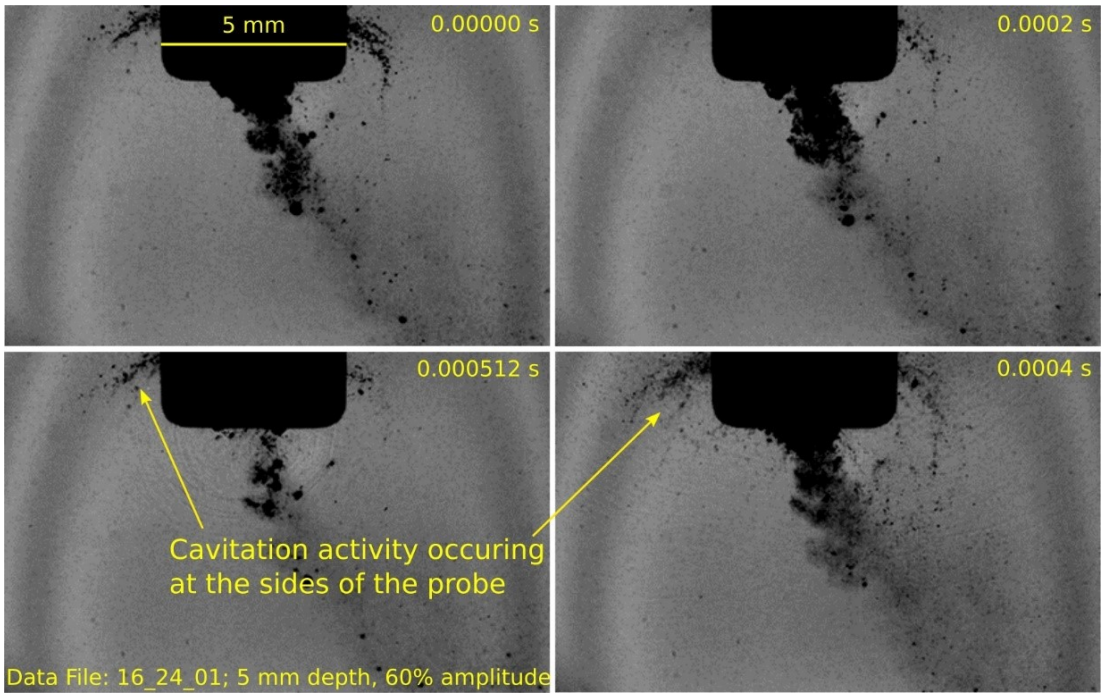

Figure 30 Cavitation activity of the probe, submerged at 5 mm, operating at 60% amplitude.

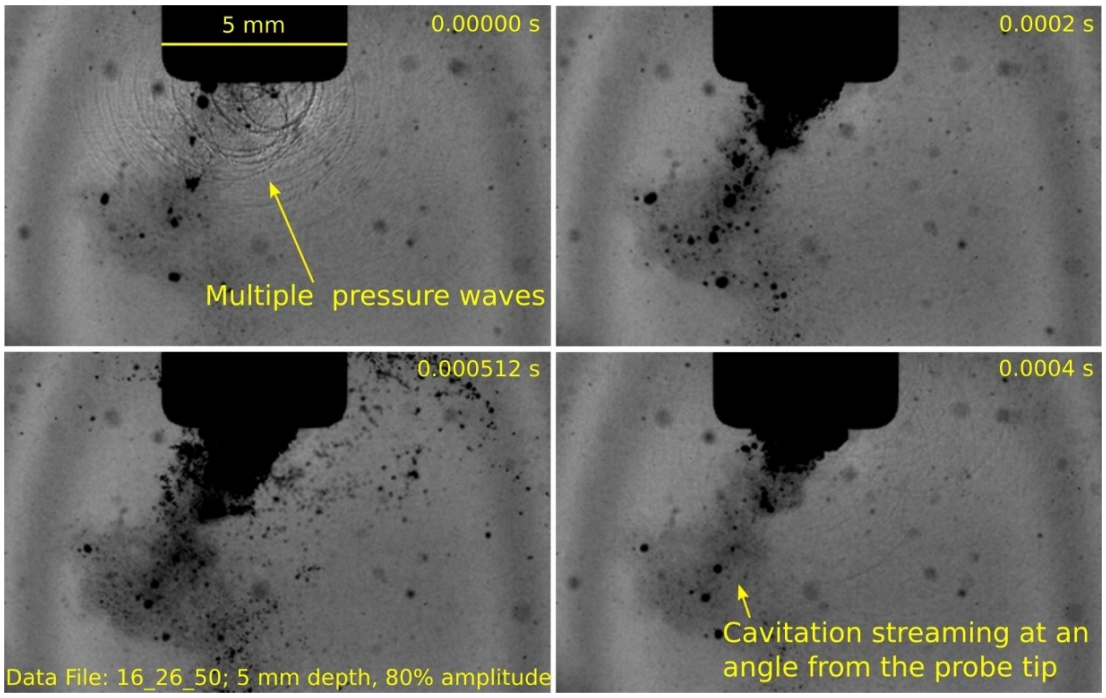

Figure 31 Cavitation activity of the probe, submerged at 5 mm, operating at 80% amplitude.

7.5 Results: Effects of Near Surface on Cavitation Activity

An aluminium plate approximately 3 mm thick was introduced below the ultrasonic probe tip. The probe tip was submerged approximately 20 mm into the tank, as in *Chapter 6*, and the introduced surface of aluminium was placed 10 mm or 15 mm away from the probe tip. Imaging was done if cavitation was detected (*Table 6*).

Depth of Probe Tip in Solution	Distance from Probe Tip to Surface	Amplitude %	Cavitation Detected
20 mm	10 mm	40	No
		60	No
		80	Yes
	15 mm	40	No
		60	Yes
		80	Yes

Table 6 Cavitation detection and imaging according to depth of probe, percent amplitude, and distance to surface.

7.5.1 Near Surface at 10 mm

With the probe tip submerged 20 mm, a 3 mm thick aluminium plate was introduced 10 mm away, parallel to the probe tip. As in the earlier experiment of 20 mm submerged depth, cavitation only occurred at 80% amplitude, however, cavitation bubble clouds spread across the surface widely, and cavitation was also seen on the side of the probe (*Figure 32*).

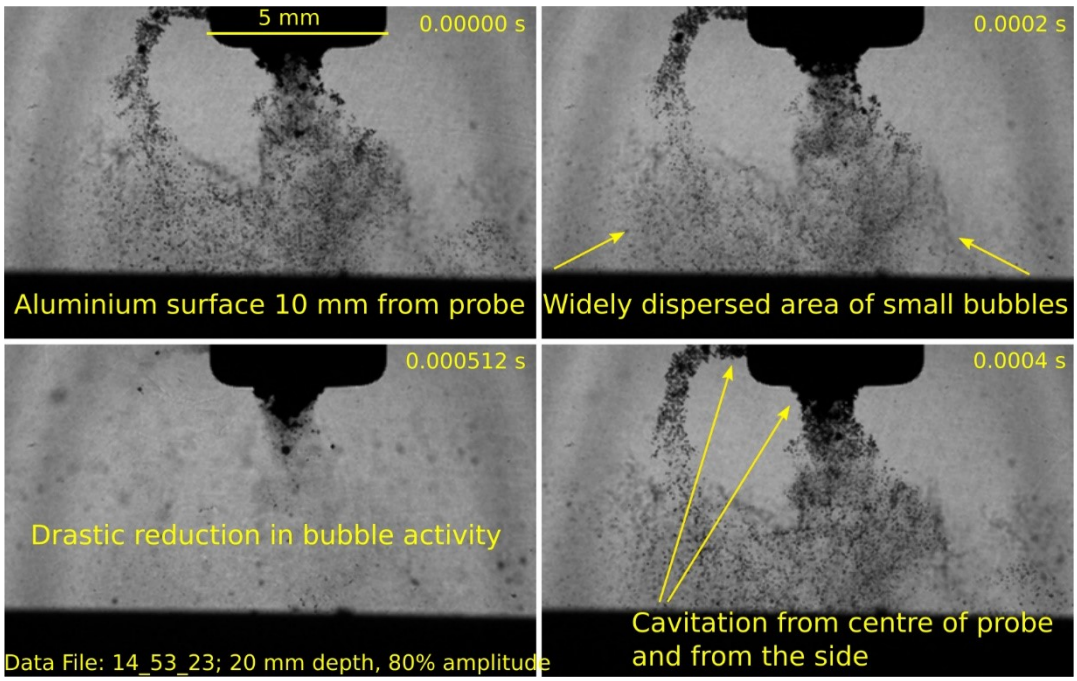

Figure 32 Cavitation activity of the probe, submerged at 20 mm with a near surface 10 mm away, operating at 80% amplitude.

7.5.2 Near Surface at 15 mm

With the probe tip still submerged at 20 mm, the aluminium plate was then moved to 15 mm below the probe. The movement of the surface 5mm further away enabled cavitation at 60% and 80% amplitudes (*Figures 33, 34)*. Due to the imagining restrictions a wider view was not possible at this magnification – the aluminium plate was just outside the frame at the bottom of each image.

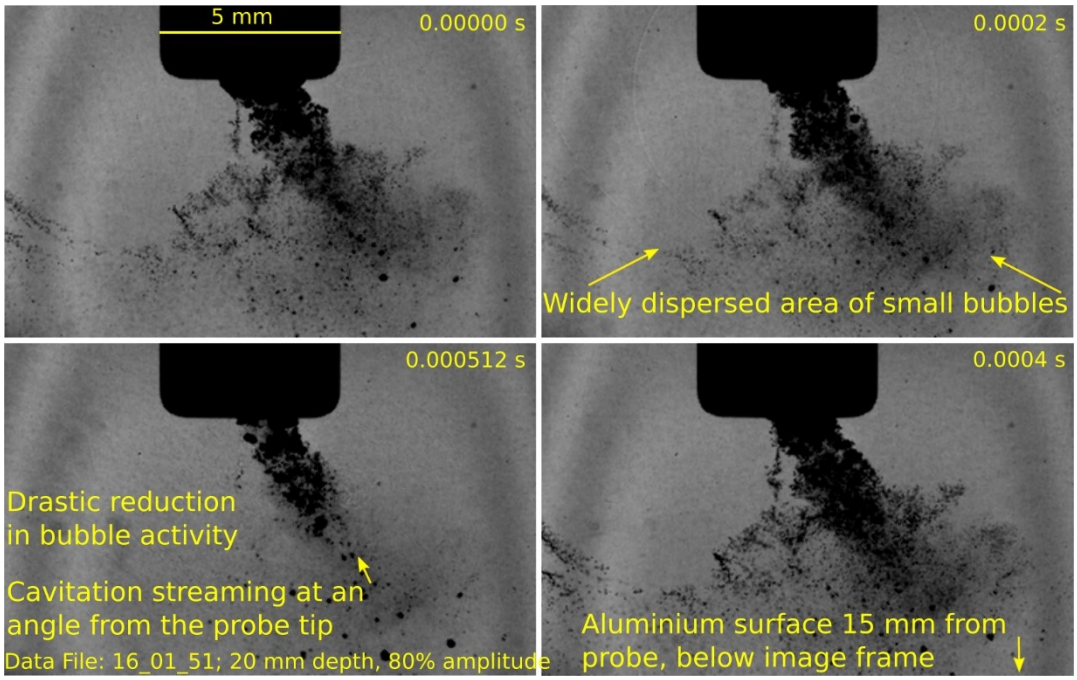

Figure 33 Cavitation activity of the probe, submerged at 20 mm with a near surface at 15 mm, operating at 80% amplitude.

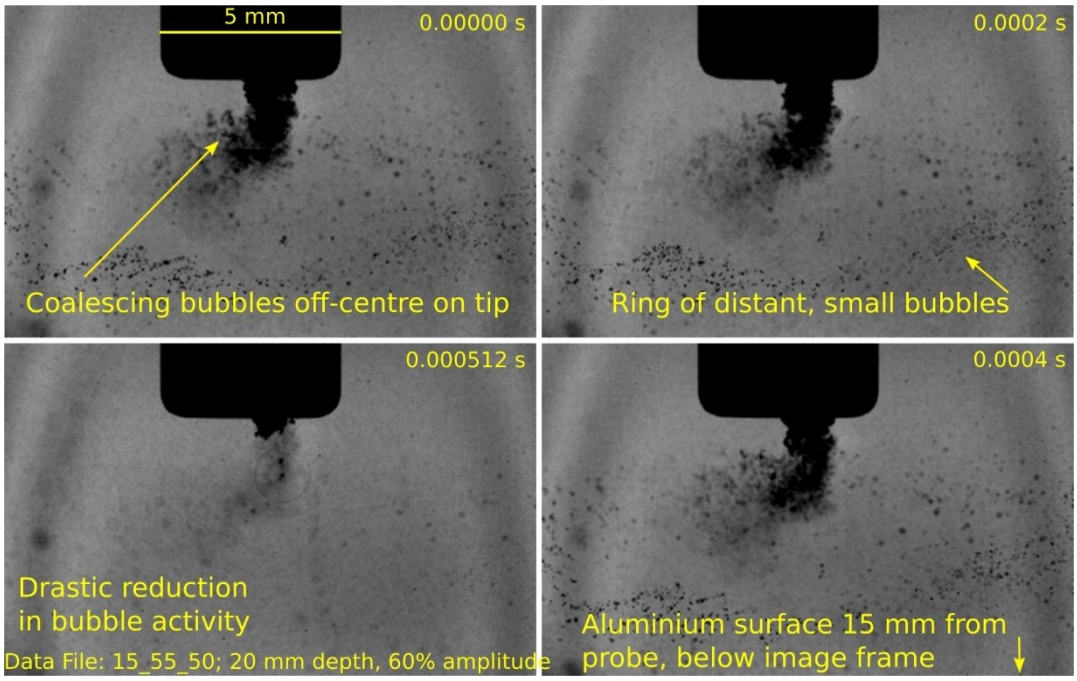

Figure 34 Cavitation activity of the probe, submerged at 20 mm with a near surface at 15 mm, operating at 60% amplitude.

7.5.3 Pressure Wave Speed

The speed of sound is fixed according to the media it flows through, and pressure waves can be visible in liquids. If wave speed surpasses the speed of sound in a medium, it is called a shockwave.[76] Pressure waves (*Figures 35, 36*) were analysed[77] to further quantification the energy output of the device (*Table 7*).

Data File	Element	Frame Number	Frames per second	Elapsed seconds	Diameter in mm	Speed
14_53_01	Cavitation bubble before collapse	1	500,000	0.000000	0.135	-
14_53_01	Pressure wave	4		0.000006	5.468	911 m/s
16_13_39	Pressure wave	103	5,000,000	0.0000206	.399	-
16_13_39	Pressure wave	106		0.0000212	1.139	1,233 m/s

Table 7 Calculations of size, area, and speed of cavitation bubble and collapse from select image data files.

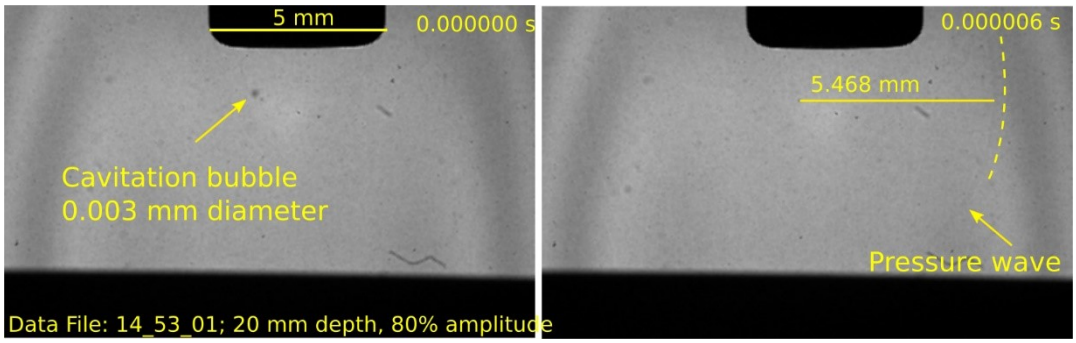

Figure 35 Measured diameter of cavitation bubble, and the diameter of the resulting pressure wave, used to measure speed of the pressure wave away from the centre of cavitation collapse at 500,000 fps.

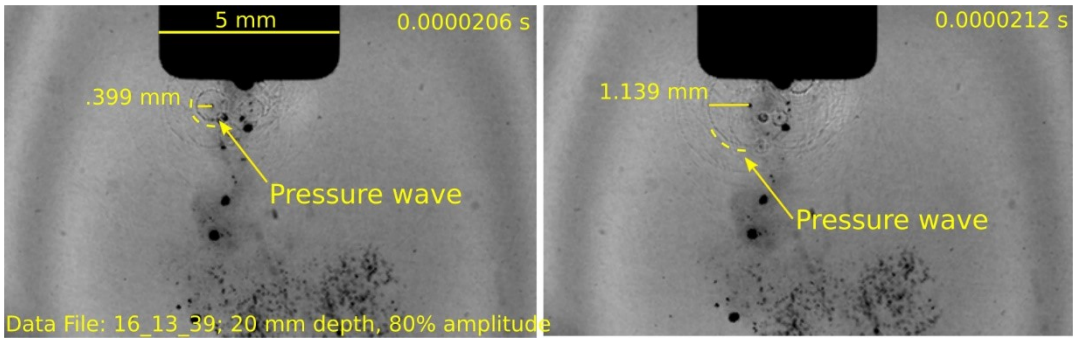

Figure 36 Measured diameter of a pressure wave used to measure speed of the pressure wave away from the centre of collapse at 5,000,000 fps.

[76] Rossing, Moore, and Wheeler, *Science of Sound*, 47.
[77] ImageJ, Java-based, free open-source image analysis software supported by the U.S. National Institute for Health, https://imagej.nih.gov/ij/features.html.

7.6 Discussion

The load force (liquid pressure) on the probe increases as the probe is submerged deeper into a liquid. This pressure inhibited the ability of the probe tip to vibrate. This in turn inhibited cavitation at low amplitudes on this device. Probe submergence depth of ≤10 mm is expected to be common in textile conservation, where shallow wash baths are common. This will result in minimal submergence of the probe, which in turn increases cavitation activity near the surface being cleaned, increasing potential cleaning or damage of a historic textile.

The introduction of an aluminium surface allowed for consideration the bottom of the wash tank as an active part of the cleaning environment that can alter the cavitation bubble cloud size and shape. A solid metal surface that reflects the ultrasonic waves will likely have a different effect on cavitation compared to a surface that readily attenuates the energy of acoustic fields, like a textile. Additionally, a surface that allows flow of liquid through it (such as a woven textile), is likely to have a different effect than a solid surface. Flexibility and density of an object are also likely to exert effects on the acoustic environment and in turn effect the ability of the probe to induce cavitation in the liquid.

The maximum speeds calculated for two pressure waves were both subsonic, occurring at speeds less than the average speed of sound in water at ambient temperatures and pressures. This does not preclude supersonic shockwaves from occurring, but none were calculated from this experiment. The speed of sound varies within different purities and solutions of water, and other physical aspects of the liquid will change the potential energy of cavitation collapse and pressure waves speeds.

7.6.1 Barrier Layers

At the V&A, Melinex®[78] barrier layers were explored using the same ultrasonic probe as this dissertation. [79] It is likely the material properties of Melinex® that can allow some of the high-energy of ultrasound to pass through. Barrier layers will have effects on the acoustic field of an ultrasonic probe due to the material properties such as the elastic constant and speed of sound through the material,[80] which can reflect, transmit, or diffract the ultrasonic waves. Properties that are desirable to pass through the Melinex® barrier may be the compression and

[78] Polyester film, biaxially oriented polyethylene terephthalate.
[79] Hackett, “Ultrasonic Cleaning,” 2018.
[80] Byoung Wan Lee et al., "Acoustic Anisotropy of Oriented Polyethylene Terephthalate Films Studied through Brillouin Light Scattering," *Journal of Information Display* 15, no. 4 (2014): 201–5, accessed 10 March, 2018, doi:10.1080/15980316.2014.971888.

rarefaction of transverse waves that results in cavitation, but the speed at which the transverse waves move may be slowed or change direction as they pass. Other effects may be desirable to deflect, such a directional current of liquid and high-speed particles. Thermal energy may be absorbed by Melinex® barriers, allowing the energy to attenuate horizontally for more controlled dissipation, rather than the textile receiving the thermal energy directly. Vertical restriction of the textile with an impermeable barrier would also limit the movement of loose fibres and yarns in a textile wet cleaning environment.

7.7 Conclusion

High-speed imaging gave context to the observations of the first two experiments. The variability of damage to aluminium foil, and the varying levels of cavitation noise were better understood through imaging of cavitation nucleation, growth, and collapse. This experiment allowed for visualisation of the complex, high-speed evolution of cavitation bubbles within the liquid, and provided data on the cavitation potential of the probe as related to submerged depth, and the proximity of a near surface.

Probe depth was a significant factor in the ability of the probe to produce cavitation. Minimal submergence of the tip of the probe into a liquid corresponded with increased ability for cavitation to occur. The introduction of a surface at 15 mm increased the ability for cavitation to occur. This may be related to reflection or diffraction of the sound waves or thermal energy that disrupts liquid pressure.

Repeated imaging showed a wide variety of cavitation patterns, bubble effects, and pressure waves for the variables. This supports the complexity of the environment having significant impact on cavitation and therefore cleaning or damage potential of ultrasound. The addition of other variables in textile wet cleaning, such as speed and direction of movement of the probe when held by hand are expected to further complicate the environment, which will in turn impact cleaning and damage potential.

8 Phase 1: Changes in Temperature and pH

8.1 Introduction

Thermal energy and potential for catalysing chemical reactions occur from cavitation collapse.[81] In turn, increased temperatures and the generation of free radicals[82] can impact pH. Temperature and pH are monitored and controlled in conservation wet cleaning procedures,[83] making exploration of these factors vital for ultrasonic cleaning of historic textiles.

8.2 Aims

The aim was to determine if temperature and pH changes of the ultrasonic probe could be quantified using standard conservation laboratory equipment. If changes were detected, a secondary aim was to explore the rate of change, and relationship of temperature and pH changes to amplitude and cleaning solution.

8.3 Methodology

As in *Chapter 6*, the ultrasonic probe was placed on a stand in the centre of a cylindrical metal beaker (*Figure* 37), using the same six solutions of 100 mL. An ultrasonic field was applied for 10 minutes for each test, using variables of solution and temperature (*Table 1*).

Readings of temperature and pH were taken with Hanna Instruments (HI) digital meter.[84] Before each experiment, meters were kept in the test solution until the pH and temperature stabilised for at least 15 seconds. The pH probe was submerged in a beaker of test solution, and the ultrasound probe was placed in cold water to cool the probe to ambient temperatures between tests. Readings of temperature and pH were taken during a 5-10 second pause in the ultrasonic pulse at two-minute intervals. Fisherbrand™ pH test strips and a glass thermometer were also used to confirm digital readings at the beginning and end of each test.[85] All testing was performed at ambient temperature (23-26 °C, 40-55% RH).

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[81] Timothy J. Mason, "Ultrasonic Cleaning: An Historical Perspective," *Ultrasonics Sonochemistry* 29 (2016): 519–23, accessed 21 December 2017, doi:10.1016/j.ultsonch.2015.05.004.
[82] Chahine, "Modeling of Surface Cleaning," 528-549.
[83] Tímár-Balázsy and Eastop, *Chemical Principles*, 207-208.
[84] HI instrument 9124 pH meter fitted with HI 1230B plastic body (PES), double junction, gel-filled, combination pH electrode with metal thermometer probe. Accuracy ±0.01 pH and ±0.4 °C.
[85] Fisherbrand™ (2.0-9.0 range), hydrophobic stick, covalently bonded pH strips.

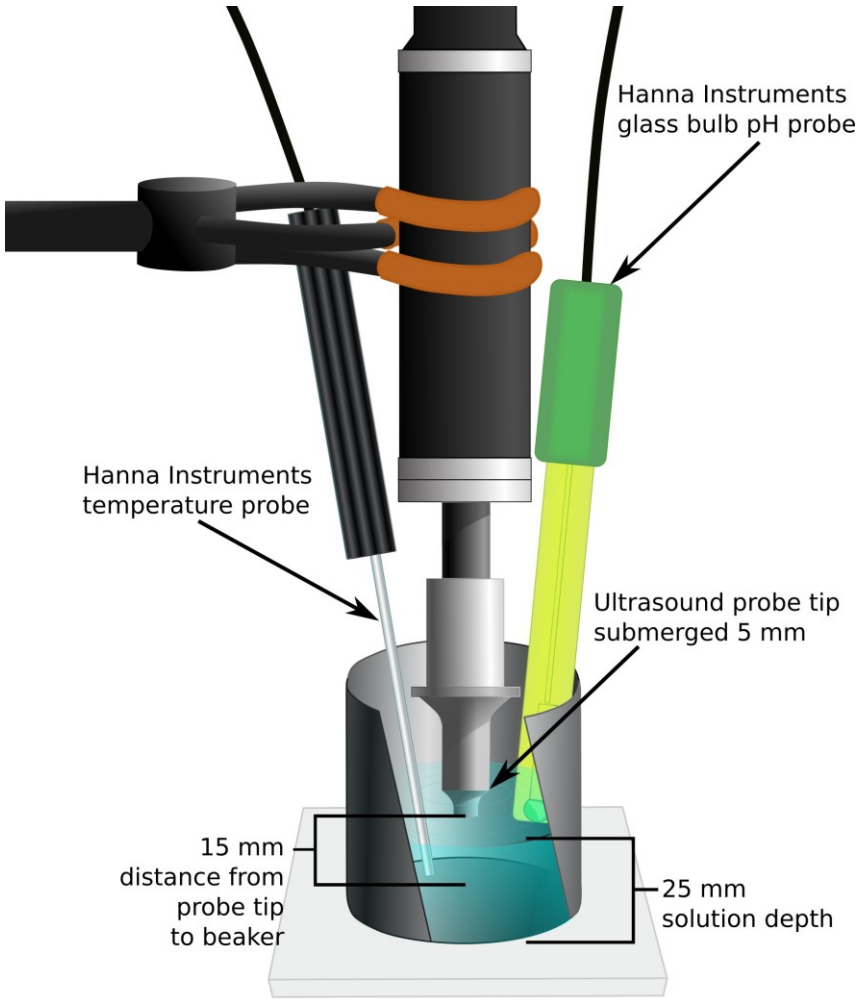

Figure 37 Diagram of testing set up for measurement of temperature and pH.

8.4 Results

8.4.1 Temperature

Temperature increased steadily during each test, and higher amplitudes resulted in more rapid increases in temperature. There was little difference in temperature gain between water purities or solutions (*Table 8* and *Figure 38*). For surfactant Dehypon® LS54, the cloud point reached at 60% and 80% amplitude (*Figure 39*).

Solution	% Amplitude	Temperature °C and Minute Tested						Total Temperature Increase
		0	2	4	6	8	10	
All Waters	40	24.2	25.1	26.3	26.8	26.8	27.3	3.1
	60	24.7	28.0	33.3	35.5	35.5	37.3	12.6
	80	25.1	33.8	36.7	38.8	38.8	40.4	15.3

Table 8 Average measured temperature of all water solutions (at a range of ambient temperatures [23-26 °C]) per minute tested.

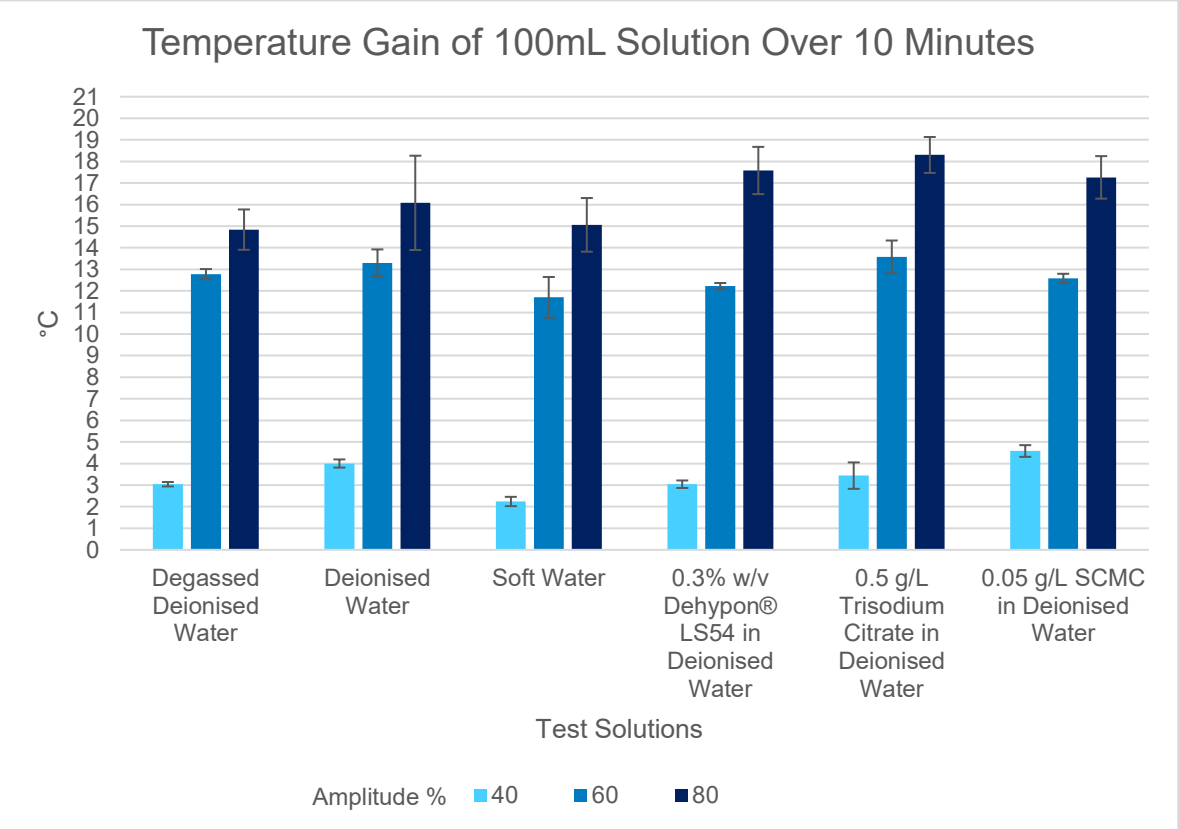

Figure 38 Chart of the averaged measurable temperature change of five replicate tests of ultrasonic exposure at 40%, 60%, and 80% amplitude for 100 mL of solution over 10 minutes.

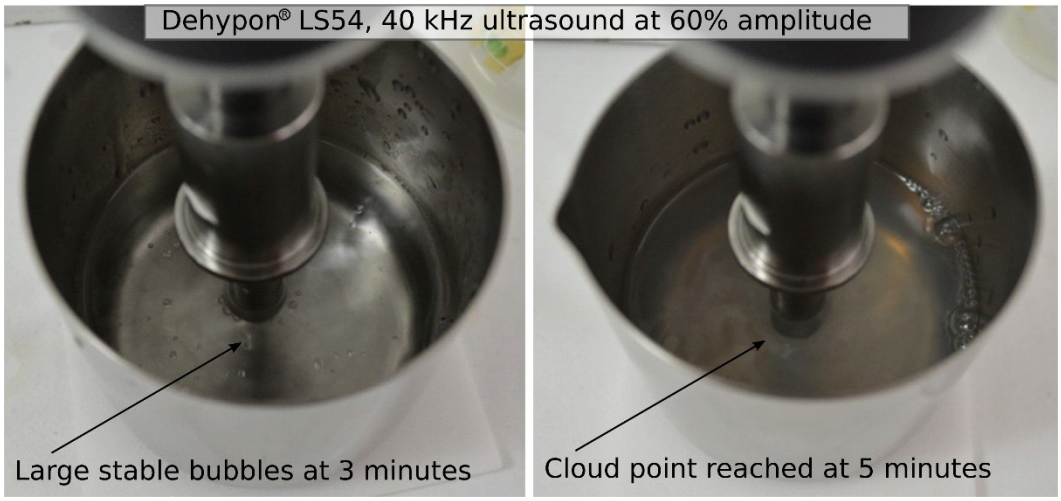

Figure 39 Initial stable bubble formation seen in clear Dehypon® LS54 at 3 minutes changed to limited bubble formation and cloudy solution the moment cloud point is reached (28-30 °C) after 5 minutes exposure to 40 kHz ultrasound at 60% amplitude.

8.4.2 pH

As expected, pH measurements of pure waters were highly variable, resulting in high standard deviations of replicate tests, and low statistical significance of any relationships to variables. Results from soft water and dilute aqueous solutions had less variance in pH due to their higher ionic concentration. However, the averaged results of each set of five readings still had high standard deviation. No statistical significance was present in the readings within or between groups. Further evaluation of the data (*Appendix D)* was not performed.

8.5 Discussion

8.5.1 Temperature

Temperature readings with a metal probe resulted in no meter reading disturbance when ultrasound was in use. This open system was similar to textile wet cleaning procedures but removed the ability for exact calorimetric analysis of the changes in temperature. In conservation practice, the quantities of water used, and surface area of the solution are likely to be higher than 100mL, giving the thermal energy a larger volume and area to dissipate quickly. However, this does not mean that a textile will not be affected by rapid localised changes in temperature.

Overall, this experiment quantified thermal energy gain for the ultrasonic device and revealed a highly significant relationship to amplitude. The change in temperature as related to amplitude were highly statistically likely, with a p-value of 5.0838E-41. The wide range of ambient temperature (23-26 °C) on different testing

days was the likely source of the variation in total temperature change between the three solutions. Averaging the temperature change across all tests gave an accessible, useful metric for estimating localised thermal energy input by an ultrasonic device to a wet cleaning environment (*Table 9)*.

Solution	% Amplitude	Thermal Energy °C/second	Thermal Energy °C/30 seconds	Thermal Energy °C/60 second
All Waters	40	0.005	0.15	0.31
	60	0.021	0.63	1.126
	80	0.0255	0.765	1.53

Table 9 Averaged measurable thermal input (°C) of 40 kHz ultrasound per second to three purities of water in an open metal vessel according to percent amplitude.

8.5.2 pH

Analysing the results of the pH metering of three purities of water was complicated by the known difficulties of measuring pH of water with little or no ionic concentration. Without ionic species (e.g. Ca^{2+}, Na^{+}, Cl^{-}), both digital and paper pH measurements can show lower pH readings, erratic drift, or unstable digital measurements.[86] The presence of air bubbles and extreme convection in the liquid which are both caused by ultrasound can also give unstable and lower readings of pH.[87] The difficulty in getting accurate readings resulted in only taking readings when ultrasound was switched off. The temperature dependence of pH also required a meter that adjusted the pH based on the changing temperature. Even this with, the variability of pH readings results in a data set that revealed no significant relationships or patterns.

Extreme shifts in the digital pH readings were seen with the pH probe immersed into the liquid near the ultrasound probe during use. This could be due to high-frequency disturbance of the pH metering equipment, or the rapid convection and cavitation resulting in bubbles of gas and vapour within the water. Paper pH strips separated from the stick when immersed near active ultrasound, likely due to cavitation. The volatility of the wash bath was also a potential source of damage to the thin glass bulb.

[86] Season Tse, *Technical Bulletin 28: Guidelines for pH Measurement in Conservation,* (Ottowa ON: CCI, 2007), 1-23.
[87] Hach Company, "Technical Note: pH Measurement in High Purity and Low Ionic Strength Water," 2012, accessed Accessed 12 June 2018, https://www.hach.com/asset-get.download-en.jsa?code=99770.

8.6 Conclusion

8.6.1 Temperature

This experiment quantified a steady increase in temperature over time when using ultrasound. The rate of temperature gain increased with higher amplitude. Temperature increases may have potential damage or change of dyes, and fibres. Additionally, the cloud point of surfactants and pH range of buffers may be altered outside of desired ranges.[88] Rapid attenuation of the thermal energy will be greater in the shallow wide wash baths that are common in textile conservation, but the localised increases in thermal energy are occurring, and must be considered before applying ultrasound to a historic object. For conservators in practice, quantifying the thermal energy input and rate of ultrasonic devices is recommended to assist in decision-making and risk-management for ultrasonic cleaning.

8.6.2 pH

It was concluded from this experiment that standard pH meters available to practicing conservators were not suitable for detecting changes in the chemical composition of purified water or dilute aqueous solutions from ultrasound. This does not mean however, that these changes may not be taking place. The findings of this experiment indicate that monitoring of pH during a wet cleaning procedure should not be done in close proximity to an ultrasonic probe when the ultrasound is on. Otherwise, the glass bulb may be damaged, paper pH strips may float in the bath, and aberrant readings are likely.

[88] Tímár-Balázsy and Eastop, *Chemical Principles*, 202, 207-208, 219-221.

9 Phase 2: Effect of Ultrasound Applied to a Textile Wet Cleaning Environment

Wet cleaning textiles generally describes an immersion process that takes place in a wash-bath of plastic or stainless steel using shallow depths of cool or cold dilute aqueous solutions. Immersion wet cleaning represents a specific type of cleaning environment for testing ultrasound that is not represented in the conservation or ultrasonic cleaning literature.

9.1 Aims

The primary aim of Phase 2 was to investigate potential cleaning or damage to a textile after a time-limited introduction of ultrasound in a simulated wet cleaning environment. A secondary aim was to explore the relationship of amplitude and cleaning solution to cleaning efficacy or damage.

9.2 Methodology

Phase 2 testing was designed to simulate typical textile conservation cleaning environments of rectangular, stainless-steel wet cleaning tables with limited depths of solution. In a 430-gauge stainless-steel tray (203 x 254 x 25 mm), 500 mLs of solution was used to create a 10 mm depth. The high mechanical and thermal action of the ultrasound device seen in Phase 1 led to the selection of short cleaning cycles (*Table 10*).

Process	**Time in Minutes**	**Solution Volume**
Wet out	5	500 mL
Apply ultrasound side 1	1	
Apply ultrasound side 2	1	
Soak in fresh solution	1	500 mL
Apply ultrasound side 1	1	
Apply ultrasound side 2	1	
Soak in deionised water	1	500 mL
Apply ultrasound side 1	1	
Apply ultrasound side 2	1	
Static rinse of deionised water	5	500 mLs
Running rinse of deionised water	2	Approximately 750 mLs
Total time of ultrasound application	**6 minutes**	
Total wash and rinse time	20 minutes	

Table 10 Experimental wet cleaning process for Phase 2.

The ultrasound probe was hand held with the tip of the probe just under the surface of the solution (<5 mm) at a slight angle (70-80 ºC) as was comfortable for the duration of cleaning. A slow, S-pattern of movement with the tool, following the grain of the warp and weft was used to ensure even coverage.[89] To prevent the drifting or rising of the small textile sample in the wet cleaning bath, a circle of 30 mm in diameter was cut from the bottom of a thin plastic weigh boat and used as a cleaning template (*Figure 40)*. This template held the textile in place without physical contact with the solution during ultrasonic cleaning. The speed and pattern of probe movement allowed for four total passes over the template area on the textile over one minute.

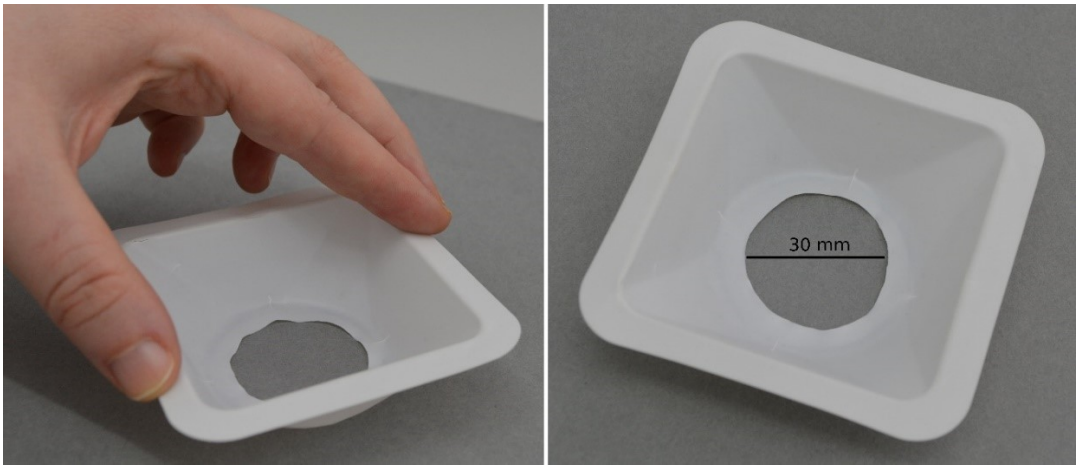

Figure 40 Plastic weigh boat used as a template and weight in ultrasonic cleaning experiments.

9.2.1 Test Fabric

Cotton was chosen as it has increased strength when wet, which can lower the potential for damage in a wet cleaning treatment. A modern, excellent condition, plain-weave fabric of well-twisted, compact yarns would also be considered an ideal candidate for wet cleaning in conservation. The selected test fabric was a balanced plain-weave cotton, of 20 x 16 yarns per centimetre with no discernible warp or weft. This cotton fabric was purchased pre-coated with soiling consisting of olive oil and carbon black.[90]

The soiling appeared largely consistent in colour and volume under macroscopic and low magnification observation. Accumulation of grey oily soiling

[89] Corrado Pedeli, "Cleaning With an Ultrasound Scaler: Technique Adjustment for Archaeological Ceramics," in *Glass and Ceramics Conservation 2007: Interim Meeting of the ICOM-CC Working Group: August 27-30, 2007: Nova Gorica, Slovenia*, ed. Lisa Pilosi (Nova Gorica, Slovenia: Goriški Muzej, 2007), 141–51.
[90] Product C-02 from Center for Test Materials, BV Stoomloggerweg 11,3113 KT, Vlardingen, the Netherlands.

was evenly distributed on warp and weft yarns on both sides of the fabric, with a matted appearance and loose fibres at 10x and 50x magnification. Samples were cut into 50 x 50 mm squares and five replicate samples were randomized into each of the nine testing groups (*Table 11)*.

9.2.2 Cleaning Solutions

Three solutions were selected based on Phase 1 testing that were expected to have low cleaning impacts on the selected soiled cotton with sponging alone. The selected solutions also presented distinctly different physical and chemical properties that could impact cavitation. This selection strategy was done to allow for the impact ultrasound had on the cleaning process to be as visibly different as possible between sample groups.

Deionised water alone was chosen as a control to see the impact of ultrasound without the aid of surfactants or additives tailored to the soiling or fabric.[91] Dehypon® LS54 at 0.3% w/v in deionised water was chosen as it was seen to have a small impact on cleaning with sponging alone on a similar test fabric.[92] A third solution combined 0.5 g/L trisodium citrate and 0.05 g/L SCMC in deionised water. Trisodium citrate was shown to have some impact on the release of oily soiling on cotton, while acting as a pH buffer.[93] SCMC is a soil suspender and emulsifier for particulate and oily soiling. This combination solution was a suitable cleaning choice for the soiling and cellulose fibre content of the test fabric but would not be expected to have as high a cleaning efficacy as an anionic surfactant.[94] All testing was done at ambient conditions (21-25 °C, 45-50% RH).

Solution	**Amplitude %**		
	40	**60**	**80**
Deionised water	Group 1	Group 2	Group 3
0.3% Dehypon® LS54	Group 4	Group 5	Group 6
0.5 g/L Trisodium citrate, 0.05 g/L SCMC	Group 7	Group 8	Group 9

Table 11 Nine test groups according to variables of solution and percent amplitude.

[91] Sato, "An Experimental Evaluation," 40-46.
[92] Ibid., 40-52.
[93] Nora Frankel, "An Investigation into the Use of Candida Rugosa Lipase for Removal of Aged Oils from Cotton Textile," (master's dissertation, University of Glasgow, 2016), 46-51.
[94] Tímár-Balázsy and Eastop, Chemical Principles, 195-198.

9.3 Analytical Methods

Each fabric sample was analysed before and after treatment to determine cleaning efficacy and damage. Analytical methods included visual analysis, low-magnification microscopy,[95] mass,[96] and colourimetry.[97] Temperature and pH measurements were taken of the solutions throughout the experiment with the HI9124 digital pH and temperature meter described in *Chapter 8*.

9.4 Results

9.4.1 Temperature and pH

The short cleaning times and wide shallow bath resulted in stable temperatures that generally reflected the ambient temperature. Only slight rises in temperature of 1-2 °C were seen across all experiments, and pH changes were very minimal in the range of ±0.25 points. The size and shape the bath allow for the temperature and pH probes to be placed ≥50 mm away from the ultrasound probe. No interference or anomalous readings were seen in Phase 2, so no further analysis was pursued of this dataset (*Appendix E)*.

9.4.2 Visual Evaluation of Soil Removal

The even distribution of soiling on the standard test fabric and the short cleaning times allowed for visible variance in ultrasound effects. Visual analysis showed a large variance in level of soil removal, evenness of soil removal, and reproducible results between and within the samples in each of the nine test groups.

The circular cleaning template confirmed the localised cavitation clouds and directional movement seen in Phase 1. All samples showed some cleaning within the circular template of the weigh boat, and little change in soiling outside the template. Samples with the most soil removal and most even reproducibility within a test group were seen with the trisodium citrate and SCMC solution at 40% amplitude (*Figures 41, 42)*. In this group, and all others, the first sample was notably less clean than the rest of the samples in the same group.

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[95] Zeiss Stemi-2000C stereomicroscope 0.65x–5x.
[96] Sartorius BP150 analytical balance, accuracy: 0.001g.
[97] Konica-Minolta CM-2600d spectrophotometer, D65 daylight illuminant with Specular Component Included (SCI) measurements; J. Schwiegerling, *Field Guide to Visual and Ophthalmic Optics*, (Bellingham, WA: SPIE Press (2004), accessed 13/08/2018, http://spie.org/publications/fg04_p71_cielab?SSO=1.

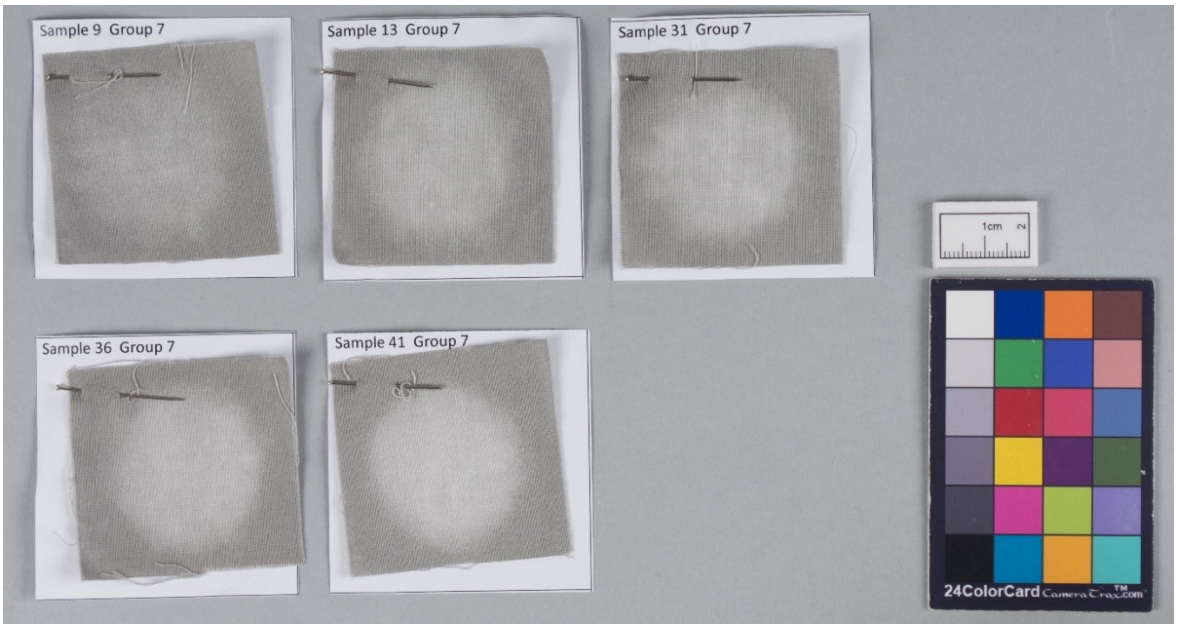

Figure 41 Group 7, trisodium citrate and SCMC at 40% amplitude showed the most visible, even cleaning results.

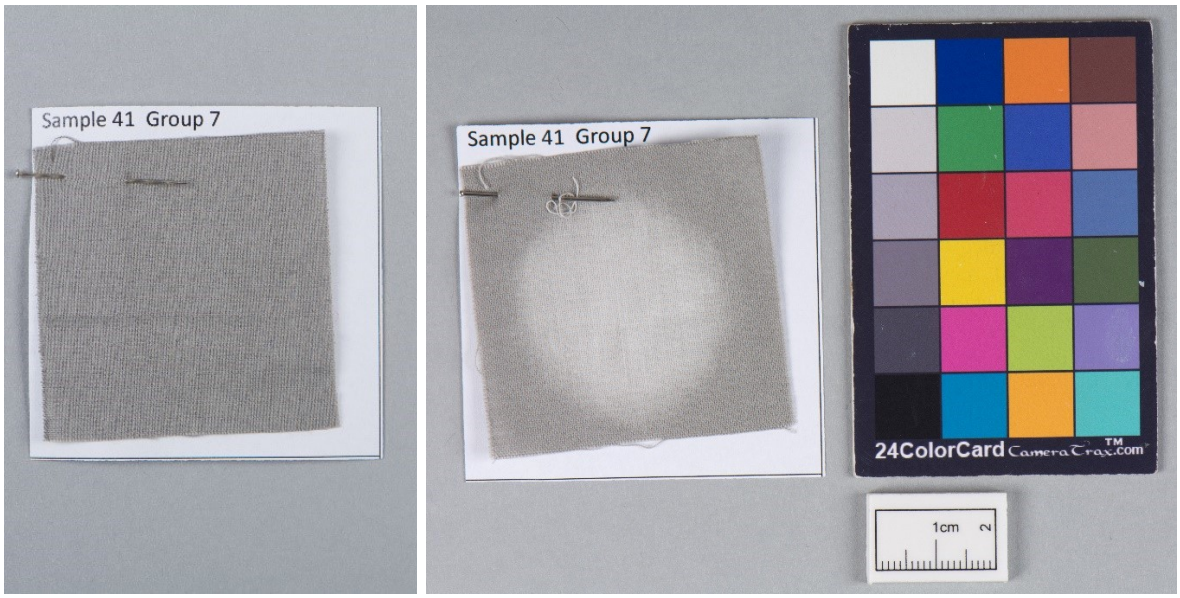

Figure 42 The most soiling release of a single sample in Group 7 was seen in Sample 41, before cleaning on the left, and after cleaning with trisodium citrate and SCMC at 40% amplitude on the right.

The most variable, uneven, and lowest cleaning effect was seen in Group 6, Dehypon® LS54 at 80% amplitude (*Figure 43*). Some samples showed low overall cleaning efficacy, while others showed extreme cleaning in highly localised areas, resulting in uneven streaks (*Figure 44*). Images of the remaining groups can be seen in *Appendix E*.

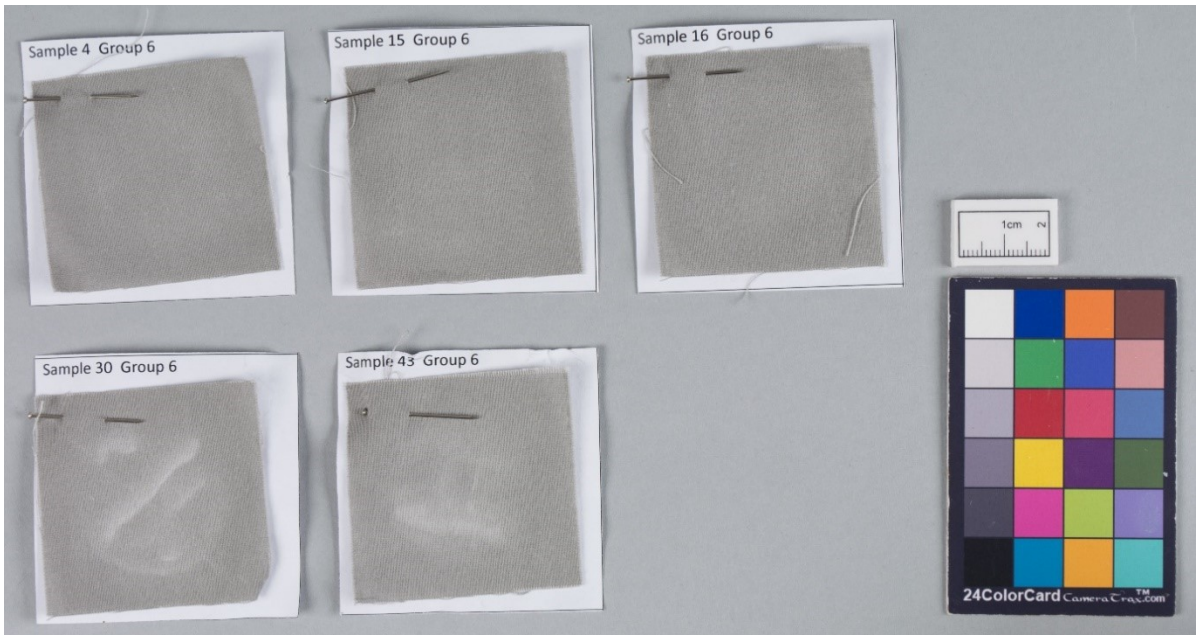

Figure 43 Group 6, Dehypon® LS54 at 80% amplitude showed the least amount of cleaning, with the most variable, uneven results.

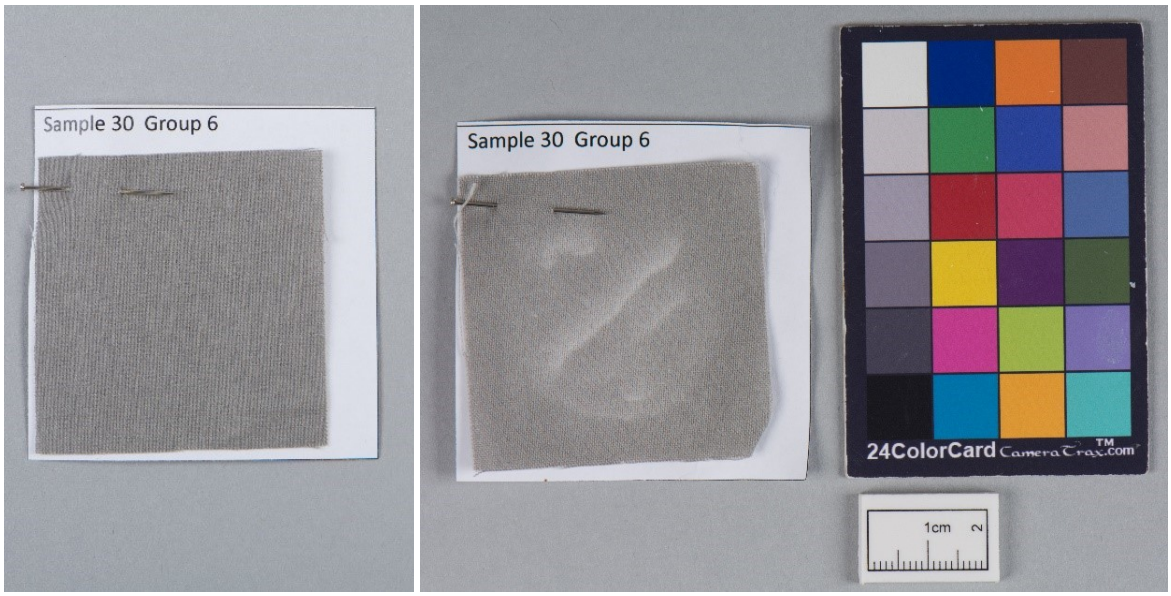

Figure 44 Sample 30 showed the most uneven, streaky cleaning effect with Dehypon® LS54 at 80% amplitude.

9.4.3 Microscopy Analysis of Damage

Low-powered stereomicroscopy showed no negative visual changes. Yarns were tightly twisted and compactly woven both before and after cleaning. On cleaner samples (*Figure 45*), the cotton fibres were more visible after cleaning, with a white, smooth, shiny surface. Samples that were overall less-clean (*Figures 46, 47)* were structurally intact. Loose fibres on the surface were present before and after cleaning, with no damage or notable visual changes to fibres or weave. These samples had a

slightly brighter, glossier, appearance due to some loss of the greasy, dark, surface soiling, but still appeared grey.

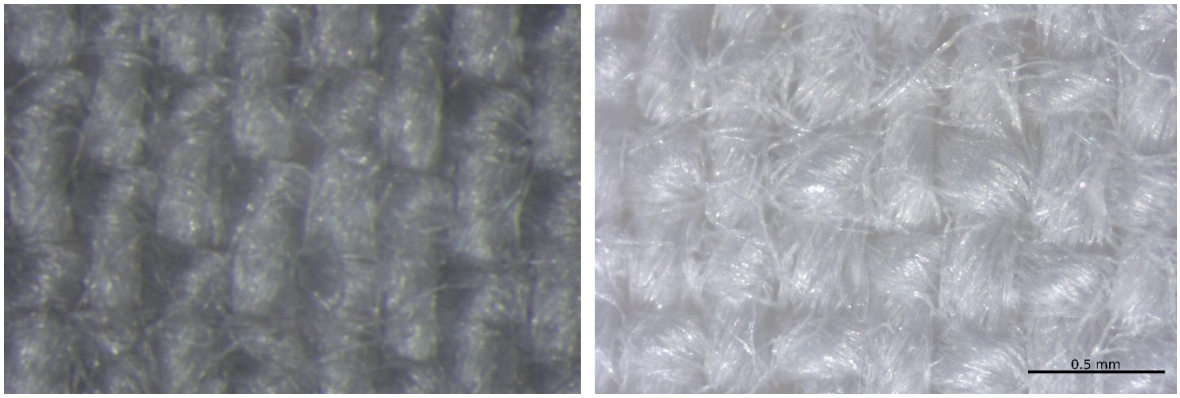

Figure 45 Sample 41 before and after treatment with trisodium citrate and SCMC at 40% amplitude, showing significant cleaning but no damage at this magnification.

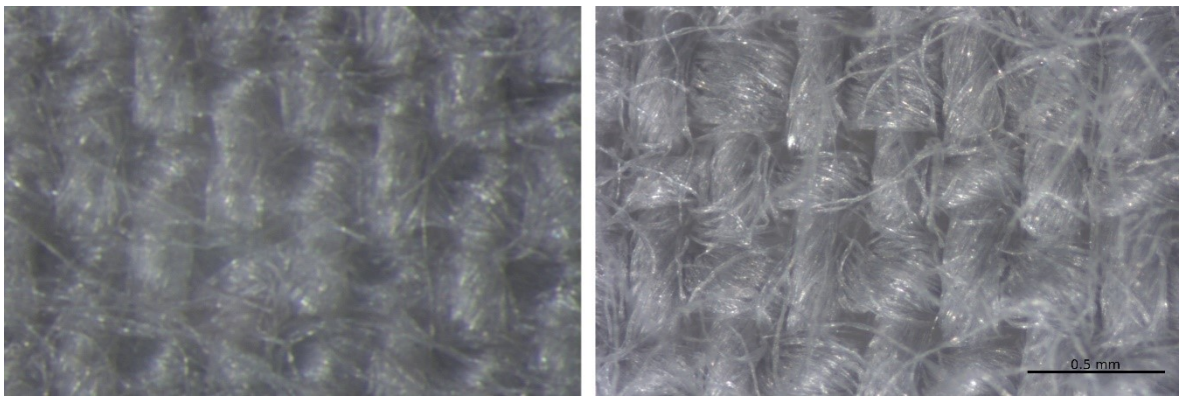

Figure 46 Sample 16 before and after treatment with Dehypon® LS54 at 80% amplitude, showing a small amount of cleaning and no damage at this magnification.

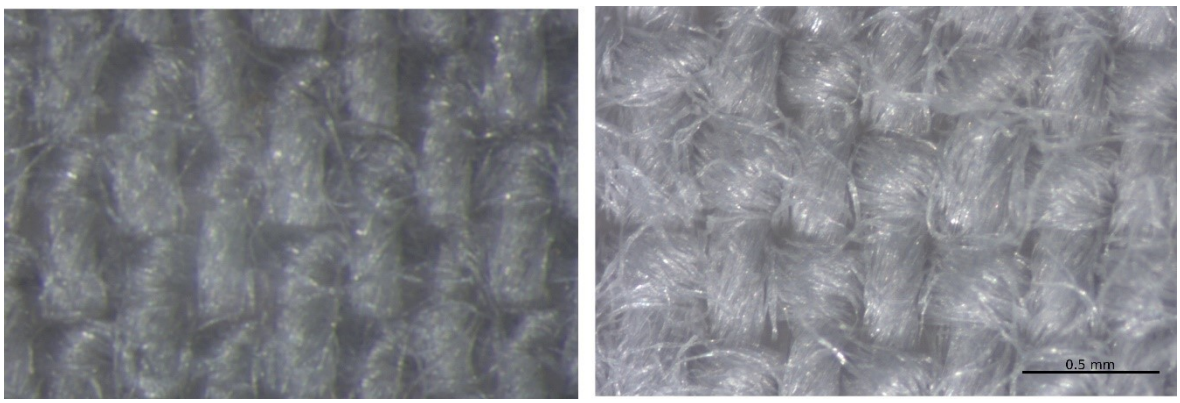

Figure 47 Sample 26 before and after treatment with deionised water alone at 40% amplitude, showing no damage at this magnification. Note the lack of greasy surface soiling despite the lack of solution additive to break up oily soiling.

9.4.4 Change in Mass

Average mass loss per group was detected on a small scale, with high standard deviation (*Figure 48*). However, the mass change correlated with the visual analysis that the first replicate in each sample set was less-clean, and generally not representative of the rest of the set. When the first sample was removed (N=4) for each test group, the average mass loss increased, and standard deviation decreased for each test group (*Figure 49*). However, overall, the scale of the mass change was very close to the accuracy range of the balanced used, which weakened the strength of this analysis.

Two-factor ANOVA for all samples (N=5) gave a p-value of 0.35, indicating there is little significance of the relationship of amplitude to mass loss between the different solutions. Removing the first sample from each group (N=4) lowered the p-value to 0.07. While this p-value still described the high variability of the results, the change in p-value showed the first sample of each set skewed the data considerably. This suggested the impact of amplitude could have more significance.

A subset analysis of the most effective solution overall, trisodium citrate and SCMC was performed to continue exploring the relationship between cleaning efficacy and amplitude. As above, the first sample of each amplitude tested was removed (N=4), which overall increased the average mass loss beyond the accuracy limits of the balance. In this subset analysis, the p-value dropped to 0.02, showing a significance between the relationship of amplitude to cleaning efficacy, where the lower the amplitude, the higher the cleaning efficacy.

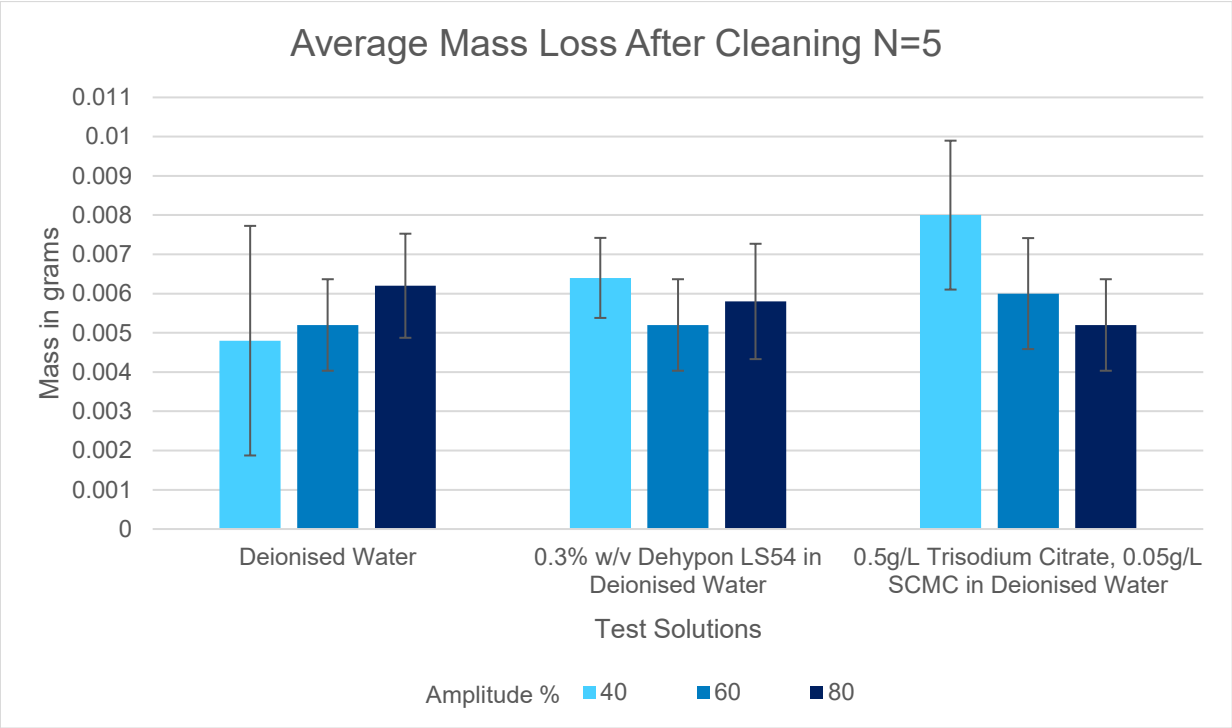

Figure 48 Chart of average mass loss after cleaning analysing all samples tested (N=5).

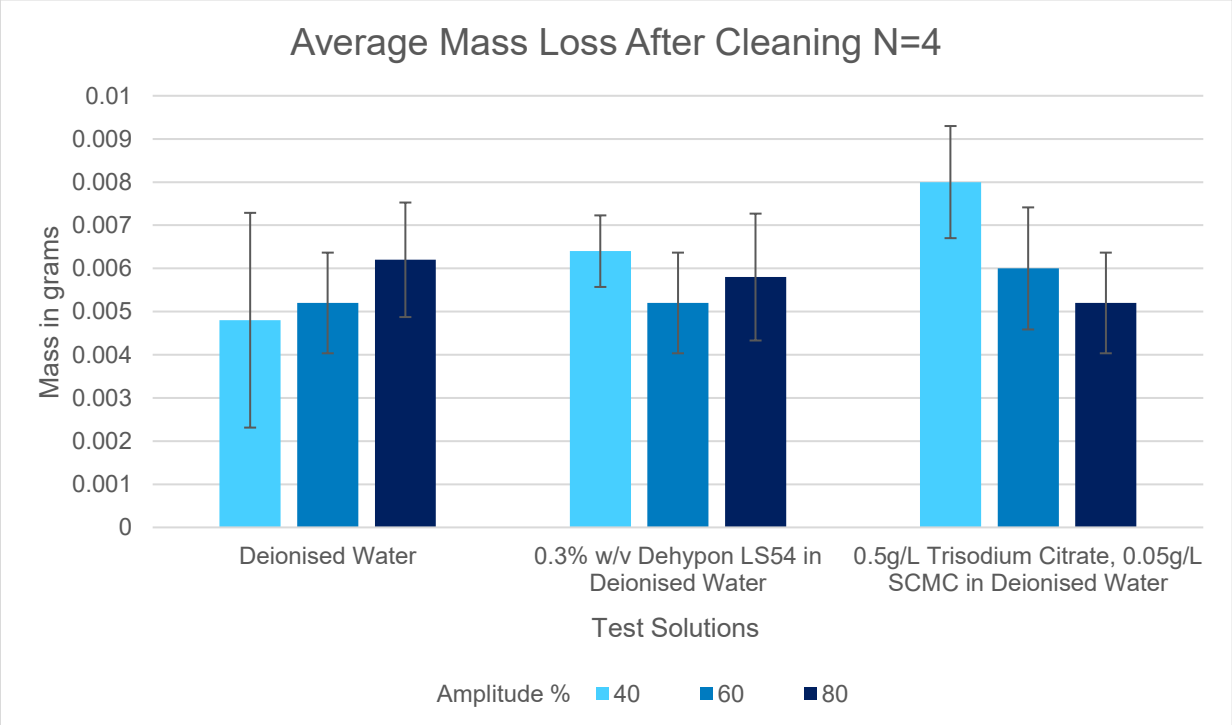

Figure 49 Chart of average mass loss after cleaning with the first sample removed from analysis (N=4).

9.4.5 Colourimetry

Colourimetry was also used to quantify cleaning efficacy based on change in brightness (*Figure 50*). Four measurements were taken in the centre of each sample before and after treatment, using a 3 mm aperture. This disregarded the uneven, streaky effect of the cleaning results and led to a less-variable data set for analysis compared to the mass change data. In the CIELAB colour space, changes in L*, or brightness, correlated with visual increases in cleaning, and mass loss trends.

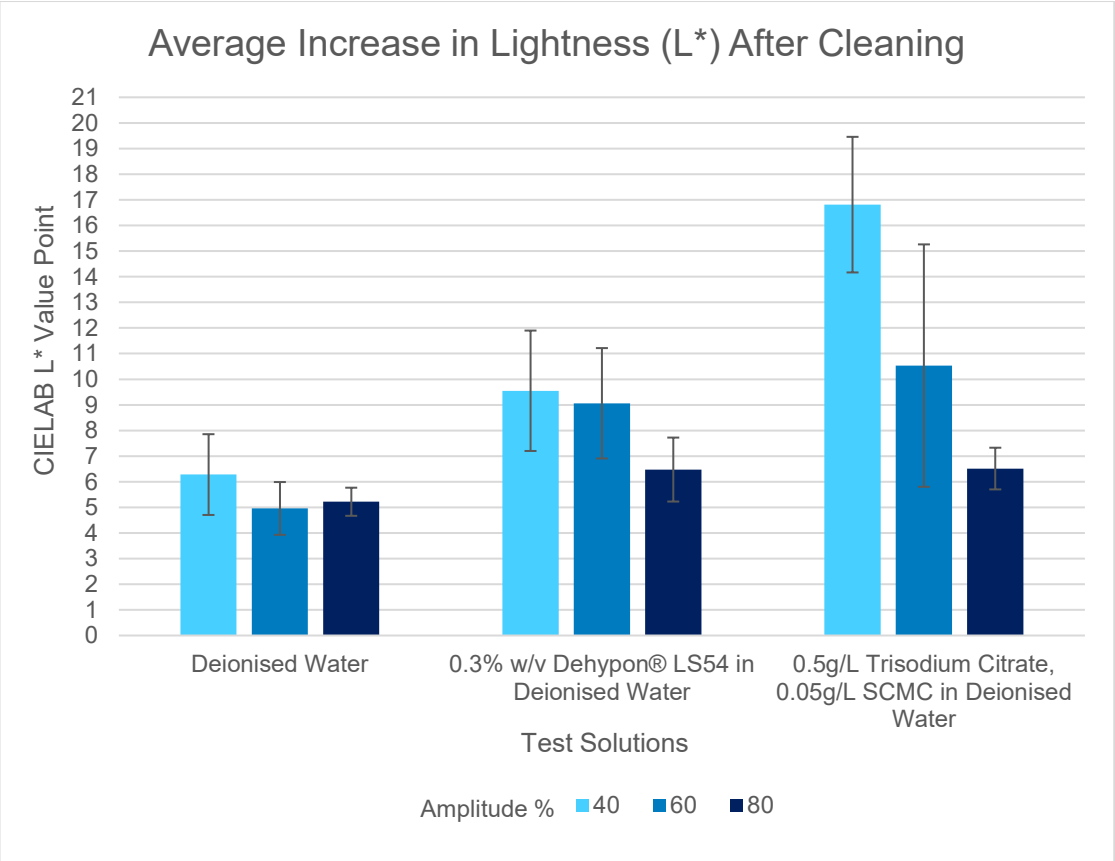

Figure 50 Chart of average increase in brightness CIELAB L value after cleaning.*

9.5 Discussion

The colourimetry data was less-variable than the mass-loss data, due to the limited area measured. As a result, colourimetry showed a highly significant relationship of amplitude and cleaning efficacy. Two-factor analysis of variance showed highly significant p-value of 0.000036, giving statistical strength to visual observations that lower amplitudes resulted in higher cleaning efficacy. Unlike mass, this analysis included the first sample of each set, which was less clean for almost every test group, but still resulted in a very low p-value.

The solution of trisodium citrate and SCMC had the highest overall cleaning efficacy by colourimetry and other analyses. The cleaning efficacy of 40% amplitude was far higher

than 60% or 80%. Visual analysis indicated that the greasy, oily surface soiling was effectively removed for all solutions, highlighting the strong emulsification action of ultrasound.[98] Phase 1 testing indicated that 40% amplitude had the least amount of thermal energy, which suggested the impact of amplitude on cleaning was not related to increased temperature. This also suggested that the higher general agitation of the bath at higher amplitudes that was seen in *Chapter 6* was not related to cleaning efficacy.

9.6 Conclusion

9.6.1 Cleaning Efficacy

The assessment of cleaning efficacy by visual analysis, mass change, and colourimetry confirmed that within the simulated textile wet cleaning environment, the application of ultrasound alone could result in some cleaning of modern cotton textile with oily and particulate soiling. The cleaning efficacy as determined by mass was highest when a highly suitable cleaning solution for the textile and soiling was used. A subset analysis on the mass loss of the most successful cleaning solution revealed that the amplitude of 40% had a higher cleaning efficacy compared to 60% or 80%.

Colourimetry analysis results showed that the amplitude had significant impact on cleaning efficacy, where again, 40% had the highest quantifiable cleaning efficacy. Within this experiment, increasing the amplitude, which increased mechanical and thermal action at the tip of the probe, did not result in cleaner textiles for any solution tested, regardless of cleaning efficacy of the solution. The cleaning efficacy of ultrasound in this experiment could not be attributed to overall temperature increase or pH change of the full wash bath (recorded pH 6.5-7.5, temperature 21-24 °C). However, localised temperature change during ultrasonic cleaning was likely still occurring based on data from Phase 1, and unknown free-radical liberation may be occurring, which would exert an effect on cleaning in the area around the probe.

Looking closely at all data suggests that the low of cleaning efficacy of the first sample may be related to probe's electro-mechanical action. Generally, the first sample of each set was cleaned with ultrasound after a prolonged pause in use or change in amplitude. The lack of cleaning for this sample across groups indicated

[98] Mingming Zhang, Michael Yewe-Siang Lee Shee We, and Hongwei Wu, "Direct Emulsification of Crude Glycerol and Bio-Oil without Addition of Surfactant via Ultrasound and Mechanical Agitation," *Fuel* 227 (September 2018): 183–89, accessed 14 August 2018 doi:10.1016/j.fuel.2018.04.099.

there was a warming up period, or latency in the action of the probe. In practice, using the probe at the intended amplitude for several minutes in a test solution will likely increase reproducible results when cleaning historic objects.

9.6.2 Physical Damage

No damage of the textiles or fibres could be detected through the low-power microscopy methods used for any of the 45 samples, indicating that ultrasound damage is not intrinsically connected to cleaning efficacy in a conservation wet-cleaning environment. Lack of damage is likely due to a combination of factors, starting with the high wet-strength of a modern cotton.[99] The tightly spun, compact plain-weave structure of unaged cotton withstood the high mechanical action of ultrasonic cleaning in a liquid solution in the given testing environment. The suppleness and strength of the fibres meant they did not break or fray, and the circular cleaning template prevented damage to the unfinished edges of the test fabric. The material properties of fibres, yarns, and weave characteristics of an object should be considered in-depth in practice before testing ultrasound.

[99] Tímár-Balázsy and Eastop, *Chemical Principles*, 11-15.

10 Conclusion

10.1 Introduction

One aim of this dissertation was to provide an understanding of ultrasonic cleaning as applied to textile conservation. This was done through a conservation literature review, and research into the physics and chemistry of ultrasound. Overall conclusions were that ultrasonic cleaning relies on a complicated interplay of variables including frequency, amplitude, distances, depths, volumes and temperatures. These variables were not uniformly understood for ultrasonic devices used in the conservation literature, and were often poorly reported in the publications, which stifled research.

Another aim was to characterise the action of a single ultrasonic device using select variables and replicate samples. Findings indicated that each ultrasonic device can be characterised and understood in practice through simple, inexpensive testing with aluminium foil and quantification of temperature. Simulating a wet cleaning environment showed further complexity of cleaning environment and linked low amplitudes to higher cleaning efficacy on the test fabrics for several cleaning solutions. Findings that further impact conservation practice, the design of bespoke ultrasonic cleaning equipment, and future research suggestions are discussed in this chapter.

10.2 Impacts on the Field

10.2.1 Ultrasonic Cleaning in Conservation Practice

The device used in this research was greatly impacted by the parameters of the environment common to textile conservation wet cleaning. The parameters of other ultrasonic devices may differ greatly, which further emphasizes the need for evaluating and characterising the capabilities of each device within the intended wet cleaning environment before use on a historic object. This research showed that thermal energy output, and aluminium foil cavitation testing are accessible, inexpensive methods to evaluate ultrasonic probes. It is recommended that these methods be used in practice to understand the action of any bespoke ultrasonic equipment, and the interaction of the device and cleaning environment prior to use on any historic object.

This research indicated that frequency and amplitude were two significant factors for cleaning efficacy and damage potential, which had not previously been discussed in detail in the conservation literature. Understanding the impact of these factors on a textile wet cleaning environment will be crucial to using any ultrasonic

device. Factors of technique and cleaning environment, such as the depth and angle of the probe, optimal temperature ranges, and potential for chemical reactions must also be carefully evaluated and chosen when developing a wet cleaning treatment that uses ultrasound. Reporting these parameters in detail in publications and documentation will contribute greatly to the conservation literature.

The results of cleaning modern, standard-soiled cotton fabric with ultrasound showed that physical damage was not an intrinsic outcome of ultrasonic cleaning in a common textile conservation wet cleaning environment. Textiles that are robust enough to endure some level of ultrasonic cleaning will have qualities that give them strength and resiliency including structural stability of fibres, yarns, and weave, as well as high wet strength. From preliminary testing of standard soiled cotton in time-limited experiments, the cleaning results were markedly different from sponging, brushing, or other traditional mechanical actions, but this outcome was not further quantified in this experiment, as relevant variables were not part of the scope. However, this was further explored in the case study found in *Appendix A*.

10.2.2 Ultrasonic Device Development for Conservation

The literature research and the experimental phases of this dissertation suggested that ultrasonic equipment designed specifically for conservation may require tailored electromechanical parameters. Devices that increase user-control and decrease variability and overall strength of cavitation collapse would be of interest to conservation. Ultrasonic probes that operate optimally at very low wash bath depths and volumes, in cold temperatures, at very low amplitudes would be of interest to textile conservation applications. This provides necessary control when cleaning historic objects, and application of ultrasound must be compatible with these parameters.

Within the experimental conditions higher amplitudes reduced the effectiveness of cavitation and therefore cleaning efficacy during all experiments in simulated wet-cleaning environments. The forceful agitation and convection of liquid, along with the high temperatures of amplitudes of 60% or 80% at 40 kHz with a 5 mm wide, flat-tip probe created extreme conditions that were not conducive to cleaning textiles in a simulated conservation wet cleaning environment. Experimental devices that work at frequencies above 40 kHz, or use other parameters to create smaller cavitation bubbles, and therefore lower energy forces per bubble during collapse, may be of higher interest to textile conservation applications.

10.2.3 Health and Safety

Health and safety recommendations were seen to be widely standardised for ultrasonic cleaning devices with similar parameters. Measures to reduce human health and safety risks were simple and inexpensive to implement. However, during testing, higher amplitudes resulted in vapour rising from the cleaning solutions at high amplitudes. With the possibility of ultrasonic vapour rising from the wash bath, containing solvents, cleaning additives, and chemical components solubilised into the solution, there could be increased contact with substances hazardous to human health. This risk should be assessed and controlled in practice.

10.3 Further Research

10.3.1 Comparison of Ultrasound to Other Techniques

Phase 2 experiments suggested that the cleaning impact of different solutions may be more complex with ultrasonic cleaning than with traditional sponging techniques. This could be explored in practice for different types of soiling, staining, and other types of fibres. In practice, comparing additional variables and their impacts to cleaning efficacy and damage could also be pursued:

- comparative impact of different solution temperatures
- comparison of ultrasonic probe tip sizes or shapes
- other cleaning solutions
- other ways of quantifying potential chemical changes in cleaning solutions such as conductivity
- methods of controlling the direction and extent of the ultrasonic field and area of cavitation within the wash bath

Many avenues of research are open to further understand and control ultrasonic cleaning in textile conservation, some of which may require consultation or collaboration with conservation science. While ultrasonic cleaning literature in conservation has utilised scanning electron microscopy, further evaluation of physical or structural changes or damage has not been done. Tensile testing could further identify unwanted changes to fibre strength and stability. Similarly, colourimetry and chromatography could be used to investigate ultrasonic-related dye degradation, or sonochemical activity between the fibres and the cleaning solution.

The impact of solutes on the density, viscosity, vapour pressure, and other factors of the cleaning solution may be impeding or increasing cavitation activity in ultrasonic cleaning. These factors can impact cleaning efficacy and damage in ways

that go beyond the traditional conservation considerations of a solution's affinity for removing soiling from a fibre type.[100] This relationship deserves significant research, and it could contribute to sustainability initiatives to use the lowest possible volumes of the least-toxic wash bath additives.

Also contributing to sustainability as well as time-efficiency, the efficacy of ultrasound at removing surface soiling could be used not just during soiling removal, but during rinsing to ensure surfactant or other residues are not left behind on the object. This could lead to the use of smaller volumes of water during rinsing, and few rinse-cycles that could shorten the length of wet cleaning treatments. Future research is recommended to quantify ultrasonic device parameters, and cleaning environment variables to maximise cleaning efficacy and minimize potential damage.

10.3.2 Interdisciplinary Research

The lack of resources on the science of sound, and lack of common terminology or conceptual understanding of ultrasonic cleaning in conservation was seen as an impediment in the literature as well as during this dissertation. Through extensive discussion with those working in the fields of cavitation and ultrasound, this dissertation worked to produce an illustrated summary on the science of ultrasound that is accessible to conservators in practice. Yet only the surface of the complexity of ultrasonic cleaning was presented in this research. Future research should focus on promoting collaboration between experts in ultrasonic cleaning, engineering, the physicochemical science of ultrasound and cavitation, and conservation. This will further identify what ultrasonic device parameters and environment factors will allow for control, safe functionality, high cleaning efficacy, and low damage risk within textile conservation practice.

[100] Tímár-Balázsy and Eastop, *Chemical Principles*, 194-213.

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Appendix A: Case Study

12.1 Introduction

As seen in the *Literature Review*, case studies form an integral part of the body of knowledge built in conservation. However, few case studies of ultrasonic cleaning have been published in textile conservation to date. However, case study research was outside the scope of the initial dissertation research. Integration of a case study as an appendix allowed for a test case of applying the statistical data from Phases 1 and 2 to a historical object. The textile selected for testing was a historical soiled linen tapestry backing similar to the textile used in Cooke's 1989 testing at the V&A.[101] As all wet cleaning can potentially involve some amount of fibre loss or change in character or appearance, this study utilised controls not found in Cooke's study.

The decision to wet clean is made when the benefits of the treatment, such as reduction of damaging soiling, plasticizing of fibres, and increased aesthetic appreciation are of a greater impact than the potential damage or loss of fibres or evidential soiling.[102] The selection of the soiled historical textile for testing ultrasonic cleaning was guided by these factors. Linen retains much of its strength when wet and swells less in aqueous treatments compared to cotton.[103] If a textile is in good structural condition, the risk of fibre loss or damage would be considered low in a traditional sponge-based wet cleaning treatment. A wet-cleaning treatment to remove acidic, abrasive, and hygroscopic soiling, while plasticizing the cellulosic fibres would generally be considered beneficial to the object's long-term preservation and increase the aesthetic appearance.

12.2 Aim

The aim of this experiment was to compare and evaluate cleaning and damage to historical and new linen textiles using traditional wet cleaning techniques, and ultrasound.

12.3 Methodology

12.3.1 Test Fabric

Historical linen was obtained from the Karen Finch Reference Collection at the CTC (object ID: CTC. 445). Provenance records indicated it was removed from the back of a tapestry with no other data. The linen was an open plain weave of approximately 11 x 11 yarns per centimetre, with tightly twisted yarns of varying

[101] Cooke, "Ultrasonic Cleaning Historic Linen," 1989, 41-48.
[102] Tímár-Balázsy and Eastop, *Chemical Principles*, 1998, 194.
[103] Ibid., 9-15.

thickness (0.2 – 0.8 mm). Loose fibres, with one end still spun within the yarns, were visible across the surfaces of the textile from both narrow and thicker yarns. The linen was slightly yellowed from cellulose degradation[104] and heavily soiled with an even coating of fine particulate soiling on both sides. It was slightly stiff, with some brittleness, but otherwise in good structural condition. Microscopic analysis confirmed the fibre as linen, present as individual fibres of varying shapes, larger fibre bundles, and some inclusions of woody, unprocessed plant material.

New linen was selected as a control from the CTC supply of modern tapestry conservation support fabrics. Similar to the historical linen, the new linen was a plain weave construction of tightly twisted yarns. Loose fibres were visible across the surface. However, on average, the yarns were thinner and of a more even diameter (0.3 – 0.6 mm) than the historical linen. The fabric was a tighter, more compact weave with 24 x 24 yarns per centimetre compared to the historical linen, with more evenly twisted yarns. The new linen was clean, not yellowed, supple, in excellent structural condition.

Five samples of 80 x 100 mm were cut from the historical and new linen, selected from a contiguous area with consistent levels of soiling and structural condition. After analysis, the edges of each sample were secured with a blanket stitch to avoid fraying of unfinished edges during wet cleaning.

12.3.2 Cleaning Solutions

Two cleaning solutions were selected for the wet cleaning treatment of each sample. A solution of 0.5 g/L trisodium citrate and 0.05 g/L SCMC was selected as the first cleaning solution. The soiling of the historical linen was likely to be highly acidic, with a large amount of fine particulate matter. Selecting a solution of a buffer and soil suspender was chosen to mitigate the pH of the wash bath and prevent soil redeposition.[105] In addition, this solution showed a high cleaning efficacy and no damage in ultrasonic cleaning cellulosic textile with particulate soiling in Phase 2.

A solution of 0.3% w/v Dehypon® LS54 was chosen as a second solution. A non-ionic detergent at a high critical micelle concentration would be suitable for removing polar and non-polar soiling components from a heavily soiled textile. The cleaning efficacy seen in Phase 2 for this surfactant was low, and highly variable,

[104] Tímár-Balázsy and Eastop, *Chemical Principles*, 1998, 25-30.
[105] Ibid., 194-213.

but previous studies had shown some cleaning efficacy and no damage with using this surfactant to remove particulate soiling using only sponging.[106]

12.3 3 Cleaning Process

The amount of time each sample spent in each wash bath solution was standardized to six minutes. Paired samples of historical and new linen received one of the following treatments:

- Sample 1: no treatment
- Sample 2: no mechanical action
- Sample 3: sponging
- Sample 4: ultrasound
- Sample 5: both ultrasound and sponging

Each sample was treated with a controlled rate of treatment and length of time (*Table 12*). All processes were performed at ambient conditions (22-26 °C, 40-50% RH).

12.3.1.1 Sponging Methods

A Ramer® synthetic sponge slightly larger than the linen samples was used, and the time indicated was cumulative. Ten compressions of the sponge on one side of the textile generally took less than one minute and was then repeated for the other side of the textiles. This was indicated as "20x sponges" in descriptions of treatment.

12.3.1.2 Ultrasound Methods

Ultrasound was applied at 40% amplitude, with the probe held by hand at a slight angle, just under the surface of the solution. With the probe approximately 5-7 mm above the textile, slow, steady passes were done along the grain of the fabric for two minutes. The textile was then turned over and the process repeated for another two minutes. This was cumulatively four minutes of ultrasound applied during each cleaning step. For samples with both sponging and ultrasound, the ultrasound was applied first as the surfactant foam created by sponging obscured the textile during the ultrasound treatment.

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[106] Sato, "An Experimental Evaluation," (2014), 40-46.

12.3.1.3 Rinsing Methods

Rinsing was done with mechanical action until the rinse solution contained no visible soiling or foaming from surfactant residues (up to 5 rinse cycles). A final rinse was then done without mechanical agitation. Samples were left to air dry on Melinex® for 72 hours before after-treatment analysis was performed. No blotting or pinning out was done.

Step	**Textile Sample Number**			
	2	**3**	**4**	**5**
Wet out	500 mLs Deionised water for 2 minutes			
1st Wash bath	500 mLs 0.5% g/L Trisodium Citrate and 0.05 g/L SCMC			
	No Agitation over 6 minutes	2 min soak followed by 20x sponges over 6 minutes	2 min soak followed by 20x sponges and Ultrasound 4 minutes over 6 minutes	2 min soak followed by Ultrasound 4 minutes over 6 minutes
2nd Wash bath	500 mLs 0.3% Dehypon LS54			
	No Agitation over 6 minutes	2 min soak followed by 20x sponges over 6 minutes	2 min soak followed by 20x sponges and Ultrasound 4 minutes over 6 minutes	2 min soak followed by Ultrasound 4 minutes over 6 minutes
3rd Wash bath	500 mLs 0.5g/L w/v Trisodium Citrate and 0.05g/L SCMC			
	No Agitation over 6 minutes	2 min soak followed by 20x sponges over 6 minutes	2 min soak followed by 20x sponges and Ultrasound 4 minutes over 6 minutes	2 min soak followed by Ultrasound 4 minutes over 6 minutes
Up to 5 Rinse Cycles*	500 mLs Deionised water			
	No Agitation over 6 minutes	3 min soak followed by 20x sponges over 6 minutes	2 min soak followed by 20x sponges and Ultrasound 4 minutes over 6 minutes	2 min soak followed by Ultrasound 4 minutes over 6 minutes
Final Rinse	500 mLs Deionised water			
	No Agitation over 6 minutes	No Agitation over 6 minutes	No Agitation over 6 minutes	No Agitation over 6 minutes
*For each treatment, rinse cycles were only pursued up until a shake-test of the water revealed that no surfactant foam was visible in the wash bath.				

Table 12 Outline of the treatment process for samples 2 through 5 for samples of new linen and historical linen CTC.445. Sample 1 of each group received no cleaning treatment.

12.4 Analytical Methods and Equipment

The following analysis were done on both the historical and new linen samples before and after treatment. No statistical replicates were done, although 1 sample each of the historical and new linen were not treated for reference.

- photography
- mass measurement[107]
- colourimetry[108]
- optical microscopy[109]
- stereomicroscopy[110]
- SEM-EDX[111]

12.5 Results and Discussion

12.5.1 Overall Evaluation of Treatment

All samples of historical and new linen cleaned with sponging and/or ultrasound appeared cleaner and brighter after treatment, with increased flexibility. The samples cleaned with no mechanical agitation (OL2, NL2) had a slightly duller appearance. All samples 1-4 for both historical and new linen had a small amount of fibre loss in the wash baths from wetting and handling.

Samples with no mechanical agitation had the least amount of fibre loss seen in the wash baths, and treated samples of new linen had overall less fibre loss than samples of historical linen. The use of blanket stitching on the edges of each sample was sufficient to avoid weave disruption or fibre loss along the edges for all samples, regardless of the type or amount of mechanical agitation (*Figure 51*). There was no noticeable visual difference between the amount of fibre loss from treatments with or without ultrasound. However, the effect of ultrasound compared to sponging on the cleaning solution alone was seen as distinctly different (*Figure* 52).

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[107] Sartorius BP analytical balance, accurate to 0.0001 gram
[108] Konica-Minolta CM-2600d spectrophotometer, D65 daylight illuminant, 3 mm aperture, 4 readings per measurement using CIELAB SCI colour specifications.
[109] Zeiss Axiolab optical microscope
[110] Zeiss Stemi-2000C stereomicroscope
[111] SEM: EVO50XVP (Carl Zeiss SMT Ltd), EDX: X-Max^N (Oxford Instruments Ltd). Additional details on SEM-EDX instrumentation and processing are found in *section 12.5.6*.

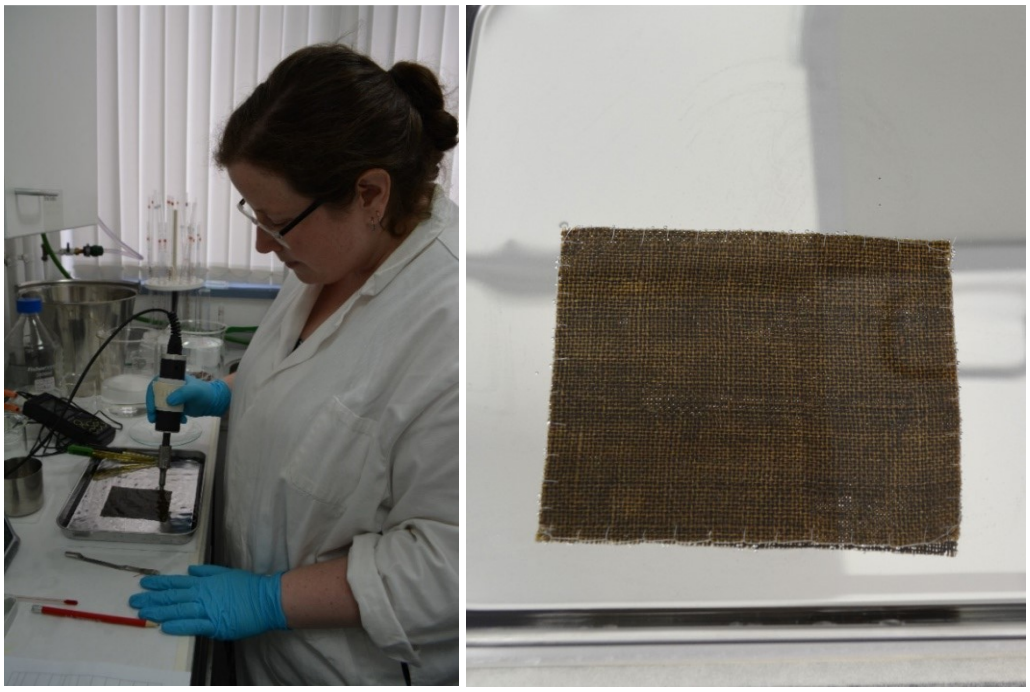

Figure 51 Showing the wet cleaning environment and blanket stitching to secure loose areas of weave.

Temperature and pH of the bath had no notable changes that could be attributed to ultrasound. Short cleaning cycles and replacement of the solutions were done to control drops in pH from soiling release, and to remove particulate soiling as it was released. Solution temperature (20-24 °C) remained in ambient ranges, and pH measurements revealed no fluctuations or anomalies between the different treatment regimens.

A significant difference of the wet cleaning process with ultrasound treated samples was the need for fewer rinsing cycles. All samples treated with ultrasound during rinsing (OL4, OL5, NL4, NL5) only required 1 rinse cycle with ultrasound (4 minutes of ultrasound over 6 minutes) before a shake test revealed no surfactant foaming of the rinse bath. Samples treated with only sponging (OL3, NL3) required 5 rinse cycles with sponging (10 minutes of sponging over 30 minutes) before a shake test revealed no surfactant foaming of the rinse bath.

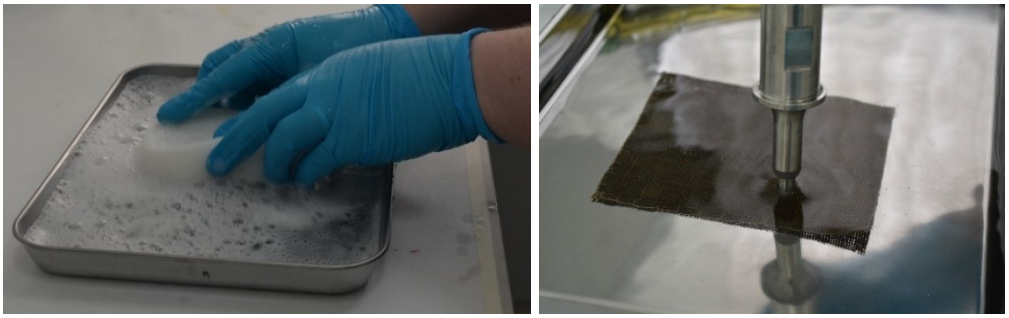

Figure 52 Wash baths with 0.3% w/v Dehypon® LS54 in deionised water with sponging (left) and ultrasound (right).

12.5.2 Treatment Evaluation and Imaging

12.5.2.1 New Linen

Macroscopic visual analysis showed little change to the colour, brightness, or handle of the new linen from the cleaning treatments (*Figures 53, 54*). Samples of the that had mechanical action (NL3, NL4, NL5) felt hydrated and supple. Fibres were bright, shiny, and appeared clean, but not significantly different from before treatment. Little to no difference between each of these samples was seen or felt. Some wrinkling was reduced, and slight shrinkage occurred as there was no dimensional control during drying. Sample NL2 had no mechanical treatment but was wet out in each cleaning solution. This sample felt slightly stiffer and darker than before treatment. Sample NL1 had no treatment as a reference/control sample.

12.5.2.2 Historical Linen

Macroscopic visual analysis of the historical linen samples showed distinctly different responses to the cleaning treatments (*Figures 55, 56*). Samples that had mechanical action (OL3, OL4, OL5) felt less brittle, and more flexible. Fine black particulate soiling was significantly reduced for all samples. There were visible differences seen between each of these samples. Some wrinkling was reduced, and no shrinkage was noted. Sample OL2 had no mechanical treatment but was wet out in each cleaning solution. This sample no longer had loose particulate soiling but was stiff and appeared slightly darker. Sample OL1 had no treatment as a reference/control sample.

Figure 53 New linen before treatment.

Figure 54 New linen after treatment.

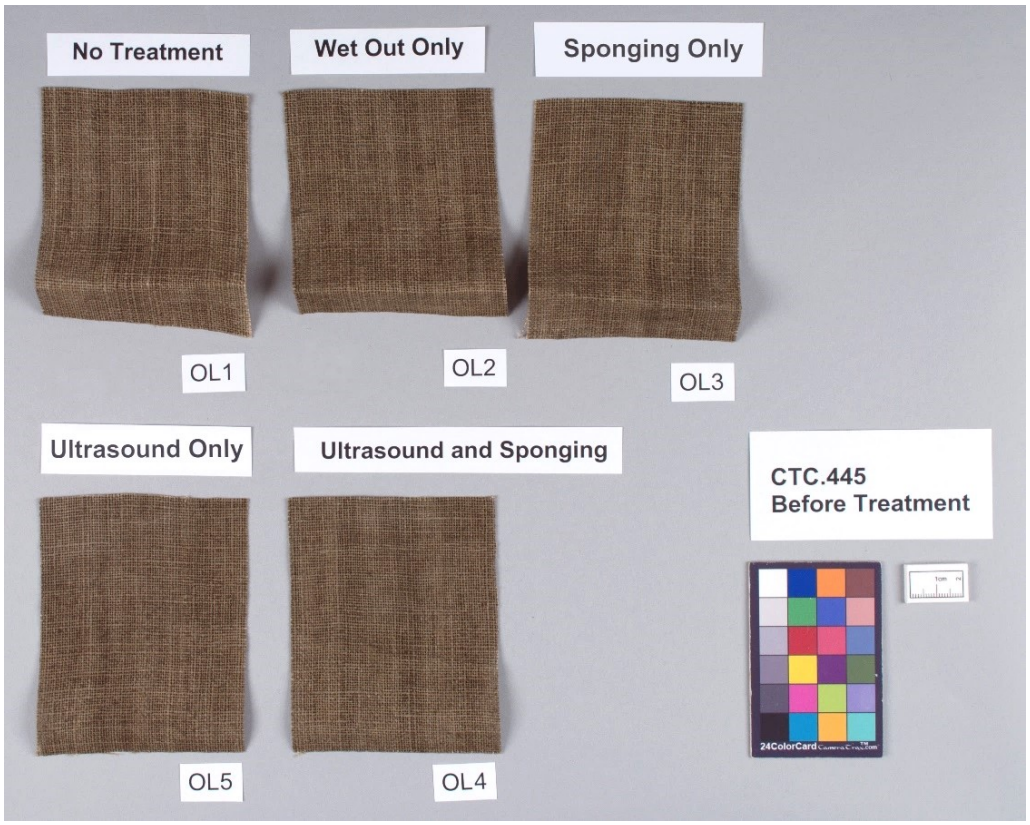

Figure 55 Historical linen CTC.445 before treatment.

Figure 56 Historical Linen CTC.445 after treatment.

12.5.3 Mass Change

12.5.3.1 New Linen

There was little change in the mass measurements of the new linen samples before and after cleaning. The slight rise in mass for all samples was attributed to retained water due to the moisture regain of linen (*Table 13, Figure* 57).

Sample ID	Treatment	Mass Lost in Grams	Mass Gained in Grams
NL 1	No Treatment		0.0148
NL 2	No Agitation		0.0141
NL 3	Sponging Only		0.0103
NL 4	Sponging and Ultrasound		0.0061
NL 5	Ultrasound Only		0.0114

Table 13 Mass change of new linen samples after cleaning.

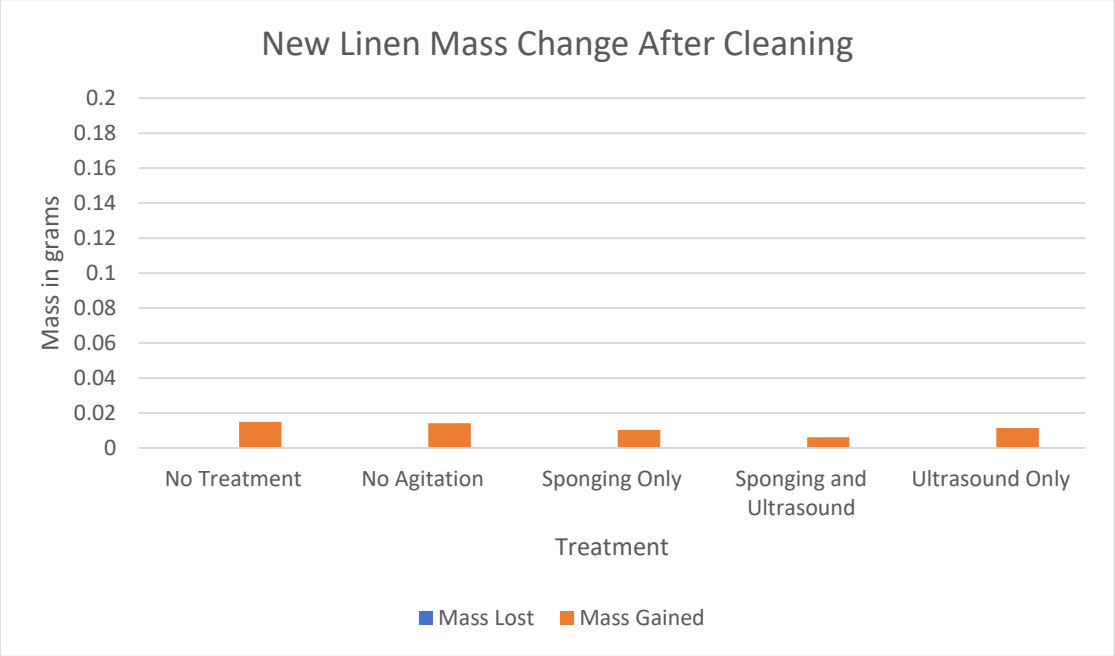

Figure 57 Chart of new linen mass change after treatment.

12.5.3.2 Historical Linen

The largest loss of mass per sample was correlated to the most mechanical action applied during treatment, with sample OL4, treated with sponging plus ultrasound losing the most mass. The slight gain in mass from sample OL1 with no treatment is likely due to the moisture regain of linen of 12%[112] (*Table 14, Figure* 58)

Sample ID	Treatment	Mass Lost in Grams	Mass Gained in Grams
OL1	No Treatment		0.0122
OL2	No Agitation	0.1488	
OL3	Sponging Only	0.1667	
OL4	Sponging and Ultrasound	0.1849	
OL5	Ultrasound Only	0.1610	

Table 14 Mass change of historical linen CTC.445 samples after cleaning.

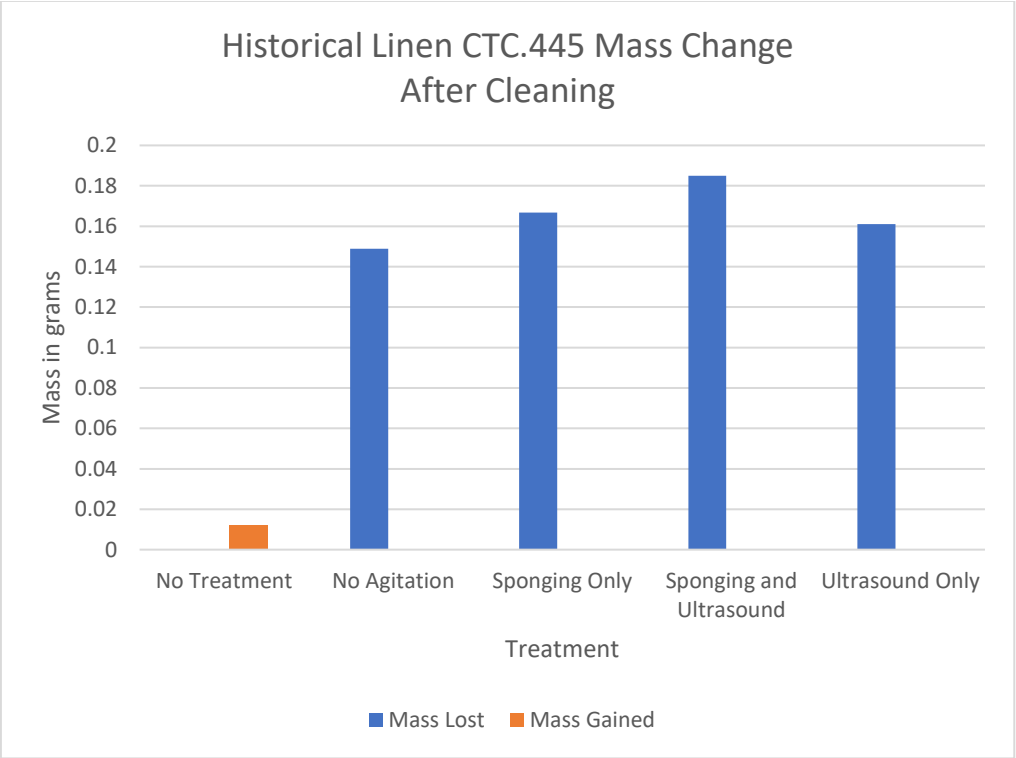

Figure 58 Chart of historical linen CTC.445 mass change after treatment.

[112] Tímár-Balázsy and Eastop, *Chemical Principles*, 15, 34.

12.5.4 Colourimetry

Colourimetry using CIELAB L* values was used to quantify the brightness before and after cleaning treatment. In this colourimetry scale, 0 is neutral black and 100 is neutral white.

12.5.4.2 New Linen

Samples of clean, new linen showed very little change in brightness before and after treatment. Sponging and ultrasound combined treatment resulted in the largest increase in brightness at only 1.7 points brighter than before treatment (*Table 15, Figure 59*).

Sample ID	Treatment	L Value Before Cleaning	Increase in Brightness	Decrease in Brightness
NL 1	No Treatment	64.68		0.9
NL 2	No Agitation	64.28	0.1	
NL 3	Sponging Only	65.11	0.93	
NL 4	Sponging and Ultrasound	64.52	1.7	
NL 5	Ultrasound Only	63.46	1.05	

Table 15 New linen CIELAB L brightness values before and after treatment.*

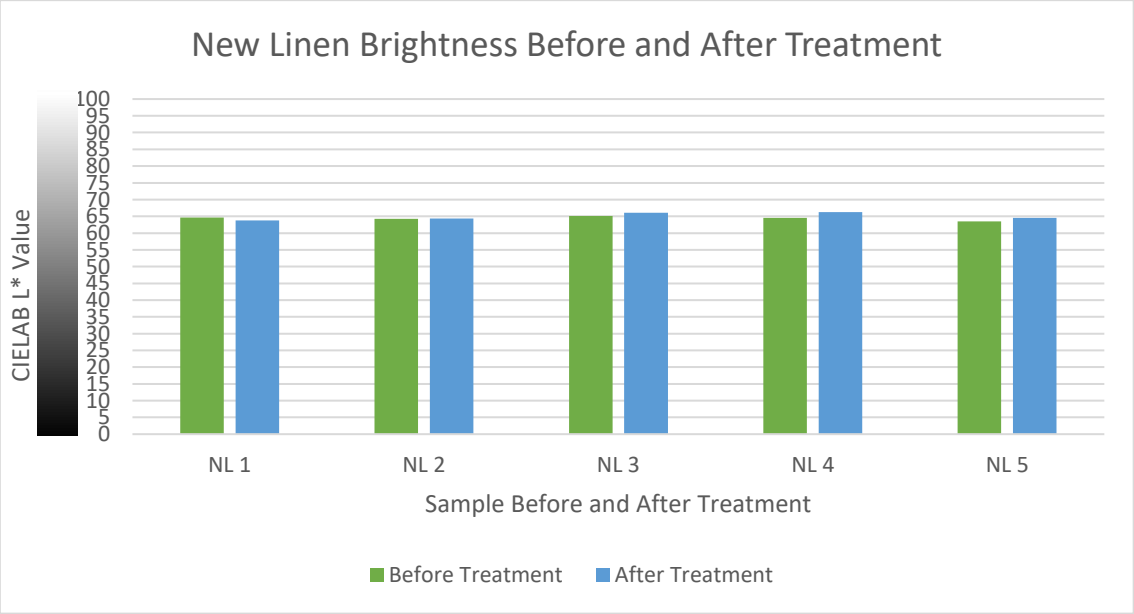

Figure 59 Chart of new linen CIELAB L brightness values before and after treatment.*

12.5.4.2 Historical Linen

Samples of historical linen with no treatment, or no agitation measured slightly darker after treatment. Sponging resulted in a textile 16.31 points brighter, while ultrasonic cleaning resulted in a textile 10.68 points brighter than before treatment. The treatment of sponging and ultrasound resulted in an increase in brightness by 17.55 points compared to before treatment measurements (*Table 16, Figure* 60). This is reflective of the overall visual impact of cleaning, and of the comparative levels of mass change.

Sample ID	Treatment	L* Value Before Cleaning	Increase in Brightness	Decrease in Brightness
OL 1	No Treatment	40.45		0.66
OL 2	No Agitation	40.19		0.97
OL 3	Sponging Only	39.21	16.31	
OL 4	Sponging and Ultrasound	39.2	17.35	
OL 5	Ultrasound Only	37.61	10.68	

Table 16 Historical linen CTC.445 CIELAB L brightness values before and after treatment.*

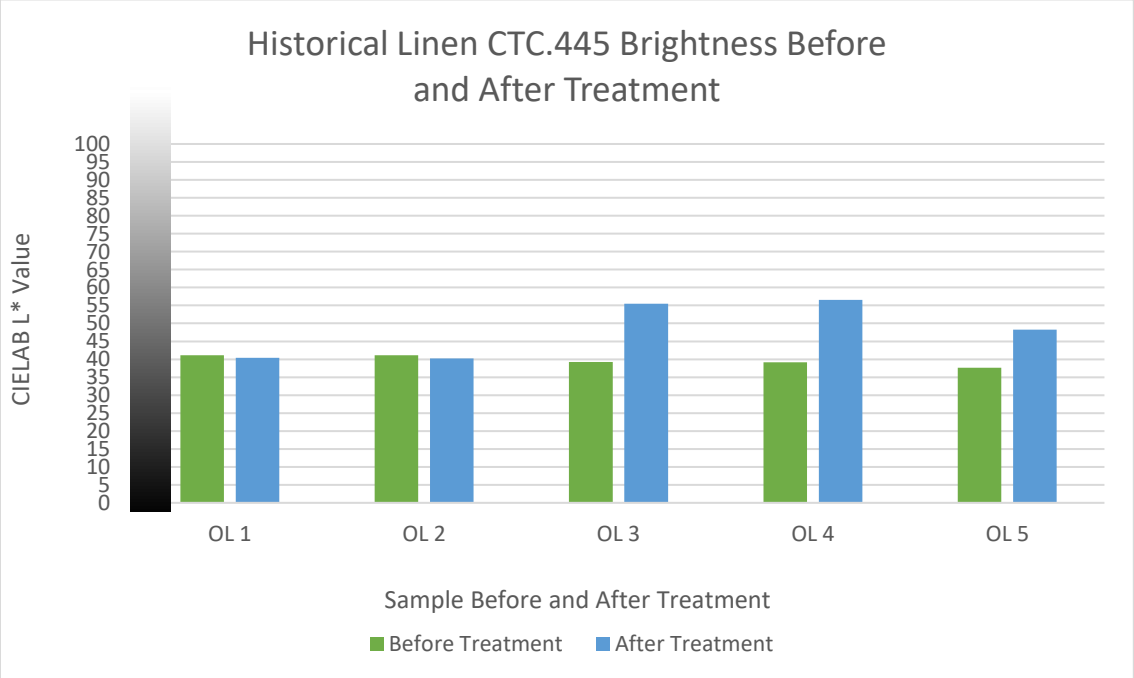

Figure 60 Chart of historical linen CTC.445 CIELAB L brightness values before and after treatment.*

12.5.5 Microscopy

12.5.5.1 New Linen

Samples of new linen looked largely the same among the different treatments both before and after cleaning in terms of structural integrity of fibres and weave. New linen samples were more compactly woven than the historical linen. Before treatment, the new linen also had larger number of long, loose fibres across the surface compared to the historical linen. In a few small, discrete areas, the samples treated with ultrasound (NL4, NL5) had areas where these fibres tangled and stood up from the surface more so than samples treated with only sponging (NL3), or with no mechanical action (NL2). Low-magnification microscopy images of new linen samples reflected the visible cleaning and structural integrity (*Figures 61, 62, 63*).

12.5.5.2 Historical Linen

In terms of visible cleanliness, microscopy results at .65x, 2x, and 5x correlated to the quantification of soiling loss seen by colourimetry and change in mass for the historical linen. Ultrasound and sponging (OL4) had the greatest shine and brightness with the least visible soiling, followed closely by sponging only (OL3). The ultrasound only treatment (OL5), was less bright than OL3 or OL4. The three samples OL3, OL4, and OL5 were significantly brighter, shinier, and had less soiling compared to before treatment (*Figures 64, 65, 66*). The sample with no mechanical agitation (OL2), looked dark, dull, and still soiled after treatment.

In terms of fibre damage, microscopy results at .65x, 2x, and 5x revealed some significant differences between the treatments for the historic linen. Historic linen samples treated with no mechanical action (OL2) just sponging (OL3), just ultrasound (OL5) were generally in good structural condition after treatment without obvious damage. Loose fibres on the surface, which were not well incorporated into the twist of the yarns, looked to be in much the same structural condition before and after treatment. However, the sample treated with sponging and ultrasound combined (OL4) had fibre breakage on both sides of the textile, seen at the intersections of the weave structure. The broken fibres seemed to be primarily the same loose surface fibres seen before treatment.

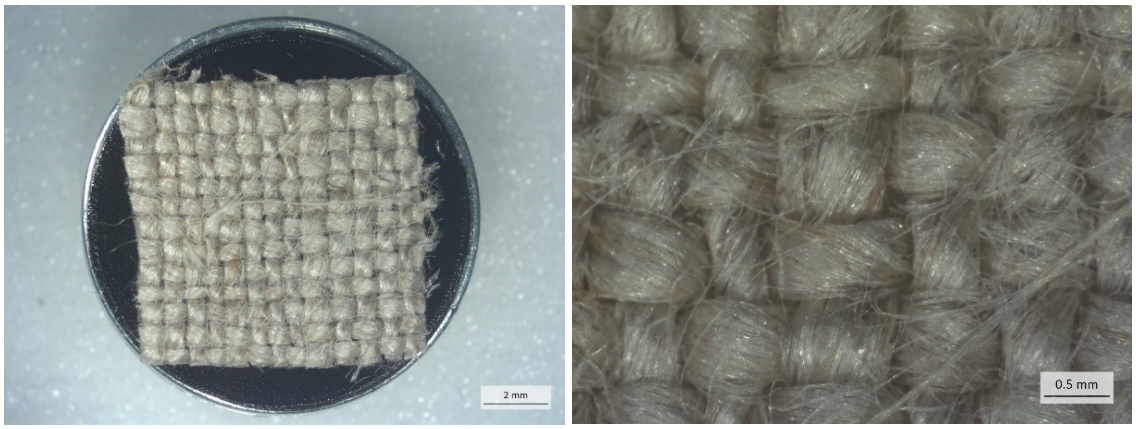

Figure 61 Sample NL3 treated with sponging only.

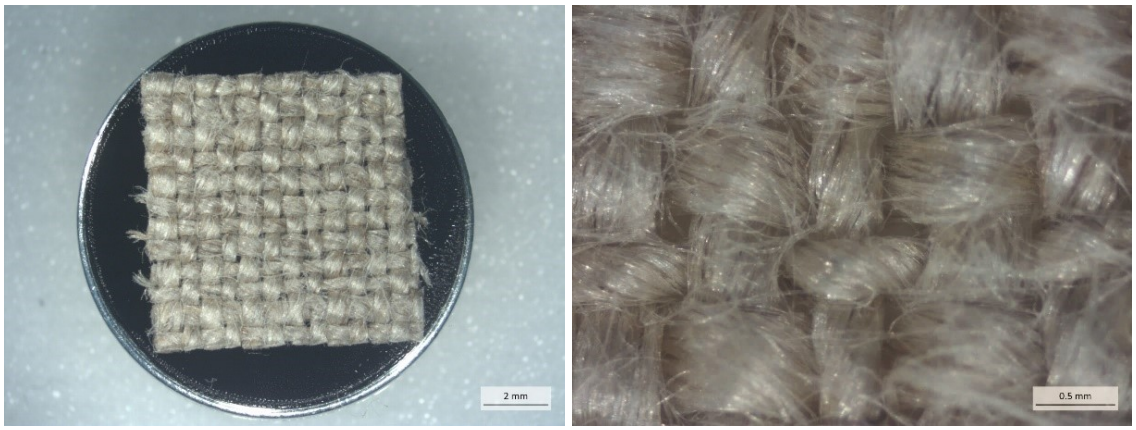

Figure 62 Sample NL4 treated with sponging + ultrasound, showing no visible damage or disarray of fibres or yarns.

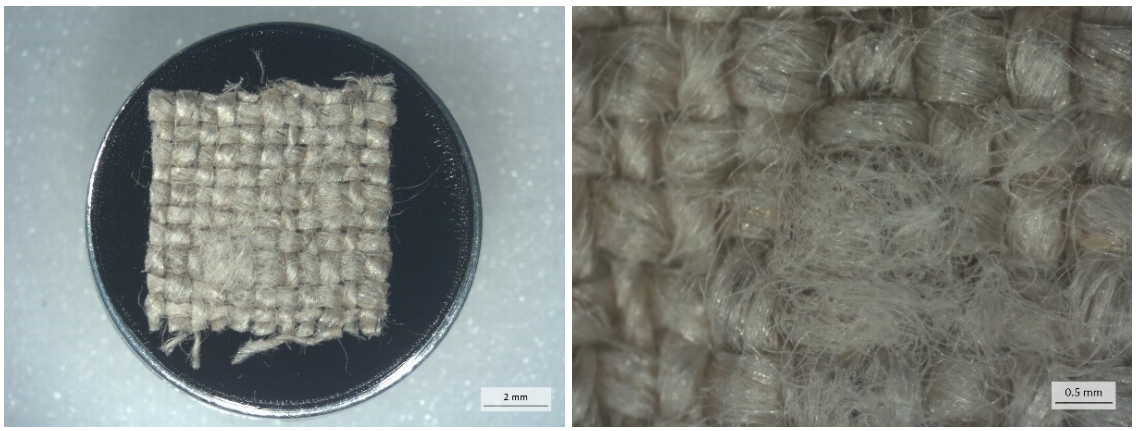

Figure 63 Sample NL5 treated with ultrasound only, showing a tangling of loose fibres on the surface.

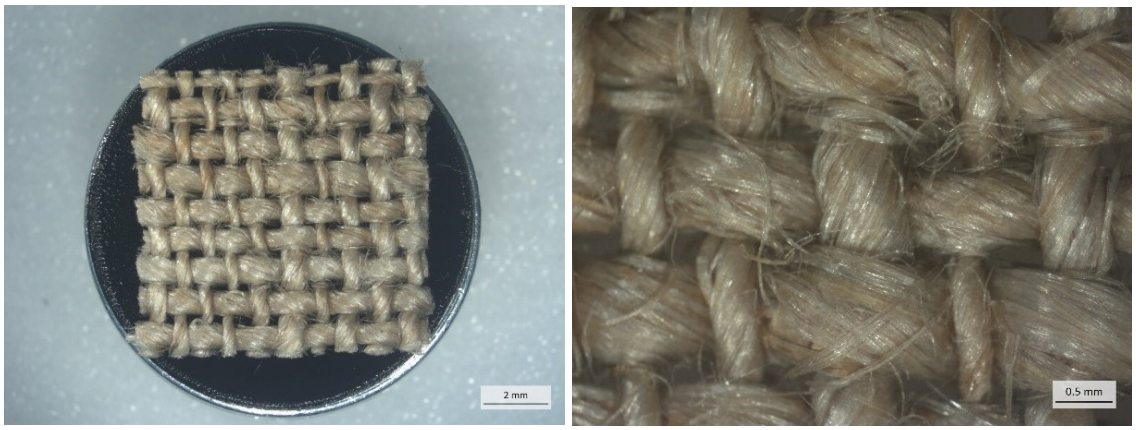

Figure 64 Sample OL3 treated with sponging only.

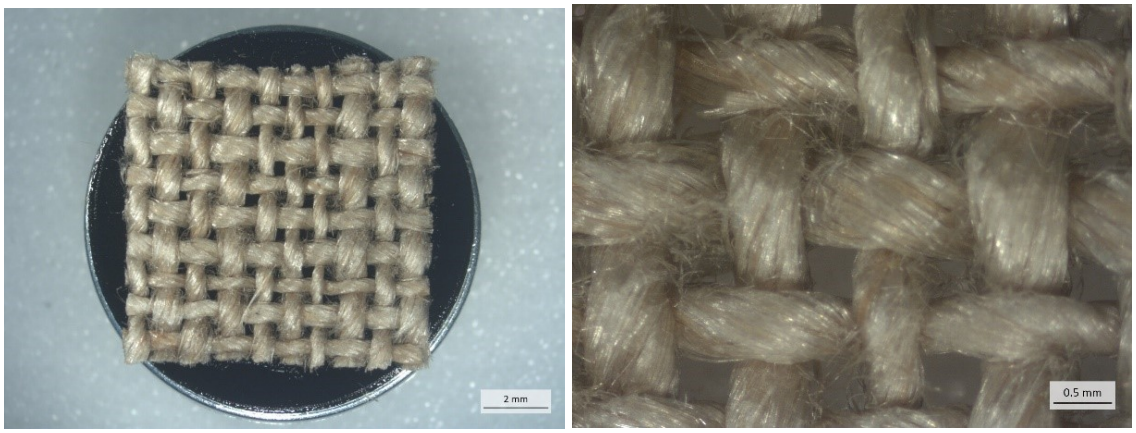

Figure 65 Sample OL4 treated with ultrasound and sponging, showing some fibre breakage at the intersection of the weave structure.

Figure 66 Sample OL5 treated with ultrasound only, appearing slightly darker than samples OL3 or OL4.

12.5.6 SEM-EDX

Scanning electron microscopy and x-ray spectroscopy (SEM-EDX) were performed on all five of the samples of new linen and historic linen. Samples 5 x 5 mm were cut from a representative area near the centre of each sample and mounted onto aluminium stubs with carbon sticky tabs. Sputter coating[113] was done to improve imaging prior to SEM[114] and EDX[115] analysis to improve imaging. Samples were evaluated for cleaning efficacy and physical damage.

12.5.6.1 Historic Linen

Sample OL1 was used to characterise the general condition and particulate soiling of the historic linen before treatment. With SEM imaging, the difference in structural stability, twist angle, and size of the yarns was readily visible. Some yarns appeared to be in good structural condition, with well-bound fibres, while others appeared broken with loose fibres and untwisted yarns. The even coating of fine particulate soiling was also visible as a crumbly coating on top of the yarns and between fibres (*Figure 67*).

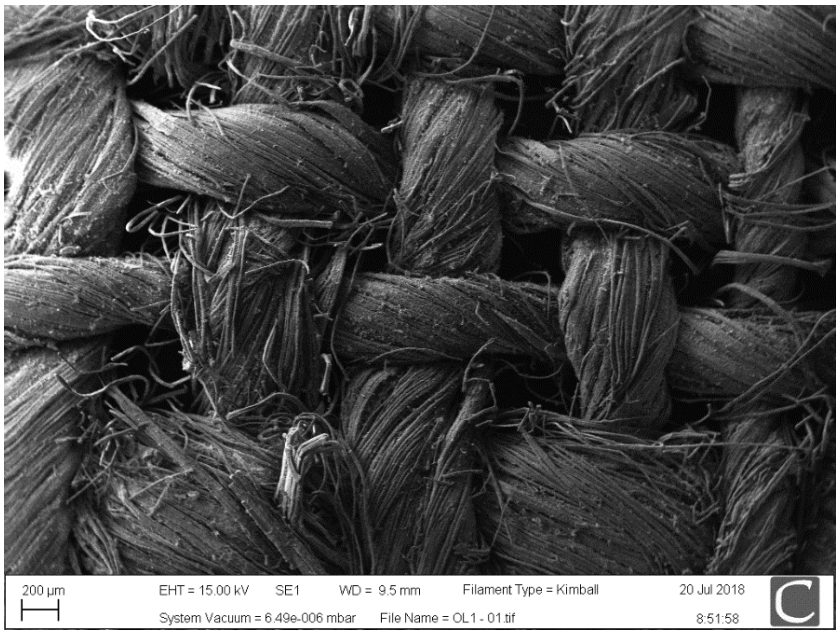

Figure 67 SEM image of historic linen sample OL1, which received no treatment.

[113] SC7640 Sputter Coater (Quorum Technologies Ltd.), target: 10% Pd / 90% Au, EHT: 2.2 KV, 10 – 20 mA current with a coating time of 60 seconds.
[114] EVO50XVP (Carl Zeiss SMT Ltd), with detectors: Secondary Electron (Everhart Thornley), Backscattered Electron (Quadrant Si diode).
[115] X-Max^N (Oxford Instruments Ltd.), SDD (50 mm^2), filament: LaB6 (Kimball), EHT: 15 KV, X-ray Collection Time: 1000 live seconds.

A closer magnification showed the texture of the aged linen fibres, revealing the compact nature of the yarns and linen fibres. Loose and broken fibres are visible, and some cracks and striations in the fibre bundles are visible underneath the film of soiling (*Figure 68*).

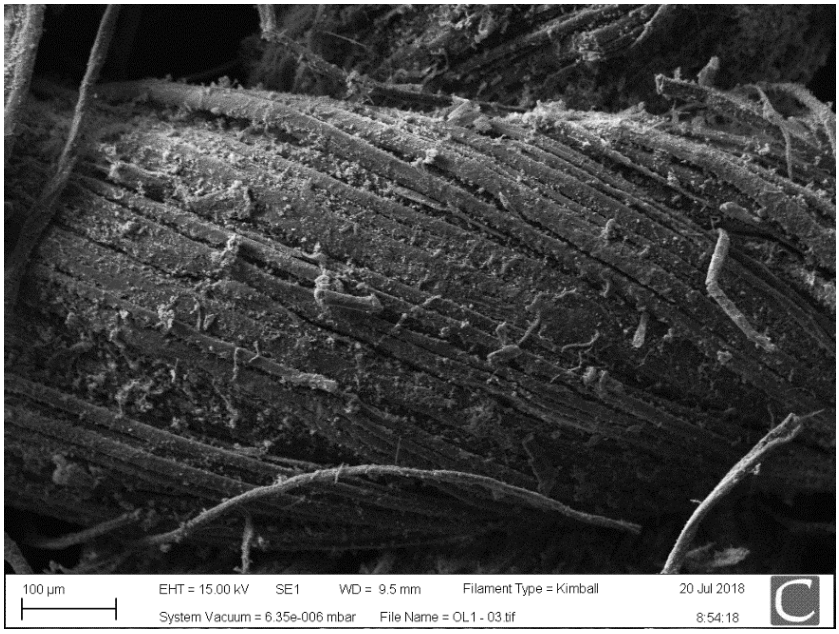

Figure 68 Higher magnification SEM image of historic linen sample OL1, which received no treatment.

Sample OL2 received only a wetting out in each of the cleaning solutions with no mechanical action. Any damage to fibres or yarns beyond what was seen from the pre-treatment condition, as well as soiling removal was due to the immersion into the solutions, the heat of wetting, and the swelling of the fibres. A total of six rinse cycles with deionised water without mechanical action were done to remove solution. Low magnification revealed much smoother surface of yarns and fibres, indicating a significant amount of particulate soiling was no longer on the surface. No residues from the cleaning solutions were detected (*Figure 69*).

Figure 69 SEM image of historic linen sample OL2 after treatment (no mechanical action).

Closer magnification showed the fibres were still coated with fine soiling (elemental composition confirmed by EDX, discussed below). However, the surface, and condition of the linen fibres were now more visible. Surface damage showing cracks and breakage were visible in loose fibres, and on some of the surfaces (*Figures 70, 71*).

Figure 70 Higher magnification SEM image of historic linen sample OL2 after treatment (no mechanical action).

Figure 71 Higher magnification SEM image of historic linen sample OL2 after treatment (no mechanical action).

Sample OL3 received treatment of sponging only with five rinse cycles followed by a sixth static rinse in deionised water (30 minutes rinsing). This sample had little soiling on the surface fibres, but soiling was found in crevices between fibres. Slightly more broken fibres were seen compared to control samples (OL1, OL2), which was partly due to the full visibility after soil removal. There were still a significant number of loose fibres across the surface after treatment (*Figures 72, 73, 74, 75*).

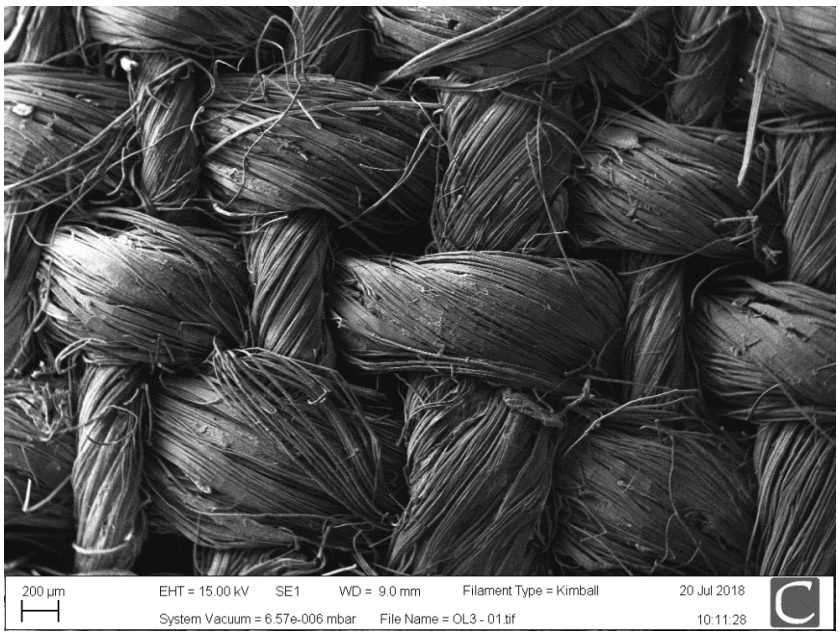

Figure 72 SEM image of historic linen sample OL3 after treatment of sponging only.

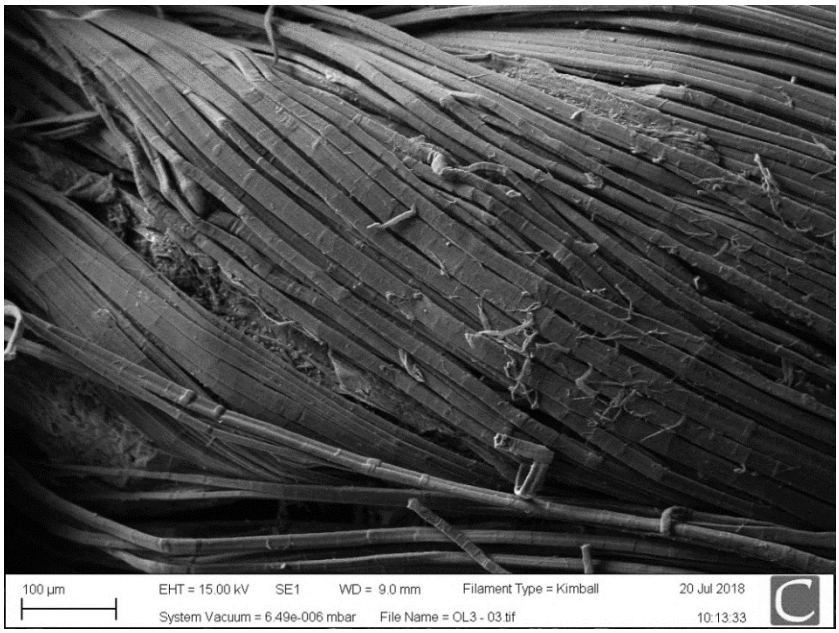

Figure 73 Higher magnification SEM image of historic linen sample OL3 showing different surface texture compared to control samples OL1 and OL2. Some stripping of the surface of the fibres and breakage is visible.

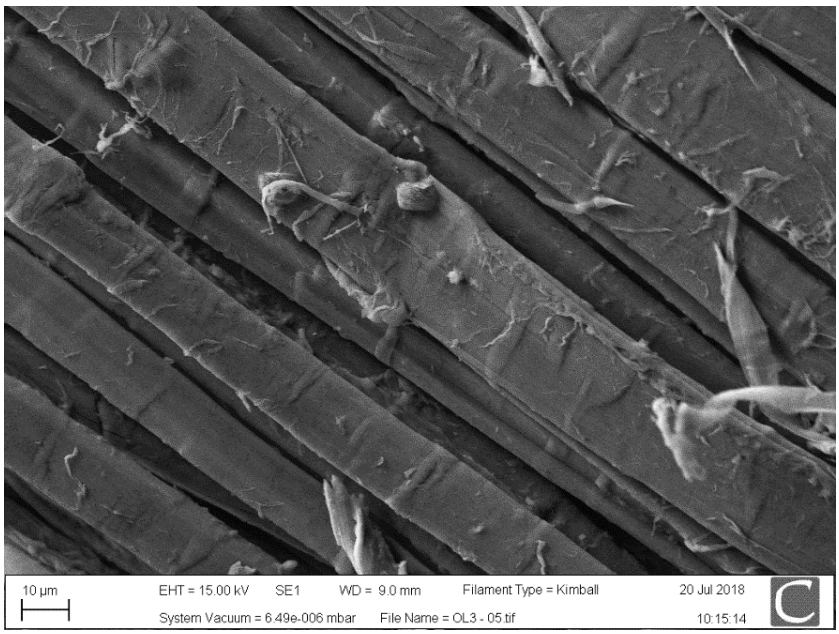

Figure 74 Higher magnification SEM image of historic linen sample OL3, focusing on the stripping of the surface of the fibres. At this magnification, striations along the fibres (perpendicular and along the length of the fibre) can be seen, as they are largely clean of soiling. However, soiling is still visible between the fibres. Note the textured fibres and thin strips of fibrous matter at the bottom right.

Figure 75 Higher magnification historic linen sample OL3 focusing on damage to the fibres which was somewhat more common on this sample than others.

Sample OL4 received treatment of sponging and ultrasound. Only 1 rinse cycles with ultrasound was completed, followed by a single static rinse in deionised water (total of 11 minutes rinsing). This sample had no soiling on the surface fibres, and almost no soiling was found in crevices between fibres. Broken fibre bundles were clearly visible at the interstices of the weave, significantly more so than other samples (*Figures 76, 77*).

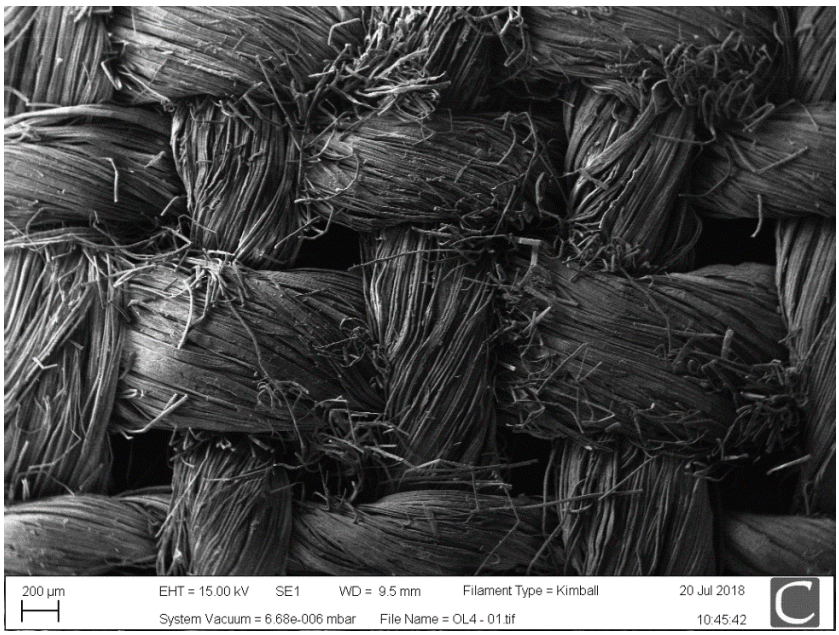

Figure 76 SEM image of historic linen sample OL4 showing the limited number of loose fibres across the surface, and the increased volume of broken fibres at the crossover of the warp and weave.

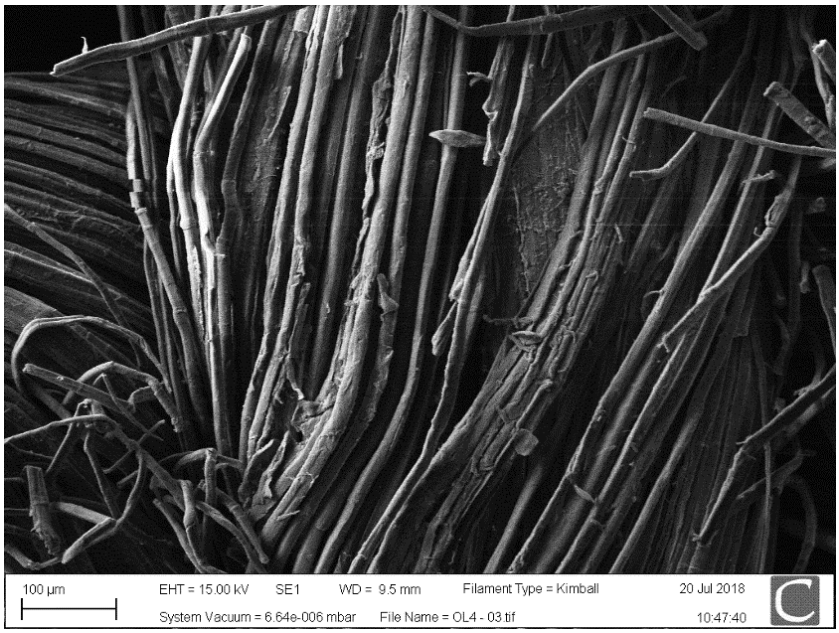

Figure 77 Higher magnification SEM image of historic linen sample OL4, showing separation of surface fibres, and broken fibres. This type of damage was more common in this sample than other treatments.

Sample OL5 received treatment of ultrasound only. Only 1 rinse cycles with ultrasound was completed, followed by a single static rinse in deionised water (total of 11 minutes rinsing). This sample had no soiling on the surface fibres, and very little soiling in crevices between fibres (*Figures 78, 79, 80*) There was no breakage of fibres as seen in sample OL4 which received ultrasound and sponging treatment.

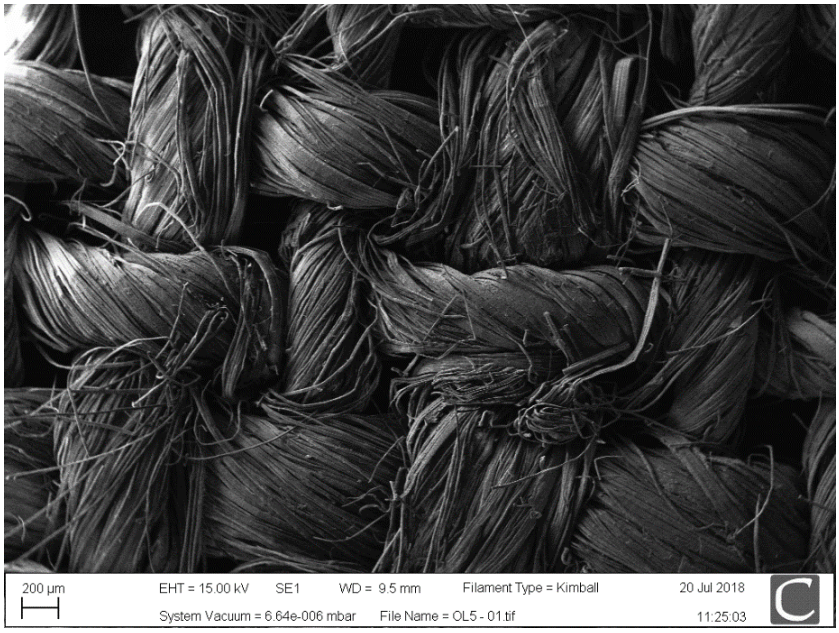

Figure 78 Historic linen sample OL5 treated with ultrasound only. Note that fibres were generally structurally intact, and that the extensive fibre breakage seen in OL4 was not visible in this sample.

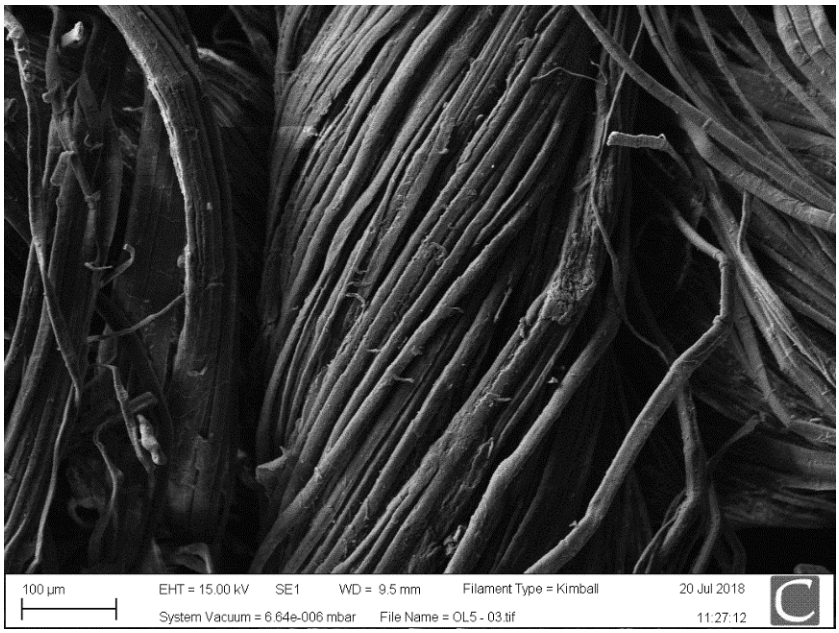

Figure 79 Higher magnification of historic linen sample OL5 treated with ultrasound only. Note that fibres are compact in this sample, and not spread apart or widely broken as seen in OL4.

Figure 80 Higher magnification of historic linen sample OL5. No surface soiling was visible, despite the less-bright appearance of the sample compared to the sponging-only sample OL3 or the ultrasound plus sponging sample OL4.

12.5.6.2 New Linen

As a control set, the samples of new linen were largely uniform in the SEM analysis (*Figures 81, 82*). Little difference was seen in the surface texture, weave structure, yarn twist, or fibre damage at the surface of the linen or as breakage of fibres. The supple new fibres of modern linen,

constructed with a tight twist and compact weave survived all combinations of sponging and ultrasonic cleaning well, which matches the lack of differentiation between mass change and colourimetry readings on each sample.

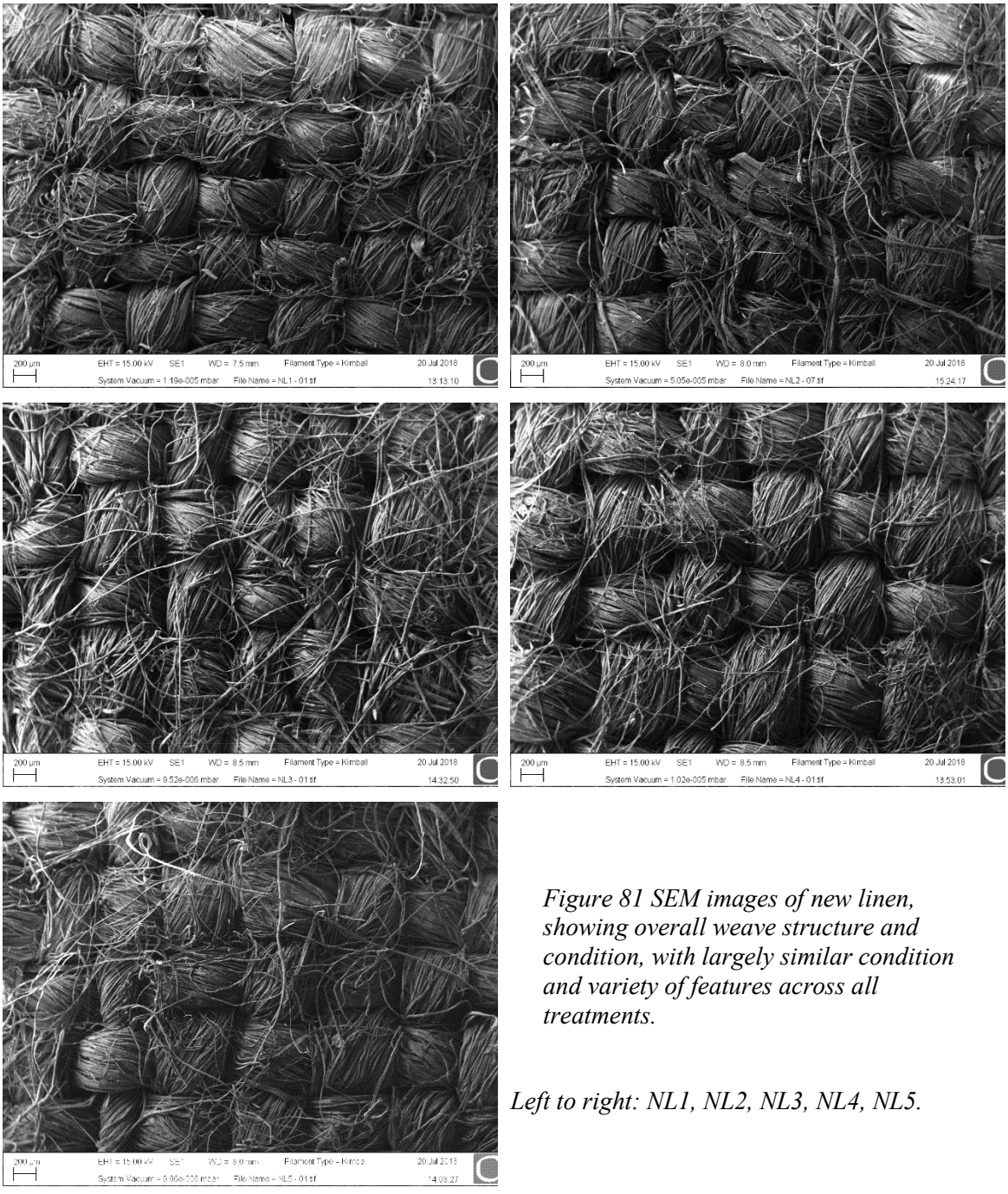

Figure 81 SEM images of new linen, showing overall weave structure and condition, with largely similar condition and variety of features across all treatments.

Left to right: NL1, NL2, NL3, NL4, NL5.

Figure 82 Higher magnification SEM images of new linen at, showing largely similar condition and variety of features on the individual fibres across all treatments.

Left to right: NL1, NL2, NL3, NL4, NL5.

12.5.7 EDX

EDX was performed to examine the fibres for elemental differences and to further characterise the soiling composition on the historic linen. No elemental changes to the historic or new linen fibres were detected through EDX. Levels of carbon and oxygen were widely present and similar across all samples of historic and new linen.

Analysis of the soiling on the historic linen showed that the composition of the soiling was largely similar across all samples after normalizing the readings on the oxygen values[116] as seen below in (*Figures 83, 84*). The yellow band shows the soiling analysis of sample OL1, which received no cleaning. All other samples show similar peak patterns, but at a lower amount due to the cleaning processes. Peaks of gold (Au) and palladium (Pd) are present due to the sputter coating for the analysis. Soiling composition was detected to be largely be calcium (Ca), sulphur (S), and silicon (Si), with smaller quantities of sodium (Na), aluminium (Al), potassium (K), magnesium (Mg), copper (Cu), iron (Fe) and chlorine (Cl). This is consistent with fine, particulate dust[117] and sooty airborne pollution comprised of polar and non-polar soiling that can be highly abrasive due to crystalline structures, as well as hygroscopic, and acidic.[118]

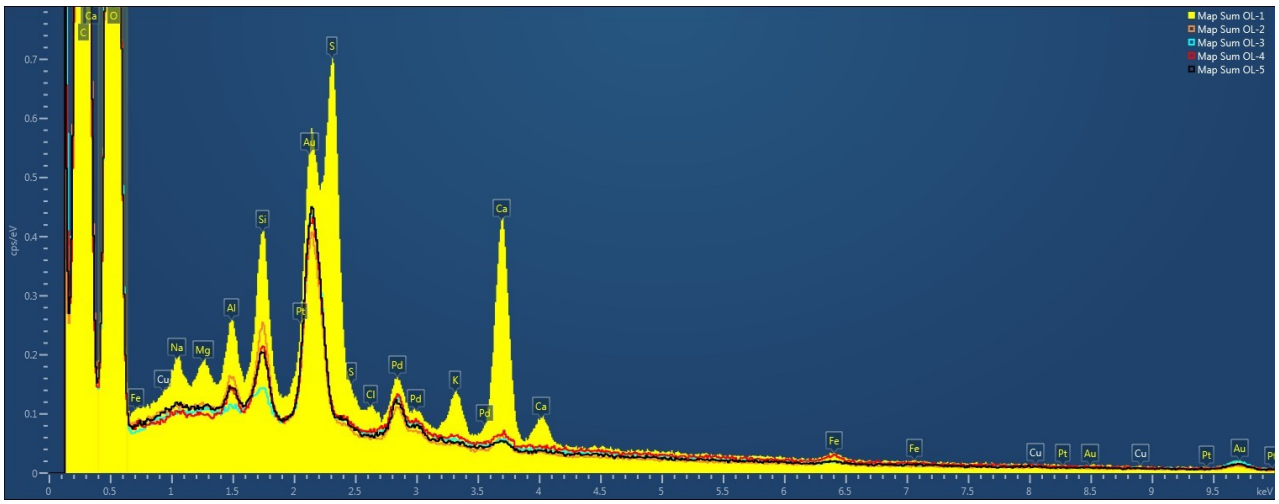

Figure 83 EDX analysis of soiling composition from each of the five historical linen samples.

[116] Data correction done with AztecEnergy correcting for atomic number (Z), absorbance (A) and fluorescence (F).
[117] K. Robert Lange, Detergents and Cleaners, (Munich: Carl Hanser Verlag, 1994), 31
[118] Peter Brimblecombe and Carlota M. Grossi, *The Identification of Dust in Historic Houses* (National Trust, 2010), accessed https://www.nationaltrust.org.uk/documents/the-identification-of-dust-in-historic-houses.pdf.

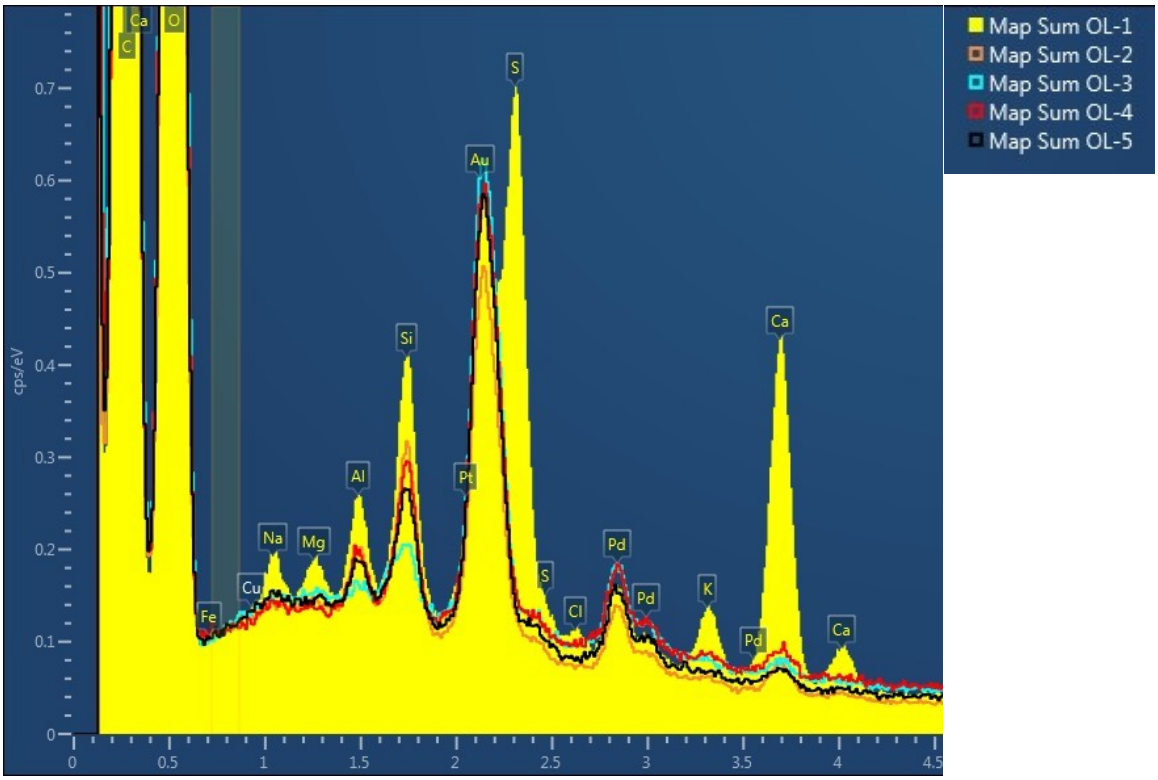

Figure 84 Detail of EDX analysis of soiling composition found on each of the five historical linen samples.

EDX sampling was restricted to the outer surface areas of fibres. The reduction of surface soiling was largely consistent across all treatments (OL2, OL3, OL4, OL5). Differential soiling removal was seen in the sampled areas for aluminium and silicon. Sponging only treatment OL3 appears to have been more effective at reducing aluminium and silicon-based soiling on the surface of the fibres. Whereas the wetting-only treatment with no mechanical action of sample OL2 had the least reduction of aluminium and silicon-based soiling for all the treated samples.

12.6 Conclusion

This case study resulted in several findings about ultrasonic cleaning historic textiles, and the nature of cleaning and damage from ultrasound. These findings are reflective of the specific parameters of the cleaning process including the textile characteristics, soiling type, solutions chosen, and using a 40 kHz ultrasonic probe of 5 mm diameter applied at 40% amplitude approximately 5 mm distance with the experimental device.

12.6.1 Cleaning

The action of sponging compared to ultrasound provided distinctly different results, indicating the nature of cleaning. Sponging alone removed significant levels of particulate soiling, although soiling remained between fibres and crevices when

analysed with SEM-EDX. Sponging was most effective at removing yellowing when analysed visually and with colourimetry. The action of sponging a textile provided both an abrasive action at the surface of the fibres, as well as a vertical compression which improved capillary exchange of the cleaning solution through the textile fibres to solubilize the chromophore compounds that are the source of yellowing from cellulose degradation.

Ultrasound alone was most effective at removing particulate soiling, even in between fibres and crevices. However, ultrasound alone resulted in very little removal of yellowing. This indicated that ultrasound alone was not as highly active on capillary forces compared to sponging. This may have been affected by the very tight twist seen in the historic linen, which can reduce or slow introduction of water to the inner core of the fibres.

Ultrasound alone was also significantly more efficient at removing surfactant residues compared to sponging. Through SEM-EDX, no residues were seen on the treatments with sponging, ultrasound, or in combination, but sponging-rinsed samples took 24 more minutes to remove residual surfactant. Ultrasound applied for four minutes during a single six-minute rinsing cycle was as effective at removing the surfactant and cleaning solutes as 100x sponges over 30 minutes in five rinse cycles. This may make ultrasound a choice for wet cleaning scenarios where overall length of time in the wash bath is a high concern.

12.6.2 Damage

The ultrasonic cleaning treatment alone did not result in more damage to a historic textile as compared directly to a sponging treatment, as determined by SEM analysis. This indicates that ultrasonic cleaning will not always cause damage in tandem with cleaning results. Overcleaning with ultrasound is a risk that is perhaps more quickly approached with ultrasound compared to traditional sponging, due to the small scale of action, which creates damage at similarly small scale that may be difficult to monitor in the wash bath.

For textiles where the fibres are supple, modern, or otherwise in good structural condition, loose fibres across the surface of the weave were highly susceptible to fraying, loosening, and tangling both ultrasonic and sponging treatments. For textiles where the fibres are brittle, aged, damaged, or otherwise in poor structural condition, loose fibres across the surface of the weave are highly susceptible to breakage and loss. In this case, sponging and ultrasound appeared to

have similar, low-levels of damage, while the combination of treatments was significantly worse.

Blanket stitching was suitable for controlling unravelling, fibre loss, and damage from wet cleaning during traditional sponging, ultrasound, and the combination of ultrasound and sponging. The plain-weave fabrics both showed no damage at the edges for any of the treatments, despite slightly different amount of twist, yarn diameter, and open-ness of the weave, indicating that controlling raw edges with standard methods may be sufficient in ultrasonic cleaning procedures.

12.7 Future Research

Given these findings, there are recommendations for future work with ultrasonic cleaning historic textiles. The tangling and damage seen from loose fibres in new and historic linen suggest that control is needed at the surface of textiles to restrict movement and protect loose elements at a very small scale. This proved true for both new and historic fibres in the experiment. As blanket-stitching the edge of new and historic linen avoided damage, a similar surface-level barrier should provide protection.

Research using Melinex® barriers during ultrasonic cleaning has been promising in terms of cleaning efficacy and lack of visible damage.[119] Melinex® may be providing a dampening effect on the energy of ultrasonic cleaning but may also be providing vertical limitations on movement of loose fibres or yarns. Other barriers of woven or non-woven, synthetic or natural fibres should also be explored to balance the force of cleaning against the potential damage.

Further comparative studies of sponging and ultrasound, as well as the effect of different cleaning solutions or other parameters and textile fibres should be explored. This is necessary to inform the nature of ultrasonic cleaning and contextualize acceptable levels of fibre change or damage as part of a beneficial treatment. Research specifically comparing ingrained staining of an oily nature, or otherwise strongly bonded soiling is recommended to characterise the limits of cleaning and boundaries of structural damage.

[119] Hackett, “Ultrasonic Cleaning,” 2018; Crowther, “Application of Ultrasonic Cleaning,” 2018.

Appendix B Glossary of Terms

This glossary list contains only a small number of terms and phrases as they are used in the physics and chemistry of sound, ultrasound, or cavitation, and also as they relate to topics covered in this dissertation. Definitions and descriptions are listed alphabetically and were compiled from widely available, accessibly written works cited at the end of this section.

Absorption Acoustic absorption is the amount of sound energy that is absorbed by a substance (solid, liquid or gas). A portion of the mechanical energy of the sound wave is absorbed as heat, while some of the sound wave is transmitted through the substance. The density, viscosity, and flexibility of the solid, liquid or gas each effect how much sound wave energy is absorbed as heat or transmitted through the substance.

Amplitude Amplitude is the distance between the midline of a sound wave and the highest or lowest point of the wave. Amplitude is related to the energy transported by a sound wave. The larger the amplitude, the more energy the sound wave has. Amplitude can change dependent upon the substance it flows through. In the ultrasonic device used in this dissertation, the amplitude cannot be altered exactly by the user, although the size of the tip of the ultrasonic probe does affect the amplitude. A smaller tip on the probe creates a higher amplitude wave, and a larger tip creates a lower amplitude wave.

Audible sound Sound in the range of human hearing, around 20Hz to 20KHz.

Cavitation The formation of bubbles of vapour within a liquid when subjected to rapid pressure changes, such as the application of an ultrasonic field

Cavitation Collapse The collapse of cavitation bubbles, which can cause rapid spikes in temperature, changes in pressure, shockwaves, and microjets in fluids.

Compression When vibrated by a sound wave, molecules in a substance (solid, liquid, or gas) are compressed during the peaks of the sound wave. This creates high pressure in the substance.

Cycle A cycle is one complete sound wave, from peak to trough. Older nomenclature for wavelength may be written as kilocycles, for which there is a 1:1 ratio e.g. 20 kilocycles = 20 kHz.

Decibel Unit of measurement that utilises a logarithmic scale to measure a material property in a ratio to another property (similar to the value and measurement of acidity with pH). This unit is abbreviated dB and often related to the

	loudness of a sound, where audible sound around 40 dB is similar to a quiet conversation, 60 dB is average background noise or conversation. Audible sound above 85 dB can cause hearing damage, and above 120 dB may be painful.
Frequency	The number of cycles of a sound wave per second, measured in Hertz (Hz).
Longitudinal Wave	A wave in which the direction of travel is the same as the direction of vibration.
Period	Unit describing the number of cycles of a sound wave per second. Note that this is the opposite way that frequency describes and measures a sound wave.
Piezoelectricity	A property of a material allowing it to take in electrical energy and convert it to mechanical energy. Quartz is a material with piezoelectric qualities.
Power	In physics, this is the rate of work, or energy over time, one of the units of power in terms of sound is the watt.
Rarefaction	When transmitting ultrasound, molecules in a solid liquid or gas are pushed apart, which creates low pressure in the substance.
Resonance	The amplification of a sound wave by a substance, when the frequency of the wave matches the structural frequency of the substance (solid, liquid, or gas).
Shockwave	A wave that moves through a substance faster than the substance can transmit it, causing disruption and rapid change in pressure, density, and temperature.
Sonochemistry	The use of ultrasound to induce chemical reactions such as oxidation, reduction, and hydrolysis.
Speed	The speed of sound is dependent upon the substance it is transmitted through. Sound can be transmitted through a solid liquid or gas, each of which will affect the speed of the sound wave. The speed of sound is measured in meters per second. Speed of sound in water at ambient temperatures is around 1,500 m/s.
Supersonic	Sound traveling at a speed faster than the substance transporting it can transmit it. In ambient temperature water, this would be sound waves travelling above the standard speed of sound in the medium, around 1,500 m/s.
Transducer	A transducer is a device that transforms one type of energy into another. Piezoeletric transducers are common in ultrasonic cleaning.

Transverse Wave A wave in which the direction of travel is perpendicular to the direction of vibration.

Ultrasound Sound above the range of human hearing, starting at 20 KHz.

Watt: A unit of power, measuring energy over time. Often used to quantify the electrical energy input and/or output by ultrasonic equipment however the Watt input to the electromechanical components does not equal the Watt output to the cleaning system.

Wave Vibrational sound energy (for audible sound and ultrasound) travel as a wave. Waves have peaks and troughs, which can be measured by their peaks and toughs of distance or pressure.

Wavelength The distance travelled by sound in one cycle, measured in meters.

Glossary Bibliography:

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Appendix C Health and Safety: Risk Assessment and CoSHH Forms

GUIDANCE ON COMPLETION OF RISK ASSESSMENT

Management Unit:	CTC-TAH	**Location: (Site/ Building/ Room)**	Robertson 3rd Floor
Assessment Date:	02/05/2018	**Review Date:**	
Assessors Name:	Megan Creamer	**Job Title:**	2nd Year Student

Task / Activity: Experimental testing of ultrasonic device in common textile conservation wet cleaning solutions including water, detergents, solvents, chelating agents, and oxidising or reducing agents. Ultrasonic device operates at a fixed frequency of 40kHz.

What are the hazards? (See list of sample hazards)	**What are the risks?**	**Who might be harmed?** (e.g. Staff, students, visitors)	**What control measures are required to eliminate or reduce the risks?**	**Risk Evaluation**			**Risk Rating**
				Consequence (1 – 3)	**Likelihood (1 – 3)**	**Overall risk (C x L)**	**Low, Medium or High**
Ultrasonic tool	Hearing damage, burns, fire, physical damage to human tissue, unknown catalysis of chemical reactions **See references:** Ahmadi 2012, Institute of Sound and Vibration Research 2001, Kardous 2015, Smagowska 2013, US Centre for Disease Control 2014.	Student, staff, visitors	**Hearing damage:** Ear protection must be worn, testing can be done in the chem lab with the door closed, and with a warning sign on the door. Decibel level and hours exposure will be recorded. **Burns, Fire:** The metal tip of the ultrasound probe produces heat. Heat-resistant rest will be used to allow the tool to cool. Do not touch or interact with metal tip. Use contact temperature monitor to check temperature of tip. Temperature of all solutions will be charted and tracked continuously. Indirect application of ultrasound may be used to reduce risk. **Physical Damage:** Do not touch metal tip when in use. Do not touch or interact with the coupling media (e.g. the liquid solution into which the probe is lowered). Do not point probe at the human body. Air exposure to ambient ultrasound under max kHz, decibel, and hour levels will not cause human tissue damage. **Catalysis/Acceleration of Chemical Reactions:** PPE (gloves, mask, eye protection lab coat, closed toe shoes), pH and temperature will be charted and tracked continuously and kept within safe levels for the specified liquid or the experiment will be stopped. Indirect application of ultrasound may be used to reduce risk. Select experiments may take place in the fume cupboard.	3	1	3	Med
Electrical equipment	Electric shock, equipment malfunction,	student	Check PAT testing dates and perform visual check to ensure equipment is in good working order.	2	1	2	Low
Wires and blocking access	Slips, trips, and falls	Staff, students, visitors	Avoid trailing wires and do not block exits. Notify others of equipment use.	2	1	2	Low

What are the hazards? (See list of sample hazards)	What are the risks?	Who might be harmed? (e.g. Staff, students, visitors)	What control measures are required to eliminate or reduce the risks?	Risk Evaluation			Risk Rating
				Consequence (1 – 3)	Likelihood (1 – 3)	Overall risk (C x L)	Low, Medium or High
Sharp tools, needles, scissors, scalpels	Cuts	Staff, students, visitors	Cap and put away sharp tools when not in use. Do not cut towards yourself, use sharpened, appropriate tools. First aid located in Chem lab and Wet lab. Dispose of sharps properly in Chem lab.	2	1	2	Low
Heated tools or liquids	Burns, fire	Staff, students, visitors	Notify others of equipment use. Create a clear and safe work space with melt and fire-resistant barriers where needed. Use signs to notify when equipment has been turned off and left to cool. Do not touch hot surfaces. First aid located in Chem lab and Wet lab.	2	1	2	Low
Spills	Slips, trips, and falls	Staff, students, visitors	Mop up spills immediately, notify others. Spill kits located in Chem lab.	2	1	2	Low
Solvents and additives	Hazards to human health. See COSHH form for details of specific solvents, TBD. General risks include inhalation of noxious or toxic vapours, splash risks	Staff, students, visitors	Use appropriate PPE per CoSHH, working under extraction. Notify others of use. Transport in trays or on trolleys, label and store all solvents appropriately. Use smallest amounts appropriate, keep absorbent materials handy during use in case of spills. Follow CoSHH form for specific health risks in case of contact or spill. First aid located in Chem lab and Wet lab. Dispose appropriately in Chem lab and log use if needed. Spill kits located in Chem lab.	3	1	3	Med
Glassware	Cuts from breakage or use of broken glassware	Staff, students, visitors	Do not use glassware for ultrasonic testing. Use stainless steel or polyethylene containers/liners. Do not use broken glassware. Notify others in workroom if there is a break, verbally and with signage until it is removed. Remove wearing and using appropriate PPE. Dispose of properly in broken glass disposal in Chem lab. First aid located in Chem lab and Wet lab.	2	1	2	Low
Repetitive Movement/Posture	Hazards to health, strain	Student	Minimise repetitive movements, use hand or arm supports if possible. Take frequent breaks to prevent strain. Use good posture and adjust height of work and seating surfaces.	2	1	2	Low
Completed by: Megan Creamer, 2nd year CTC student,				**Date:** **25/05/2018**			
Approved by:				**Date:** **25/05/2018**			
Signed by:				**25/05/2018**			

CoSHH Assessment

Assessment Title: Ultrasonic cleaning experiments

Assessment Reference Number:

School / Service / Location: History of Art, CTC-TAH, 3rd floor Robertson Building 56 Dumbarton Road

Safety Coordinator: Karen Thompson

Details of Hazardous Substances (Please attach safety datasheets where available)

Name of Substance (Include all substances used or produced)	Quantity kg / g / ml	Physical Form	GHS Hazard Classification (Tick all that apply)								
1. Trisodium citrate dihydride	<10 gram	White powder					X				
2. Sodium carboxymethylcellulose	<10 grams	White powder									
3. Dehypon® LS54	<10mls	Clear liquid									X

Special Hazards (*Separate risk assessment may be required)

Carcinogenic Substance	Details:	Skin Sensitiser	Details: Trisodium citrate is an eye irritant	Respiratory Sensitiser	Details:
Biological Material*	Details:	Radioactive Substances*	Details:	Explosive Atmosphere*	Details:

Further Details / Other Special Hazards:
Dehypon® LS54 toxic to fish

Exposure to Hazardous Substances						Workplace Exposure Limits	
Substance	Possible Exposure Route (Please tick)					8h TWA	15min STEL
	Inhalation	Ingestion	Skin	Injection	Other (State)		

Description of Activity (Continue on a separate sheet if required)

Testing of 100mL dilute aqueous solutions using ultrasonic tool (40kHz) in metal beakers at ambient temperature and pressure. Further testing of wet cleaning standard soiled cotton (olive oil and carbon black) with aqueous solutions using ultrasonic tool (40KHz) in 500mL of water in a metal tray. Temperature, pH will be measured during cleaning process.
The ten test solutions will be: deionised water alone, soft water alone, degassed deionised water, Dehypon® LS54 at 0.3% w/v in deionised water, 0.5g/L trisodium citrate in deionised water, 0.05g/L sodium carboxymethyl cellulose in deionised water, and a solution will also be made of 0.5g/l trisodium citrate and 0.05 g/L sodium carboxymethyl cellulose together in deionised water.

Persons at risk: Students, Staff, Visitors (general public in vicinity).

Summary of Control Measures

Assessment of risks and any existing control measures	**Risk of chemical exposure through inhalation, contact, ingestion.**		
Risk Rating (Before Control)	**High**	**Medium**	**Low X**
Procedural Controls (e.g. lone working, hygiene)	**No food or liquid consumption in work rooms, secure entry to work room, all solutions will be labelled and disposed of properly. Hands will be washed before changing activities. No working alone**		

Engineering Controls (e.g. fume cupboard)	None needed.			
PPE Requirements (Please give details) **Face fit testing required	 Dust Mask**	P3 dust mask for powders.	 Gloves	
	 Respirator**		 Footwear	Closed toed shoes.
	 Eye Protection	Safety glasses when pouring full concentrate or mixing dry powder	 Protective Clothing	Labcoat
	 Face Shield		 Other (Specify)	Long trousers, no exposed skin. Hearing protection do not touch ultrasonic components, bath, or cleaning vessel
Instruction and Training	Yes, wet cleaning modules.			
Supervision Required?	Yes – course tutors present during workroom hours.			
Other safety precautions: (Including specialist first aid requirements)	Notify others when powders are in use. First Aid: Inhalation (dizziness, stupor): move to fresh air Skin contact: wash thoroughly with soap and water, rinse thoroughly Eye contact – flush eyes with water Ingestions – do not induce vomiting, if conscious rinse mouth with water, contact medical assistance.			
New Risk Rating	High	Medium	Low X	
Supporting Information Checklist (Include details for each where relevant)				
Waste Disposal	Very dilute concentrations can be disposed of down drain. Run plenty of water.			
Emergency Procedures (including spill / leak control)	Mop up spills using paper towels or for large spills, the spill kit in chem lab. Notify others of spills – move to fresh air/open windows or increase extraction as needed.			
Atmospheric Monitoring	NA			
Health Surveillance	NA			
Supporting Risk Assessments (Please attach where relevant)	Biological NA	DSEAR NA	Radiation NA	
Assessment Details				
Assessed By:	Megan Creamer		30/05/2018	
Approved By:			Date: 31/05/2018	
Date of next review:				
Description of Activity (Continuation sheet)				
Continuation sheet number:				

CoSHH Assessment Acknowledgement

By signing this document I acknowledge that I have read and understood the attached CoSHH assessment and have familiarised myself with the safety control measures and protective equipment necessary to carry out the task safely. I hereby agree to follow the safe system of work required and implement the required safety procedures fully.

Full Name	Signature	Date Completed
Megan Creamer		31/05/2018

Appendix D Phase 1 Data

Temperature

Temperature readings taken throughout Phase 1 testing, discussed in *Chapter 8.*

Group	Sample ID Number	Replicate Number	Digital Temperature Readings (°C) at Minute #						
			0	2	4	6	8	10	12
1	D.G. 1	1	24.0	25.0	25.6	26.3	26.7	27.1	
1	D.G. 2	2	24.3	25.5	26.3	26.8	27.1	27.3	
1	D.G. 3	3	24.4	25.5	26.2	26.7	27.2	27.3	
1	D.G. 4	4	24.0	25.1	25.7	26.3	26.7	27.0	
1	D.G. 5	5	24.3	25.2	25.9	26.5	27.0	27.5	
2	D.G. 6	1	25.3	28.1	32.4	34.7	36.6	38.0	
2	D.G. 7	2	25.2	28.5	31.3	33.9	36.2	38.0	
2	D.G. 8	3	24.9	28.6	31.5	34.0	36.1	38.1	
2	D.G. 9	4	25.4	28.6	31.6	33.9	36.2	38.1	
2	D.G. 10	5	25.2	28.2	31.0	33.6	35.9	37.7	
3	D.G. 11	1	25.7	31.0	36.7	38.2	40.7	41.6	
3	D.G. 12	2	24.7	29.5	33.0	35.7	37.6	39.2	
3	D.G. 13	3	26.0	31.2	35.6	39.1	41.4	42.0	
3	D.G. 14	4	26.2	30.1	34.5	37.0	38.7	40.0	
3	D.G. 15	5	26.3	30.8	34.6	37.4	39.2	40.3	
4	D.I. 1	1	24.6	25.7	26.6	27.3	28.1	28.8	
4	D.I. 2	2	24.3	25.3	26.0	26.8	27.4	28.0	
4	D.I. 3	3	24.3	25.5	26.2	26.8	27.6	28.2	
4	D.I. 4	4	24.2	25.2	26.2	27.0	27.6	28.2	
4	D.I. 5	5	24.1	25.3	26.5	27.2	27.7	28.3	
5	D.I. 6	1	23.9	27.8	31.0	33.8	36.2	38.3	
5	D.I. 7	2	24.8	28.5	31.4	33.9	36.2	38.3	
5	D.I. 8	3	24.9	28.4	31.4	33.9	36.1	38.0	
5	D.I. 9	4	24.9	28.2	31.1	33.6	35.8	37.8	
5	D.I. 10	5	24.7	28.0	30.7	33.1	35.3	37.3	
6	D.I. 11	1	24.6	28.5	31.6	34.0	35.8	37.5	
6	D.I. 12	2	24.5	29.2	33.9	36.3	37.2	38.8	
6	D.I. 13	3	25.1	29.9	34.3	37.8	40.8	43.2	
6	D.I. 14	4	24.9	29.7	33.8	38.9	41.1	43.5	
6	D.I. 15	5	24.8	29.7	33.8	37.0	39.2	41.3	
7	S.W. 1	1	24.4	24.8	25.3	25.8	26.1	26.8	
7	S.W. 2	2	24.1	24.9	25.1	25.7	26.0	26.4	
7	S.W. 3	3	24.0	24.6	25.1	25.5	25.8	26.1	
7	S.W. 4	4	23.9	24.7	25.3	25.8	26.1	26.4	
7	S.W. 5	5	23.7	24.2	24.4	24.8	25.2	25.6	
8	S.W. 6	1	24.1	27.4	30.4	32.9	35.1	37.0	
8	S.W. 7	2	23.8	26.8	29.7	32.1	34.3	36.1	

Group	Sample ID Number	Replicate Number	Digital Temperature Readings (°C) at Minute #						
			0	2	4	6	8	10	12
8	S.W. 8	3	24.4	27.4	30.2	32.4	34.4	36.2	
8	S.W. 9	4	25.0	27.9	30.5	32.7	34.7	36.4	
8	S.W. 10	5	24.6	27.3	29.5	31.6	33.3	34.7	
9	S.W. 11	1	24.4	28.9	31.9	34.5	36.4	37.9	
9	S.W. 12	2	25.1	29.8	33.8	37.0	39.5	41.4	
9	S.W. 13	3	24.2	29.1	33.4	36.7	38.7	40.0	
9	S.W. 14	4	24.3	29.0	32.8	25.9	38.6	40.4	
9	S.W. 15	5	25.0	29.2	32.4	35.2	37.2	38.6	
10	LS54 1	1	21.4	22.0	22.6	23.4	24.0	24.3	22.9
10	LS54 2	2	21.3	22.1	22.9	23.5	24.0	24.6	23.5
10	LS54 3	3	21.2	22.1	22.6	23.3	23.8	24.3	23.4
10	LS54 4	4	21.2	22.0	22.6	23.3	23.7	24.3	23.5
10	LS54 5	5	21.0	21.7	22.6	23.3	23.6	23.8	22.5
11	LS54 6	1	21.2	24.6	27.4	30.1	32.5	33.3	32.0
11	LS54 7	2	21.3	24.4	27.2	29.6	32.8	33.6	32.0
11	LS54 8	3	21.2	24.3	27.1	29.4	31.7	33.5	32.3
11	LS54 9	4	21.7	24.8	27.7	30.0	32.3	34.1	32.9
11	LS54 10	5	21.6	24.5	27.2	29.6	31.7	33.6	32.7
12	LS54 11	1	21.5	26.1	30.1	33.4	35.9	37.6	36.3
12	LS54 12	2	21.6	25.9	30.0	33.2	36.0	38.3	36.2
12	LS54 13	3	21.8	26.6	31.3	35.5	38.5	41.0	38.5
12	LS54 14	4	21.4	26.2	30.5	33.9	37.1	39.6	37.8
12	LS54 15	5	21.1	25.9	30.5	33.5	36.4	38.8	36.8
13	T.S.C. 1	1	23.1	24.2	25.0	25.7	26.5	27.0	25.7
13	T.S.C. 2	2	22.7	23.8	24.8	25.3	26.4	26.8	25.1
13	T.S.C. 3	3	22.5	23.6	24.4	25.1	25.9	26.3	24.9
13	T.S.C. 4	4	22.6	23.4	23.8	24.3	24.8	25.3	22.8
13	T.S.C. 5	5	22.3	23.0	23.5	24.0	24.5	25.0	23.3
14	T.S.C. 6	1	21.8	25.3	28.3	31.3	33.4	35.9	35.0
14	T.S.C. 7	2	21.8	25.3	28.4	31.1	33.4	35.5	33.6
14	T.S.C. 8	3	21.7	25.2	28.2	30.9	33.2	36.2	34.9
14	T.S.C. 9	4	21.6	25.0	27.8	30.5	32.7	34.9	33.2
14	T.S.C. 10	5	21.5	24.8	27.6	30.0	32.1	33.8	32.1
15	T.S.C. 11	1	21.6	26.8	31.7	35.3	38.0	40.0	38.3
15	T.S.C. 12	2	21.6	26.6	31.0	35.1	38.2	40.1	38.4
15	T.S.C. 13	3	21.6	26.5	30.8	34.0	36.8	39.0	37.0
15	T.S.C. 14	4	21.6	26.6	30.7	34.2	36.7	39.1	37.4
15	T.S.C. 15	5	21.6	26.8	31.3	34.2	36.9	41.3	38.7
16	S.C.M.C. 1	1	20.3	20.6	22.3	23.0	24.0	24.6	23.9
16	S.C.M.C. 2	2	20.2	21.7	22.5	23.5	24.2	25.0	24.3
16	S.C.M.C. 3	3	20.3	21.8	22.7	23.7	24.2	25.1	24.3
16	S.C.M.C. 4	4	20.3	21.6	22.4	23.5	24.2	25.1	24.3

Group	Sample ID Number	Replicate Number	Digital Temperature Readings (°C) at Minute #						
			0	2	4	6	8	10	12
16	S.C.M.C. 5	5	21.0	22.1	23.1	23.9	24.6	25.2	24.5
17	S.C.M.C. 6	1	21.3	24.7	27.7	30.2	32.4	34.2	33.1
17	S.C.M.C. 7	2	21.3	24.7	27.6	29.9	32.0	33.9	32.5
17	S.C.M.C. 8	3	21.2	24.4	27.3	29.8	32.0	33.9	32.3
17	S.C.M.C. 9	4	21.2	24.4	27.1	29.6	31.7	33.6	31.9
17	S.C.M.C. 10	5	21.4	24.5	27.3	29.8	31.9	33.7	32.3
18	S.C.M.C. 11	1	22.6	27.6	31.4	34.7	36.7	38.3	36.2
18	S.C.M.C. 12	2	21.5	26.6	30.7	34.0	36.3	38.0	35.8
18	S.C.M.C. 13	3	21.6	26.7	31.1	34.5	37.5	39.6	37.0
18	S.C.M.C. 14	4	21.7	26.7	30.8	34.4	37.5	39.6	37.3
18	S.C.M.C. 15	5	21.2	26.4	30.0	33.0	36.8	39.4	37.9

pH

pH readings taken throughout Phase 1 testing, discussed in *Chapter 8.*

Group	Sample ID Number	Replicate Number	Digital pH Meter Reading at Minute #							Paper pH Reading at Minute #	
			0	2	4	6	8	10	12	0	10
1	D.G. 1	1	7.72	7.29	7.06	6.89	6.67	6.40		7.5	7.5
1	D.G. 2	2	6.75	6.70	6.58	6.45	6.36	6.16		7.5	7.5
1	D.G. 3	3	6.93	6.45	6.35	6.27	6.20	6.11		7.5	7.5
1	D.G. 4	4	5.84	5.89	5.89	5.85	5.61	5.80		7.0	7.0
1	D.G. 5	5	5.83	5.80	5.88	5.90	5.82	5.82		7.5	7.5
2	D.G. 6	1	6.63	6.25	6.12	6.05	6.05	6.89		7.5	7.5
2	D.G. 7	2	6.42	7.20	7.40	7.51	7.60	7.57		7.5	7.5
2	D.G. 8	3	7.61	8.05	7.85	7.81	7.94	7.84		7.5	7.5
2	D.G. 9	4	7.80	7.74	8.01	8.00	7.85	7.83		7.5	7.5
2	D.G. 10	5	7.78	8.01	7.92	7.80	7.80	7.73		7.5	7.5
3	D.G. 11	1	7.86	7.94	7.61	7.67	7.63	7.63		7.5	7.5
3	D.G. 12	2	6.52	7.03	7.18	7.29	7.35	7.36		7.5	7.5
3	D.G. 13	3	7.66	7.45	7.51	7.40	7.52	7.51		7.5	7.5
3	D.G. 14	4	7.39	7.61	7.57	7.64	7.60	7.64		7.5	7.5
3	D.G. 15	5	7.64	7.66	7.70	7.63	7.56	7.59		7.5	7.5
4	D.I. 1	1	6.78	6.88	6.53	6.32	6.51	6.95		7.0	7.0
4	D.I. 2	2	7.13	6.79	7.15	7.25	7.34	7.38		7.0	7.0
4	D.I. 3	3	6.98	6.45	6.21	6.04	5.95	5.79		7.0	7.0
4	D.I. 4	4	6.79	6.57	6.94	7.16	7.20	7.22		7.0	7.0

Group	Sample ID Number	Replicate Number	Digital pH Meter Reading at Minute #							Paper pH Reading at Minute #	
			0	2	4	6	8	10	12	0	10
4	D.I. 5	5	7.23	6.85	6.61	6.47	6.28	6.12		7.0	7.0
5	D.I. 6	1	6.15	5.18	5.08	5.02	5.01	4.96		4.9	7.0
5	D.I. 7	2	5.88	5.81	5.73	5.69	5.75	5.59		7.0	7.0
5	D.I. 8	3	6.13	5.77	5.75	5.86	5.70	5.63		7.0	7.0
5	D.I. 9	4	6.38	6.18	5.98	5.85	5.86	5.92		7.0	7.0
5	D.I. 10	5	6.30	5.67	5.54	5.50	5.46	5.40		7.0	7.0
6	D.I. 11	1	6.02	6.79	7.20	7.42	7.53	7.59		7.0	7.0
6	D.I. 12	2	6.63	7.25	7.42	7.51	7.48	7.53		7.0	7.0
6	D.I. 13	3	7.26	6.83	6.54	6.26	6.05	5.85		7.0	7.0
6	D.I. 14	4	5.54	5.53	5.52	5.67	5.67	5.68		7.0	7.0
6	D.I. 15	5	5.83	5.90	6.03	5.99	6.09	6.02		7.0	7.0
7	S.W. 1	1	7.25	7.35	7.37	7.36	7.37	7.35		7.5	7.5
7	S.W. 2	2	7.45	7.43	7.49	7.41	7.38	7.38		7.5	7.5
7	S.W. 3	3	7.18	7.43	7.44	7.43	7.41	7.38		7.5	7.5
7	S.W. 4	4	7.22	7.43	7.41	7.39	7.37	7.36		7.5	7.5
7	S.W. 5	5	7.45	7.44	7.41	7.40	7.37	7.35		7.5	7.5
8	S.W. 6	1	7.39	7.32	7.28	7.23	7.23	7.18		7.5	7.5
8	S.W. 7	2	7.54	7.44	7.38	7.29	7.25	7.20		7.5	7.5
8	S.W. 8	3	7.51	7.40	7.35	7.27	7.26	7.22		7.5	7.5
8	S.W. 9	4	7.43	7.37	7.32	7.27	7.21	7.19		7.5	7.5
8	S.W. 10	5	7.45	7.37	7.31	7.25	7.22	7.18		7.5	7.5
9	S.W. 11	1	7.46	7.42	7.28	7.21	7.18	7.15		7.5	7.5
9	S.W. 12	2	7.38	7.25	7.18	7.15	7.12	7.10		7.5	7.5
9	S.W. 13	3	7.52	7.31	7.21	7.16	7.12	7.12		7.5	7.5
9	S.W. 14	4	7.46	7.32	7.23	7.18	7.13	7.10		7.5	7.5
9	S.W. 15	5	7.39	7.52	7.38	7.26	7.21	7.17		7.5	7.5
10	LS54 1	1	7.38	7.56	7.56	7.43	7.38	7.25	6.69	7.5	7.5
10	LS54 2	2	7.00	6.88	6.97	9.92	6.90	6.78	6.51	7.5	7.5
10	LS54 3	3	7.02	7.02	6.91	6.95	6.88	6.61	6.49	7.5	7.5
10	LS54 4	4	6.53	6.65	6.81	6.78	6.76	6.66	6.51	7.0	7.0
10	LS54 5	5	6.48	6.67	6.93	6.97	6.99	7.00	6.42	7.0	7.0
11	LS54 6	1	6.80	6.98	6.88	6.99	7.05	7.05	6.37	7.0	7.0
11	LS54 7	2	6.91	6.98	7.01	7.08	7.07	7.06	6.34	7.5	7.0
11	LS54 8	3	6.95	6.96	7.06	7.02	7.12	7.09	6.48	7.5	7.0
11	LS54 9	4	7.23	7.29	7.17	7.16	7.28	7.20	6.56	7.5	7.0
11	LS54 10	5	6.95	6.68	6.82	6.75	6.98	6.96	6.37	7.5	7.5
12	LS54 11	1	7.18	7.14	7.16	7.21	7.14	7.22	6.77	7.5	7.5
12	LS54 12	2	7.07	7.24	7.08	7.11	7.14	7.13	6.63	7.5	7.0
12	LS54 13	3	7.09	7.12	7.14	7.12	7.18	7.16	6.61	7.5	7.0
12	LS54 14	4	7.04	7.10	7.08	7.11	7.15	7.09	6.62	7.5	7.0
12	LS54 15	5	7.13	7.05	7.12	7.15	7.18	7.06	6.60	7.5	7.0

Group	Sample ID Number	Replicate Number	Digital pH Meter Reading at Minute #							Paper pH Reading at Minute #	
			0	2	4	6	8	10	12	0	10
13	T.S.C. 1	1	8.18	8.07	7.97	7.89	7.83	7.88	7.94	8.0	7.5
13	T.S.C. 2	2	8.15	7.93	7.91	7.81	7.81	7.81	7.83	8.0	7.5
13	T.S.C. 3	3	8.22	7.95	7.85	7.84	7.82	7.79	7.81	7.5	7.5
13	T.S.C. 4	4	8.22	7.92	7.87	7.82	7.82	7.79	7.79	7.5	7.5
13	T.S.C. 5	5	8.22	7.90	7.85	7.80	7.77	7.75	7.78	8.0	7.5
14	T.S.C. 6	1	8.06	7.80	7.79	7.67	7.64	7.61	7.64	8.0	7.5
14	T.S.C. 7	2	8.13	7.79	7.71	7.72	7.68	7.64	7.72	8.0	7.5
14	T.S.C. 8	3	8.11	7.80	7.70	7.68	7.65	7.66	7.69	8.0	7.5
14	T.S.C. 9	4	8.68	7.73	7.68	7.65	7.66	7.66	7.65	7.5	7.5
14	T.S.C. 10	5	8.06	7.74	7.70	7.65	7.60	7.62	7.63	7.5	7.5
15	T.S.C. 11	1	7.99	7.66	7.61	7.55	7.54	7.53	7.54	7.5	7.5
15	T.S.C. 12	2	8.01	7.65	7.56	7.53	7.53	7.53	7.48	7.5	7.0
15	T.S.C. 13	3	7.95	7.75	7.68	7.66	7.62	7.61	7.65	7.5	7.5
15	T.S.C. 14	4	7.91	7.73	7.66	7.65	7.65	7.68	7.75	7.5	7.5
15	T.S.C. 15	5	7.98	7.63	7.68	7.65	7.63	7.70	7.72	7.5	7.5
16	S.C.M.C. 1	1	7.06	6.92	6.84	6.81	6.69	6.76	6.67	7.0	7.0
16	S.C.M.C. 2	2	6.81	6.69	6.67	6.67	6.68	6.63	6.66	7.0	7.0
16	S.C.M.C. 3	3	6.76	6.60	6.61	6.65	6.60	6.64	6.61	7.0	7.0
16	S.C.M.C. 4	4	6.73	6.59	6.56	6.63	6.64	6.63	6.62	7.0	7.0
16	S.C.M.C. 5	5	6.72	6.59	6.64	6.62	6.61	6.64	6.62	7.0	7.0
17	S.C.M.C. 6	1	6.87	6.72	6.74	6.73	6.73	6.75	6.77	7.0	7.0
17	S.C.M.C. 7	2	6.78	6.69	6.71	6.72	6.75	6.72	6.76	7.0	7.0
17	S.C.M.C. 8	3	6.80	6.70	6.72	6.76	6.76	6.71	6.80	7.0	7.0
17	S.C.M.C. 9	4	6.82	6.72	6.73	6.72	6.78	6.80	6.72	7.0	7.0
17	S.C.M.C. 10	5	6.78	6.68	6.71	6.73	6.77	6.73	6.78	7.0	7.0
18	S.C.M.C. 11	1	6.92	6.73	6.74	6.77	6.77	6.76	6.84	7.0	7.0
18	S.C.M.C. 12	2	6.86	6.77	6.79	6.79	6.77	6.81	6.77	7.0	7.0
18	S.C.M.C. 13	3	6.94	6.74	6.79	6.79	6.82	6.75	6.82	7.0	7.0
18	S.C.M.C. 14	4	6.85	6.71	6.79	6.82	6.85	6.81	6.72	7.0	7.0
18	S.C.M.C. 15	5	6.83	6.72	6.76	6.80	6.79	6.86	6.74	7.0	7.0

Aluminium Foil Samples

Physical samples of aluminium foil included in print copy only.

Appendix E Phase 2 Data and Imaging

Temperature and pH

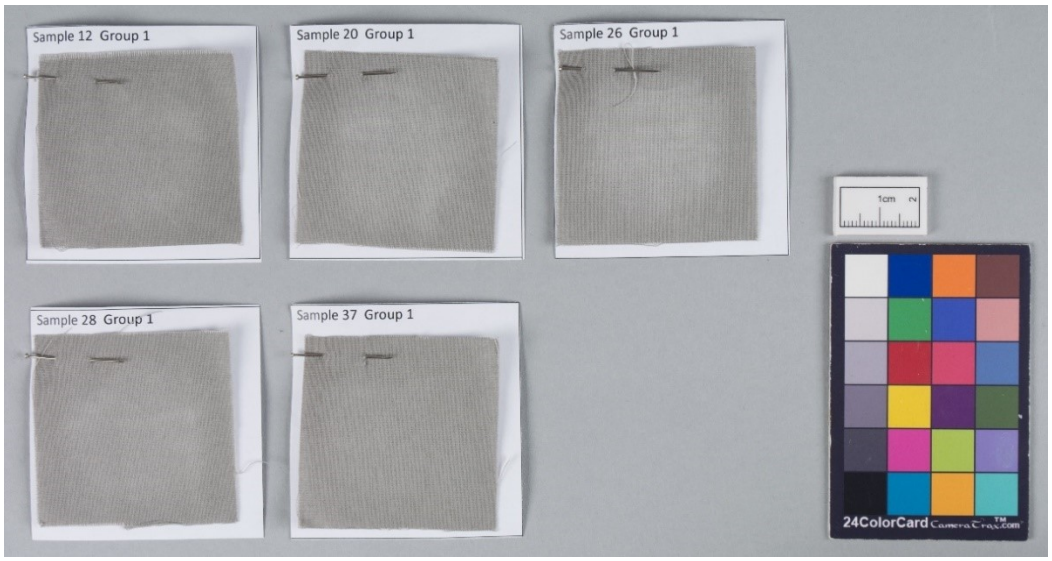
Temperature (°C) and pH were recorded during Phase 2 in *Chapter 9*. No trends were seen, and initial analysis showed wide standard deviation, so further analysis was not pursued. Temperature and pH were recorded in tandem throughout the test cleaning, with four recordings taken. Recording 1 was taken before the first application of ultrasound and recording 2 was taken after ending ultrasound. Recording 3 was taken before the second application of ultrasound and recording 4 was taken after ending ultrasound.

Group Number	Sample Number	Replicate Number	pH 1	pH 2	pH 3	pH 4	temp 1	temp 2	temp 3	temp 4
1	12	1	6.93	6.79	6.80	6.67	20.1	20.1	20.7	20.5
1	20	2	6.55	6.32	6.13	6.29	20.0	20.2	21.4	21.7
1	26	3	6.36	6.19	6.46	6.08	20.7	20.7	20.8	20.9
1	28	4	5.98	6.12	6.17	5.84	21.3	21.2	23.0	22.5
1	37	5	6.74	6.01	6.50	6.06	19.6	20.2	20.2	20.5
2	2	1	7.21	6.9	6.98	6.53	24.8	24.4	24.5	24.3
2	35	2	6.42	6.27	6.36	6.26	22.6	22.5	24.3	24.0
2	39	3	6.16	6.05	6.13	5.97	22.1	21.9	23.7	23.6
2	40	4	6.15	5.98	5.93	5.89	22.9	22.9	23.5	23.3
2	45	5	6.22	5.86	5.97	5.84	23.0	23.2	23.3	23.6
3	7	1	7.80	7.67	7.58	7.25	25.1	24.5	26.0	25.5
3	17	2	6.76	6.63	6.68	6.68	24.9	24.2	25.4	24.9
3	29	3	6.31	6.24	6.43	6.09	24.8	24.1	25.2	24.7
3	33	4	6.24	6.07	6.23	5.96	25.0	24.7	25.7	25.5
3	34	5	6.20	5.97	6.46	5.94	25.0	24.8	26.0	26.0
4	1	1	5.92	5.74	5.81	5.34	23.6	22.8	24.0	23.5
4	5	2	5.34	5.39	5.33	5.23	23.0	22.9	24.2	23.1
4	8	3	6.23	6.10	6.12	6.18	22.5	22.6	23.9	22.3
4	11	4	6.54	6.05	6.32	5.74	22.5	22.3	23.3	23.4
4	44	5	6.26	5.93	6.31	6.52	22.4	22.2	22.3	22.4
5	10	1	7.03	6.50	6.37	6.08	24.6	23.9	25.2	25.0
5	14	2	6.85	6.54	6.61	6.03	24.2	23.9	24.6	23.9
5	23	3	6.67	6.35	6.53	6.09	23.1	23.1	24.3	23.9
5	32	4	6.71	6.54	6.69	6.05	23.2	23.2	23.3	24.0
5	42	5	6.98	6.49	6.53	6.19	23.0	23.0	23.2	23.8
6	4	1	6.40	6.19	6.07	5.80	23.8	23.4	24.4	24.3
6	15	2	6.00	5.92	5.35	6.28	22.5	22.4	23.0	22.8
6	16	3	5.77	5.75	6.03	5.70	22.4	22.2	22.4	22.9
6	30	4	5.98	5.88	5.76	5.68	21.6	21.5	22.2	22.4
6	43	5	7.02	6.35	6.98	6.09	21.5	21.7	22.0	21.9
7	9	1	7.96	7.93	7.92	8.04	22.1	22.1	22.1	22.3
7	13	2	8.1	7.99	8.1	8.04	22.0	21.7	22.5	22.2

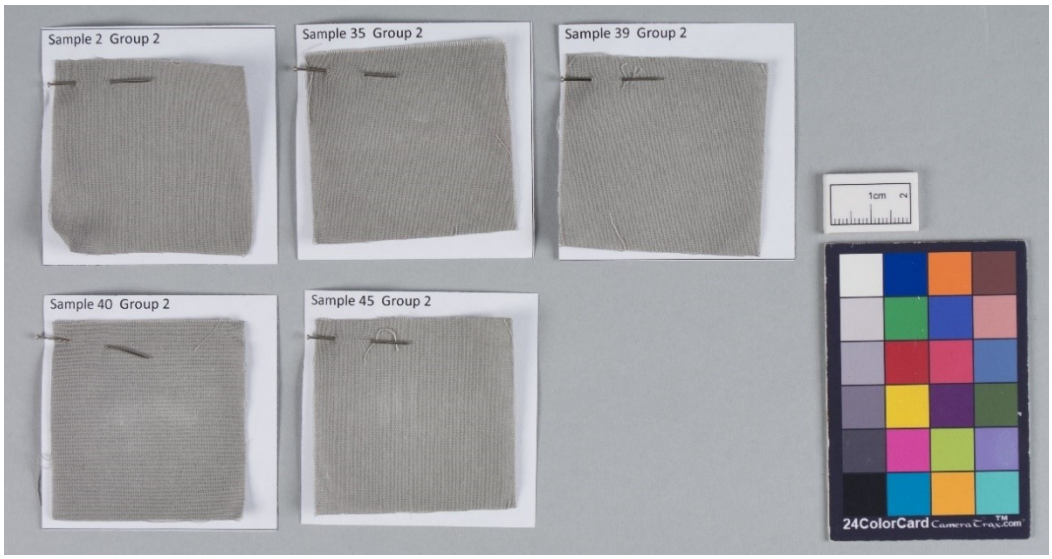
Group Number	Sample Number	Replicate Number	pH 1	pH 2	pH 3	pH 4	temp 1	temp 2	temp 3	temp 4
7	31	3	8.07	7.99	8.10	8.00	21.8	21.7	22.5	22.3
7	36	4	8.04	7.96	8.08	8.04	21.9	21.3	21.7	22.2
7	41	5	8.01	7.9	8.03	7.99	22.0	21.6	22.0	22.2
8	3	1	7.90	7.82	7.98	7.89	21.9	22.3	22.0	22.7
8	6	2	7.92	7.88	7.98	7.9	22.1	22.0	22.4	22.6
8	19	3	7.90	7.81	7.89	7.85	22.0	21.8	22.5	22.7
8	21	4	7.93	7.79	7.91	7.89	22.1	22.3	22.1	22.3
8	25	5	7.92	7.84	7.89	7.88	23.4	23.5	24.3	24.1
9	18	1	8.02	7.88	7.98	7.97	22.4	22.1	23.2	23.6
9	22	2	8.02	7.92	8.07	8.01	23.0	22.4	23.8	23.1
9	24	3	8.05	7.93	8.04	7.96	22.7	22.4	23.5	23.0
9	27	4	7.98	7.86	8.06	7.99	22.6	22.4	23.3	23.3
9	38	5	7.99	7.88	7.93	7.94	22.8	22.6	23.2	22.9

After Treatment Imaging of Test Fabric

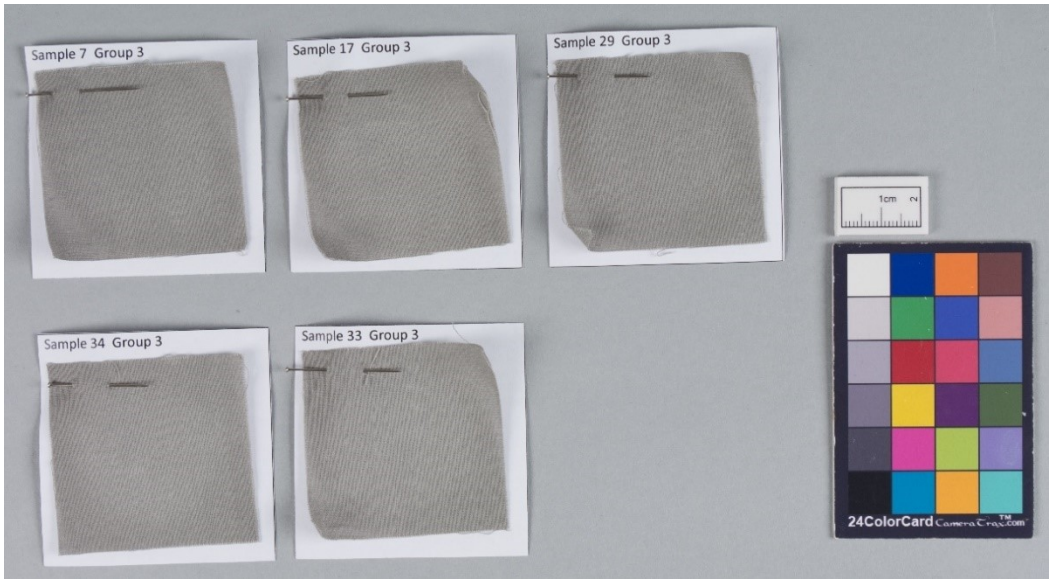
As discussed in *sections 9.2, 9.4.2, 9.5.*



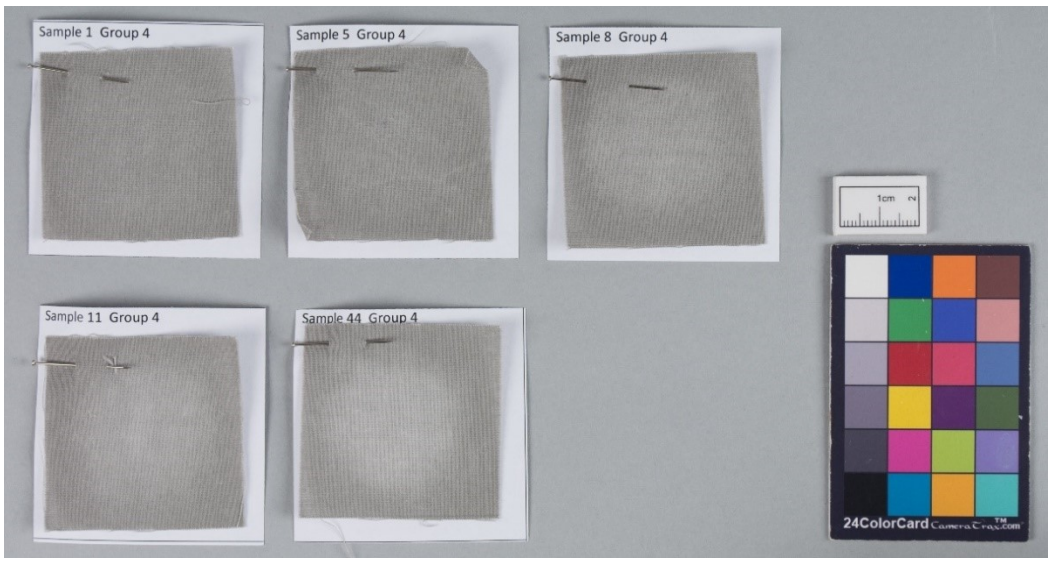
Group 1: deionised water at 40% amplitude.



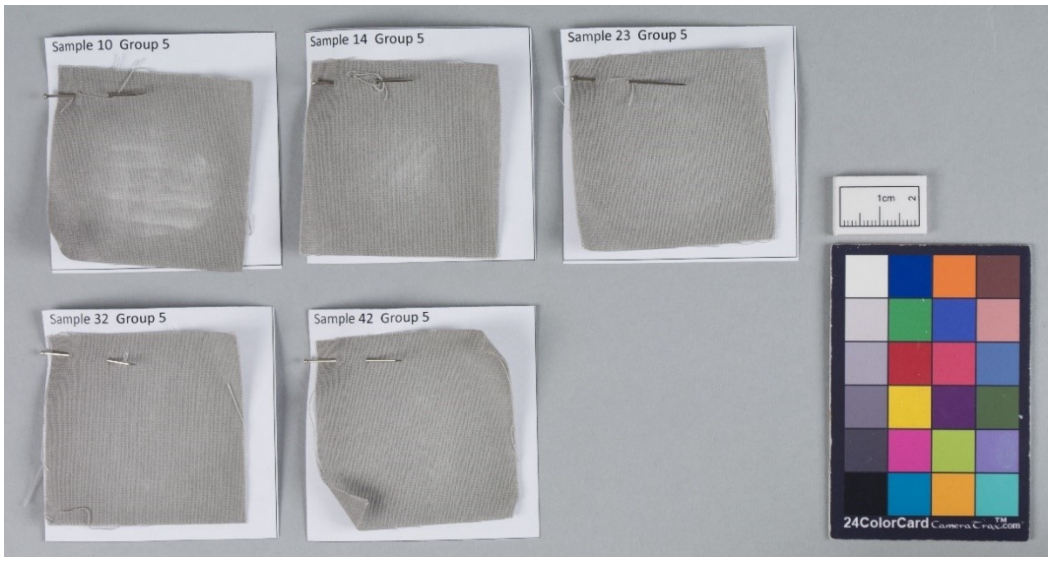
Group 2: deionised water at 60% amplitude.



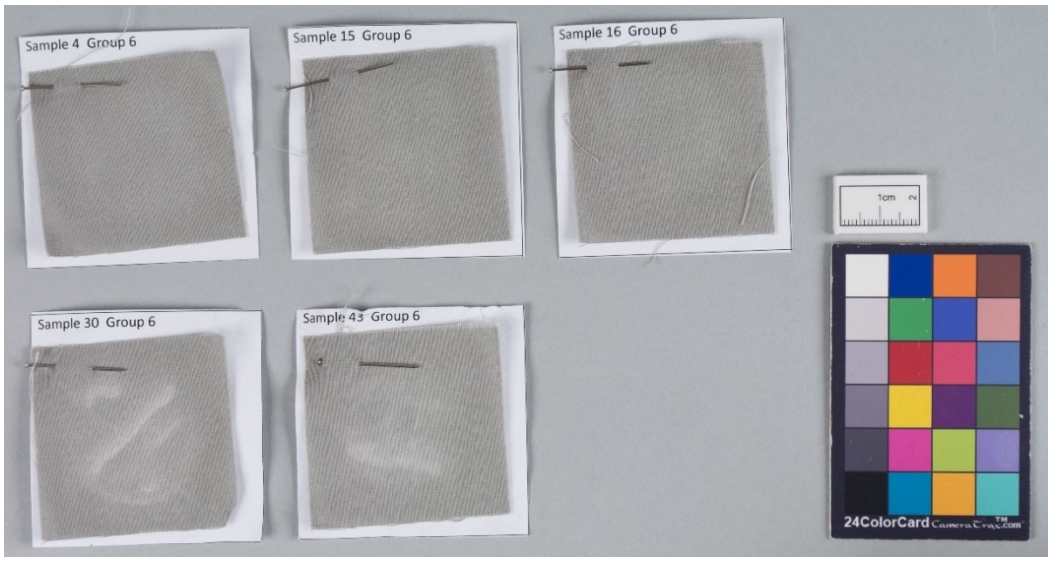
Group 3: deionised water at 80% amplitude.



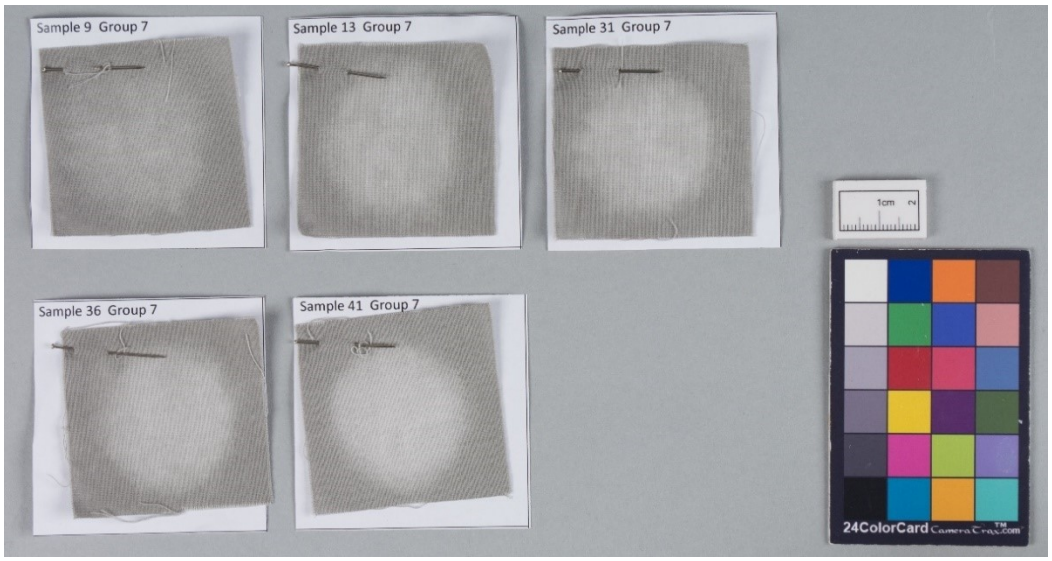
Group 4: 0.3% w/v Dehypon® LS54 at 40% amplitude.



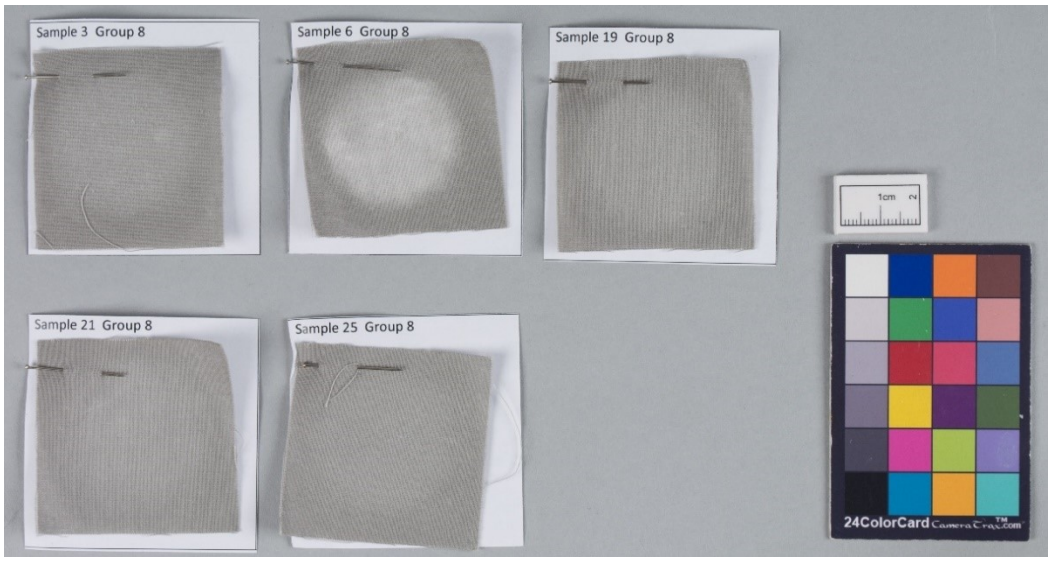
Group 5: 0.3% w/v Dehypon® LS54 at 60% amplitude.



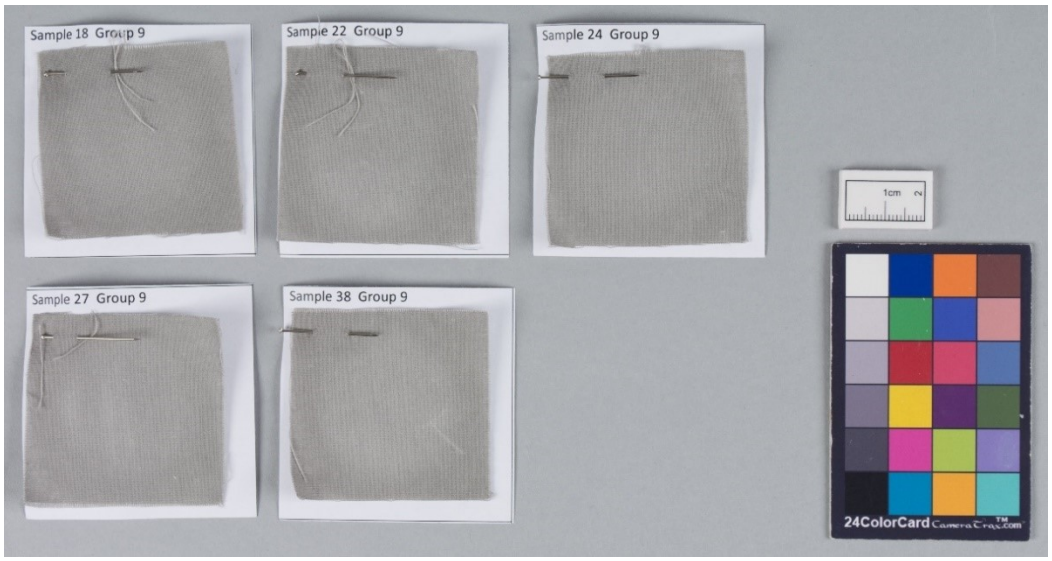
Group 6: 0.3% w/v Dehypon® LS54 at 80% amplitude.



Group 7: 0.5 g/L trisodium citrate and 0.05 g/L SCMC at 40% amplitude.



Group 8: 0.5 g/L trisodium citrate and 0.05 g/L SCMC at 60% amplitude.



Group 9: 0.5 g/L trisodium citrate and 0.05 g/L SCMC at 80% amplitude.

Mass Data

As discussed in *sections 9.3, 9.4.4*.

Group Number	Sample Number	Replicate Number	Before Treatment Mass (g)	After Treatment Mass (g)
1	12	1	0.305	0.304
1	20	2	0.307	0.303
1	26	3	0.306	0.302
1	28	4	0.305	0.300
1	37	5	0.306	0.296
2	2	1	0.305	0.302
2	35	2	0.302	0.296
2	39	3	0.303	0.297
2	40	4	0.304	0.298
2	45	5	0.305	0.300
3	7	1	0.305	0.299
3	17	2	0.302	0.294
3	29	3	0.308	0.301
3	33	4	0.302	0.296
3	34	5	0.296	0.292
4	1	1	0.299	0.294
4	5	2	0.305	0.297
4	8	3	0.305	0.299
4	11	4	0.309	0.302
4	44	5	0.308	0.302
5	10	1	0.309	0.303
5	14	2	0.309	0.303
5	23	3	0.307	0.301
5	32	4	0.305	0.300
5	42	5	0.303	0.3
6	4	1	0.303	0.298
6	15	2	0.306	0.302
6	16	3	0.303	0.298
6	30	4	0.305	0.297
6	43	5	0.305	0.298
7	9	1	0.305	0.3
7	13	2	0.306	0.298
7	31	3	0.304	0.294
7	36	4	0.306	0.299
7	41	5	0.305	0.295
8	3	1	0.307	0.301
8	6	2	0.306	0.298
8	19	3	0.300	0.293
8	21	4	0.308	0.304
8	25	5	0.308	0.303

Group Number	Sample Number	Replicate Number	Before Treatment Mass (g)	After Treatment Mass (g)
9	18	1	0.302	0.296
9	22	2	0.307	0.304
9	24	3	0.305	0.299
9	27	4	0.301	0.296
9	38	5	0.306	0.300

Colourimetry Data

As discussed in *sections 9.3, 9.4.5.*

Group Number	Sample Number	Replicate Number	Before Treatment			After Treatment		
			L*(D65)	a*(D65)	b*(D65)	L*(D65)	a*(D65)	b*(D65)
1	12	1	55.51	0.79	3.00	60.97	0.77	2.89
1	20	2	56.70	0.78	2.79	63.41	0.74	2.61
1	26	3	57.12	0.73	2.94	65.75	0.77	2.78
1	28	4	55.53	0.80	2.96	62.15	0.77	2.82
1	37	5	56.70	0.79	3.02	60.53	0.77	2.78
2	2	1	56.14	0.79	2.98	59.98	0.74	2.71
2	35	2	55.10	0.77	3.08	60.15	0.87	2.99
2	39	3	57.25	0.73	2.79	60.92	0.75	2.76
2	40	4	57.37	0.73	2.93	63.57	0.74	2.77
2	45	5	57.00	0.75	2.86	62.87	0.77	2.80
3	7	1	55.08	0.76	3.06	59.67	0.75	2.84
3	17	2	55.32	0.83	3.32	60.70	0.82	3.03
3	29	3	55.40	0.80	3.04	59.90	0.78	2.86
3	33	4	55.69	0.77	3.02	61.24	0.74	2.69
3	34	5	55.73	0.78	3.09	61.64	0.79	2.93
4	1	1	55.94	0.80	3.12	63.02	0.76	2.78
4	5	2	55.17	0.78	3.05	61.88	0.76	2.81
4	8	3	55.28	0.78	3.11	64.82	0.74	2.76
4	11	4	55.45	0.80	2.95	67.74	0.79	3.10
4	44	5	56.97	0.73	2.94	68.98	0.64	2.55
5	10	1	55.60	0.81	3.07	68.04	0.77	2.89
5	14	2	55.25	0.77	3.10	65.91	0.73	2.74
5	23	3	57.43	0.73	2.92	64.15	0.70	2.67
5	32	4	55.99	0.75	2.99	63.62	0.73	2.74
5	42	5	57.11	0.74	2.79	64.85	0.66	2.39
6	4	1	55.49	0.78	3.09	61.11	0.71	2.64
6	15	2	55.16	0.79	3.16	61.63	0.71	2.71
6	16	3	55.73	0.80	3.01	61.09	0.76	2.80
6	30	4	55.47	0.78	2.86	64.30	0.74	2.78

Group Number	Sample Number	Replicate Number	Before Treatment			After Treatment		
			L*(D65)	a*(D65)	b*(D65)	L*(D65)	a*(D65)	b*(D65)
6	43	5	57.23	0.73	2.87	63.17	0.72	2.66
7	9	1	55.36	0.80	2.98	67.36	0.74	2.97
7	13	2	55.07	0.78	3.03	71.96	0.78	3.24
7	31	3	56.20	0.77	2.86	73.39	0.77	3.21
7	36	4	55.12	0.78	3.12	72.96	0.79	3.27
7	41	5	57.31	0.73	2.85	77.42	0.70	3.28
8	3	1	55.96	0.79	3.01	65.85	0.71	2.72
8	6	2	55.37	0.77	3.10	75.10	0.78	3.41
8	19	3	56.58	0.76	3.06	64.88	0.72	2.68
8	21	4	57.00	0.76	2.83	65.22	0.73	2.80
8	25	5	57.07	0.71	2.86	63.46	0.70	2.55
9	18	1	55.45	0.81	3.04	63.02	0.74	2.80
9	22	2	57.18	0.72	2.81	62.92	0.73	2.61
9	24	3	57.56	0.70	2.83	62.92	0.71	2.66
9	27	4	56.77	0.73	2.91	63.69	0.72	2.80
9	38	5	56.90	0.78	2.81	63.73	0.76	2.67

Test Fabric Samples

Physical samples of test fabric included in print copy only.