THE CHARACTERIZATION OF FOXING ON TEXTILES

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ABSTRACT—In 2016, exploratory research evaluating common methods used to characterize foxing on textiles was undertaken by the author as a postgraduate intern at the Canadian Conservation Institute. The project involved a literature review exploring foxing and related degradation phenomena on textiles, a survey distributed to textile conservators around the world, and case-study testing on a foxed textile. UV fluorescence observation and adenosine triphosphate/adenosine monophosphate (ATP/AMP) bioluminescence testing were compared for their ability to identify microbiological activity in foxing stains. Levels of microbiological activity were found not to correlate with levels of ultraviolet fluorescence. XRF spectroscopy and bathophenanthroline strip testing were compared as methods for characterizing iron-catalyzed degradation in foxing. Although trace amounts of iron were detected, XRF analysis did not find the iron content to be higher in foxing spots. The textile did not test positive for reactive iron ions by bathophenanthroline strip testing, indicating that the foxed textile was not at risk of iron-catalyzed deterioration.

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RESUMEN—En 2016, el autor realizó una investigación exploratoria para evaluar los métodos comunes utilizados para caracterizar el foxing en los textiles, como pasante de posgrado en el Instituto Canadiense de Conservación. El proyecto incluyó una revisión de la literatura sobre el fenómeno de foxing y la degradación relacionada en los textiles, una encuesta distribuida a conservadores de textiles de todo el mundo y un caso práctico sobre un textil con foxing. La observación de fluorescencia ultravioleta y la prueba de bioluminiscencia adenosín trifosfato/adenósín monofosfato (ATP/AMP) se compararon por su capacidad para identificar la actividad microbiológica en las manchas de foxing. Se encontró que los niveles de actividad microbiológica no se correlacionan con los niveles de fluorescencia ultravioleta. La espectrometría de XRF y pruebas con bathofenantrolina se compararon como métodos para caracterizar la degradación catalizada por hierro en el foxing. Si bien se detectaron pequeñas cantidades de hierro, el análisis de XRF no encontró que el contenido de hierro fuera más alto en las zonas de foxing. El textil no arrojó resultados positivos para los iones reactivos de hierro mediante la prueba de bathofenantrolina, lo que indica que el textil con foxing no estaba en riesgo de deterioro catalizado por hierro.

1. INTRODUCTION

"Foxing," a term used to describe yellow to brown spotted staining, has long been researched and debated in the paper conservation literature and has been attributed to fungal activity, metal ion–catalyzed degradation, localized moisture condensation, or a combination of these factors. Although a visually similar phenomenon is frequently observed on textiles and the term foxing has been adopted by textile conservators, it has not yet been sufficiently characterized in a textile context. Existing literature on foxing is wide-ranging, and investigations into the chemical nature and causes of this type of degradation follow often-conflicting streams of research. Despite significant research and analysis on foxing, there seems to be no clear consensus on its cause. This ambivalence, along with overly general application of the term foxing as a catchall, has caused some convolution in how foxing is characterized and therefore treated.
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The plenitude of paper conservation research is a tremendous resource for textile conservators, but it still remains to be determined which correlations can and cannot be made between foxing on paper and foxing on cellulosic textiles. An initial study of foxing on textiles was necessary—one that explored the methods currently used to assess foxing and attempted to clarify foxing research in textile conservation.

An exploratory research project was undertaken as part of the 2016 postgraduate internship in textile conservation at the Canadian Conservation Institute (CCI). The author carried out the project under the supervision of CCI textile conservator Renée Dancouse. The project began with a literature review of existing foxing research to demarcate the common ground between paper and textile conservation and which factors specific to textiles may require the issue to be considered differently. To supplement this, a survey was distributed to conservators around the world in an effort to establish how most textile conservators use the term foxing, and gauge the magnitude of this issue for textiles in heritage collections. A case study of a foxed study-collection tablecloth was then used to explore and evaluate the advantages and limitations of different characterization methods commonly used in the examination of foxing.

2. LITERATURE REVIEW

The literature review initially covered roughly 30 years of conservation research on foxing stains, as well as related issues such as the treatment of iron oxide stains, mildew staining, and the formation of “brown line” staining at wet-dry interfaces on cellulosic textiles (Bogaty 1952). Because there was paucity of conservation literature regarding foxing specific to textiles, the review was expanded beyond the conservation sphere and examined the textile industry and dry cleaning industry literature for mention and discussion of foxing on textiles. Very little such material was found, and the cause of so little mention of foxing on textiles was questioned when it is ostensibly observed as a common issue in textile collections. This absence in the textile industry and dry cleaning industry literature seemed to suggest that foxing is not an issue for textiles that are still in regular use, but rather seems to be unique to heritage collections. More generally, foxing on textiles appears to be specific to textiles that are stored for extended periods of time.

With this in mind, the focus of the literature survey turned to a non-museum context where textiles were routinely placed into storage for long periods: large Victorian households. Nineteenth century household and laundry manuals were studied in the hopes of finding mention of foxing in a textile context. Although the term foxing was never used in these texts, the term “ironmould” was found to closely match today’s interpretation of the phenomenon. This term seemed to perfectly encapsulate the debate and confusion about metal versus fungal catalysts of foxing. In their book, Crinolines and Crimping Irons. Victorian Clothes: How They Were Cleaned and Cared For, Walkley and Foster (1978) describe ironmould just as textile conservators might describe foxing: as spotted discoloration formed on linens during storage, caused by damp conditions and fungal activity. However, their description is not consistent with all other references to ironmould in 19th-century literature; the term seems to have been used widely to describe iron staining, mildew staining, and even ink spots. Nevertheless, advice on removing ironmould stains was often listed separately from these other types of staining, suggesting that it was conceived of as its own unique phenomenon.

The ancillary exploration into 19th-century household and laundry guides also highlighted some of the cleaning and finishing techniques used on historic textiles that may have contributed to the development of foxing. These practices further differentiate foxing on textiles from foxing on paper. General laundry
practices, such as the use of copper basins or the boiling of linens in iron or copper kettles (Malcolmsons 1986), are certainly potential early sources of transition metal inclusions that could have catalyzed foxing. The starch finish frequently present on historical cellulosic textiles may also be a contributor to foxing development in providing another food source for microbes. Additionally, starch may also foster an uneven absorption of moisture throughout the textile, resulting in localized degradation. Another example of a historical cleaning practice that could potentially have served as a contributing factor to foxing-like degradation is the process of “blueing” white textiles by adding dilute blue pigment to a final rinse bath after washing to counteract yellowing. Toward the end of the 19th century, most of the blues on the market were in fact Prussian blue pigments and dyes (Richards 1882, 78). Prussian blue, an iron-based mineral pigment, is a potential source of iron ions that could catalyze foxing. It is particularly sensitive to alkali, so a subsequent history of washing these blued textiles with alkaline soaps or other cleaning agents could potentially have resulted in the formation of brown spots similar to foxing. Ellen Swallow Richards actually warned against this latter phenomenon in the 1882 edition of her early home economics text, The Chemistry of Cooking and Cleaning: A Manual for Housekeepers (78).

Although the literature of the Victorian household provides insight into only one particular moment of Western domestic textile history, it shows the relevance of textile-specific properties to the research and assessment of foxing and emphasizes that the paper conservation literature can only go so far in explaining how this phenomenon manifests on textiles.

3. FOXING ON TEXTILES SURVEY

A survey about foxing on textiles was distributed on the Conservation DistList and the TexCons e-mail list in July 2016. The survey received a total of 28 responses from around the globe. The results confirmed that foxing is in fact a common issue that affects textiles: roughly 76% of respondents who work with a medium to large collection observed foxing on approximately 2% or more of their textile collections. There is some debate among textile conservators whether the term, so closely affiliated with a paper degradation issue, should even be used to describe textiles. However, it was found that the term foxing, while apparently used regularly, is not used consistently among textile conservators. In fact, textile conservators do not even reserve the term foxing for cellulosic textiles, as 33% responded that they had observed foxing on silk and 15% on wool.

The most-used description of foxing selected by survey respondents was also the most general: “localized brown spots.” The survey made it clear that a more unified consensus is needed on what the term foxing should be used to describe on textiles. It was felt that the first step toward achieving this would be developing a clear, easy, and accessible means of characterizing foxing and related degradation patterns on textiles. The next phase of the project sought to begin this process by assessing foxing characterization methods using a case-study textile.

4. EVALUATION OF CHARACTERIZATION METHODS THROUGH A CASE-STUDY TEXTILE

While evaluating foxing characterization methods, it was essential to keep in focus the larger context of how the characterization would be used from a practical conservation standpoint. Primarily, how would the characterization most effectively assess levels of active risk associated with the foxing? An additional priority was to focus on the simplest and most accessible characterization methods possible for this goal and compare
their suitability to that of established but less accessible methods, such as XRF. While a full-fledged research project evaluating these methods across many samples would be tremendously useful, the benefit of a case study was that it created the opportunity to evaluate how useful different characterization methods are for informing the treatment decision-making process.

Throughout the exploration of characterization methods, it was noted that identifying the catalyst of the foxing would not necessarily provide a holistic view of the entire system in place that contributed to the textile’s degradation. Other factors, such as the presence of moisture, high humidity, acidic vapors, soiling, or other storage conditions, need to also be considered when characterizing the degradative process. Moreover, the initial cause of the foxing does not necessarily indicate the present, active, associated risks to the textile. For instance, according to Mary-Lou Florian in her article “The Role of the Conidia of Fungi in Fox Spots,” fungal conidia do not often remain viable for longer than 20 years (1996). Therefore in many cases of foxing, if fungal activity was the original catalyst, under correct environmental conditions the textile may no longer be at risk of this form of deterioration. Similarly, in the case of foxing stains catalyzed by metal ions, the presence of metal contaminants such as iron or copper does not necessarily mean that these metal inclusions are actively degrading the textile. As with iron mordants and iron tannate dyes, it is only when free, reactive iron (II) ions are present that iron-induced degradation will occur (specifically, when iron ions catalyze cellulose oxidation through reactions that promote the formation of hydroxyl and alkoxy radicals) (Neve and Reißland 2005). Identifying the presence of overall iron content in the foxed textile is therefore not enough to properly characterize whether the textile continues to be at risk.

Identifying the root cause of the foxing may also not be particularly useful for targeting the treatment of foxing stains from an aesthetic standpoint. Even among those authors who agree that fungal activity is a root cause of foxing, there is some debate as to whether the actual discoloration of foxing spots is a result of staining from melanin pigments contained within the fungal structures and the enzymes they excrete (Aranyanak 1995; Florian and Purinton 1995; Florian 1996; Nieto-Fernandez et al. 2003), or if the color is simply due to the oxidation of the cellulose catalyzed by the microbial activity (Biccheri et al. 2001; Missori, Righini, and Selci 2004; Choi 2007; Boruvka 2008). Similarly, it is necessary to differentiate between the approach for treating iron oxide staining (caused by the corrosion of iron inclusions or direct contact with adjacent corroding iron) and for discoloration caused by iron ion-catalyzed oxidized cellulose (which is a result of conjugated double bond systems formed during oxidation).

4.1 Fungal Activity Assessed by UV Fluorescence and ATP/AMP Bioluminescence Testing

To address the above-mentioned issues, two common methods presently used to diagnose foxing stains—UV fluorescence and XRF—were compared to two alternative, low-tech, more accessible methods—ATP/AMP bioluminescence surface hygiene monitoring and bathophenanthroline testing.

The case-study textile is an embroidered tablecloth in the CCI study collection that exhibited foxing in localized areas (fig. 1). The tablecloth is white cotton, decorated with red and green embroidery. It was highly starched (confirmed by an iodine test) and had been stored folded, likely for quite some time. In general, the foxing was amorphous in shape, scattered, and localized to specific areas, primarily toward two corners of the tablecloth (fig. 2). Foxing spots were all yellow to brown in color but they varied greatly in depth of shade. Some of the foxed areas also suffered from overall oxidative yellowing, particularly along fold lines.
Fig. 1. Foxed case-study textile, Canadian Conservation Institute, 2016.

Fig. 2. Detail of foxing along proper left edge of tablecloth, Canadian Conservation Institute, 2016.
After overall visual inspection, pH testing, and microscopic examination, the foxed tablecloth was observed under a handheld UV light with a wavelength of 360 nm (fig. 3). The use of UV fluorescence is found throughout the literature as a method of identifying foxing (Derow and Owen 1992; Choisy 1997; Bicchieri 2001; Mina 2016). However, the utility of UV fluorescence in identifying fungal structures in foxing (at least without the use of specific fluorescent stains) has been questioned owing to inconsistencies in fluorescence behavior reported in literature (Florian 2000), and the similar fluorescence characteristics of oxidizing cellulose (Missori, Righini, and Selci 2004; Boruvka 2008). The foxing stains on the tablecloth fluoresced in varying intensities and in colors ranging from white to yellow to orange. Some spots that could not be seen under visible light could be seen by their fluorescence under UV, whereas some foxing spots seen in visible light did not exhibit any fluorescence. Under 20–40x magnification, none of the areas that fluoresced were found to have any visible fungal structures. Pigmented fungal structures are reportedly visible under magnification as low as 20x (Florian and Purinton 1995).

The results of overall UV examination of the foxing were compared with ATP/AMP bioluminescence testing, using a Kikkoman Lumitester PD-30 and LuciPac Pens (fig. 4). The LuciPac Pen consists of a swab used to take a sample and a tube containing a luciferase enzyme. This enzyme is reactive to microbial activity and emits light when exposed to it. Once the sample is taken, the swab is inserted into the enzyme and shaken for 1 minute. Then the LuciPac Pen is inserted into the Lumitester, which reads the resulting luminescence of the enzyme and measures its intensity in relative light units (RLUs). The RLU value of a sample can be compared to the RLU value of another sample or set of samples to determine their comparative microbial activity. To assess the foxed tablecloth, 24 samples of foxed areas and 14 background samples of unfoxed areas were tested. A template with a 3 cm × 3 cm opening was used to standardize the size of each sample area being swabbed (fig. 5). The LuciPac Pen swabs were consistently rolled 10 times in the warp direction and 10 times in the weft direction, using a uniform pressure.

On average, ATP/AMP bioluminescence testing did find higher RLU readings in foxed areas versus unfoxed areas, indicating higher past or current microbiological activity. However, it is important to note the standard deviation of the testing: not all foxed areas were found to have high readings, and not all unfoxed
areas were found to have low readings (fig. 6), nor were the color and intensity of the foxing spots found to correlate with microbial activity levels. Rather, microbial activity could be more closely correlated to the location of the sample spot on the textile. Samples taken on what would have been exposed outer surfaces of the folded textile while it was stored over long periods showed the highest readings. Readings progressively decreased through what would have been the layers of the folded textile. A similar phenomenon has been observed in books, in which three-dimensional structures of foxing stains have been linked to the stacks of printed quire sheets piled before being bound into books (Lijferink, Porck, and Smit 1991). Similarly, when the foxed tablecloth was refolded in its original folding pattern, the foxing spots lined up through the stratigraphy of the folded textile. This discovery highlighted the likely role of storage conditions and localized moisture absorption or condensation in the development of the foxing.

Although ATP/AMP testing did find higher microbial levels in foxed regions of the textiles, these higher levels were not generally consistent with fluorescence under UV. Foxing spots that fluoresced orange under
UV—which, according to some authors is indicative of fungal activity—were not found to have higher RLU values than those areas that did not fluoresce. This does not mean that UV fluorescence is never a symptom of fungal activity, but it does demonstrate that fluorescence alone does not indicate an active fungal issue, as it is often related to other processes. Natalie Boruvka’s research published in 2008 demonstrated that the colors of UV fluorescence seen in foxing spots on paper are directly related to different stages of cellulose oxidation, specifically the progressive formation of conjugated double bond systems. The use of UV fluorescence to detect microbial activity in foxing stains is most aptly used during microscopic analysis using specific fluorescent stains that detect different fungal species under UV.

![Fig. 5. Swabbing a 3 cm × 3 cm foxing test with LuciPac Pen.](image)

![Fig. 6. Relative light unit (RLU) values (measuring relative microbial activity levels) by location and degree of foxing.](image)
4.2 ION CONTAMINATION ASSESSED BY XRF AND BATHOPHENANTHROLINE TESTING

To investigate the possibility of iron contaminants in the foxed tablecloth as a potential foxing catalyst, the textile was analyzed by both x-ray fluorescence spectroscopy and bathophenanthroline strip testing. Bathophenanthroline strip testing was performed first, following the procedure outlined by Vuori and Tse (2005). This test method is extremely useful in assessing foxing stains because of its ease of use and low cost. Furthermore, it tests specifically for iron ions (iron(II) ions that are not bound and are responsible for catalyzing cellulose oxidation) rather than overall iron content (which is what XRF detects). All eight of the bathophenanthroline tests performed across the textile tested negative for iron ions.

For comparison, XRF spectroscopy was performed on the case-study textile at CCI by conservation scientist Jason Anema (fig. 7). Readings were taken in 10 locations, covering 6 foxing spots and 4 spots that did not exhibit any foxing (fig. 8). Zinc, iron, calcium, and potassium were found to be present in the tablecloth in low

Fig. 7. XRF spectroscopy of foxed tablecloth, using an Artax spectrometer equipped with a rhodium target x-ray tube, using a 40-kV tube voltage, a 1-mA tube current, and a 120-second collection time.
levels, but levels in foxing spots were not found to be any higher than in unfoxed areas. This showed that the foxing was not directly related to iron content in this particular case. However, an iron peak was still present across all of the test locations, with count levels similar to some listed in recent foxing literature. Because XRF analysis is a semi-quantitative technique, providing only a general and relative idea of how much of an element is present, a known sample was required to put the detected iron peak into context. A laboratory-induced iron oxide stain on a different sample textile was analyzed for this purpose. Readings were taken of a spot of a color and color density similar to the foxing spots analyzed on the tablecloth. The iron signal of the sample iron oxide stain was roughly 100 times stronger than the iron signal of the foxing spots on the tablecloth. This put into perspective that although iron was found to be present on the foxed tablecloth, it was not present in quantities that would pose a similar risk to cellulose as do iron stains. Levels were so low in comparison that it is even possible that the iron found on the tablecloth was simply a component of surface soiling or dust.

The negative bathophenanthroline tests, in addition to the low levels of iron detected by XRF, led to the conclusion that iron was non-reactive, and therefore did not pose an active degradation issue for this textile. Stabilization methods, such as those involving chelators, were therefore not considered necessary for its eventual treatment. The use of chelators to reduce the aesthetic or visual appearance of the foxing spots was also ruled out, as the discoloration of the spots was attributed to cellulose oxidation rather than iron oxide staining.

5. CONCLUSIONS

The literature review and survey on foxing included in this project provided an essential framework for characterizing foxing on textiles. It offered insight into the magnitude of the foxing problem in textile collections, its potential causes, and its associated textile phenomena. The comparative case-study assessment of different
characterization methods found that UV fluorescence and XRF spectroscopy are less suitable for identifying active degradation associated with foxing than are ATP/AMP bioluminescence testing and bathophenanthroline testing. While low levels of iron were detected by XRF, the risk posed by reactive iron ions was ruled out through bathophenanthroline testing. UV fluorescence was found not to directly correlate with microbial activity levels measured by ATP/AMP testing. Furthermore, it remains unclear whether the slightly higher microbial levels detected in foxed areas was a cause of the foxing, or an effect of the higher pH, soiling, and moisture content of the oxidized cellulose in the foxed regions, providing more viable conditions for microbial life. The microbial activity levels found on the foxed tablecloth were not deemed to be at levels dangerous to the textile, and could be addressed by traditional, low-intervention methods such as high-efficiency particle air (HEPA)–filter vacuuming, maintaining a stable display environment, and careful consideration of storage materials and conditions.

Perhaps the most useful features of the two recommended characterization methods outlined in this article are their relative low cost and accessibility and their ability to identify the degradative agents that pose active risks to textiles. This, supported by a research framework considering foxing within a textile context, allowed for a clear and informed assessment of the foxed tablecloth that resulted in a straightforward and risk-based decision-making process for the treatment of the foxed textile.

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REFERENCES


THE CHARACTERIZATION OF FOXING ON TEXTILES


FURTHER READING


**SOURCES OF MATERIALS**

Kikkoman Lumitester PD-30 and Kikkoman LuciPac Pen Swabs
Luminultra Microbial Monitoring
520 King Street
Fredericton, New Brunswick E3B 6G3
Tel: (506) 459-8777
[www.luminultra.com](http://www.luminultra.com)

Bathophenanthroline iron(II) test paper (Iron Gall Ink Test Paper)
University Products Inc.
517 Main Street
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