Abstract

Bathophenanthroline iron test strips were used to test a variety of textiles that contained iron in the form of mordants, pigments and stains. The strips were found to be an effective means of identifying the presence of iron in cellulosic textiles. They can also be used to detect iron in acidic collagen and silk fibres. In many cases they were unable to detect iron in keratin fibres. The strips have potential for detecting iron in brown stains of unknown origins. Results from the test strips were compared with those from instrumental analyses.

Keywords

textiles, bathophenanthroline, iron, rust stain, Prussian blue

A preliminary study of the use of bathophenanthroline iron test strips on textiles

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Introduction

Ferrous, Fe(II), ions catalyse oxidative degradation of organic materials such as paper via the 'Fenton Reaction' (Neevel and Mensch 1999, Neevel 2000, Kolar et al. 2003). Degradation is often manifested as charring, brittleness and eventual loss of the substrate. Because of the extensive problem of iron-gall ink on paper and parchments, a great deal of research has been invested in studying the causes of deterioration and treatment options (see the web-sites Transition Metals in Paper (http://www.miponline.org), InkCor (http://www.infosrvr.nuk.uni-lj.si/jana/InkCor/index.htm) and Ink Corrosion (http://www.knaw. nl/ecpa/ink)).

Damage to textiles due to iron-catalysed oxidation is also well known. Examples include the fracturing of black areas in some printed textiles and the brittleness of black-dyed ethnographic textile fibres such as New Zealand flax (Hofenk de Graaff 2004, Daniels 1999). Substrates and dyeing methods vary but the damage in all has been attributed to the use of iron salts in combination with tannin-containing materials (iron-gall). It has been observed that some textiles dyed with iron-gall are in sound condition whereas others are not (Hofenk de Graaff 2002, 2004). Unless a textile is already exhibiting weak, brittle dark fibres, it is difficult to know if it is at risk of iron-catalysed oxidation. Simple, non-destructive, diagnostic tools that indicate the presence of free Fe(II) ions, will help identify those textiles that are potentially most at risk of iron-catalysed oxidation.

Iron can also be present in textiles due to accidental staining. Knowing whether a brown stain on a textile contains iron will have a direct bearing on treatment decisions. For instance, the presence of even small quantities of iron may preclude the use of oxidative bleaches or require the use of chelating agents in the bleach bath.

A paper test strip for identifying the presence of Fe(II) ions in iron-gall ink in paper and parchments was developed by Han Neevel and Birgit Reissland at the Institute for Cultural Heritage (ICN) in The Netherlands and is commercially available (Neevel and Reissland 2004). The paper strip incorporates bathophenanthroline (4,7-diphenyl-1,10-phenanthroline), a highly selective indicator for iron. In the pH range 2–9, bathophenanthroline reacts with Fe(II) ions to form a magenta-coloured iron complex that has very low water solubility. The test strips are thus essentially non-bleeding and can be used directly on the artifact. As long as minimal moisture is used there is little risk of the magenta colour being transferred to the artifact. Fe(III) ions can also be detected by the addition of ascorbic acid, a reducing agent, onto the test strip to Fe(II) ions which then react with the bathophenanthroline causing an enhancement of the magenta colour.

The idea of using the bathophenanthroline test strips on textiles, particularly for identifying iron-gall inks and dyes on cellulosic textiles, was suggested by Barker (2002). Indeed, some conservation laboratories use the bathophenanthroline test strips as a standard test in pre-treatment documentation of textiles exhibiting signs of iron catalysed degradation.¹ However, several questions remain about their effectiveness in a wider range of applications. For example:

- Are the test strip results from textiles consistent with those from instrumental analyses?
- Could the strips be used to detect iron on non-cellulosic textiles?
- · Could they detect iron mordants in dyes or iron pigments?
- Could they help distinguish iron-containing stains from other brown stains?

This study was undertaken to examine the effectiveness and limitations of the test strips in these applications.

Experimental procedures

Samples

Yarns and fabrics used in the study came from a variety of sources. The results from selected samples are reported here. These included textiles from the Canadian Conservation Institute (CCI) Textile Laboratory collection that exhibited brown stains of unknown origin (samples 1–3, 12; Table 1), textiles suspected of containing an iron mordant (samples 6, 7, 8, 11, 13, 16), new fabrics deliberately stained with iron (samples 5, 10, 15, 17, 19), yarns custom dyed with an iron mordant (samples 4, 9, 14, 20, 21), artifacts in the laboratory for treatment and analysis (samples 18 and 22), and artifacts tested by other researchers (samples 11 and 23).

Table 1. Bathophenanthroline Fe(II) test, pH and SEM/EDS results for textile samples

| No | Description | Fe(II) ^a | Fe(III) ^a | Iron (SEM/EDS) | рН (pH strips) | рН (extracted) |
|-------|---|---------------------|----------------------|------------------------|-------------------|-------------------|
| Cellu | Ilosic fibres | | | | | |
| 1 | Cotton, embroidery, dark orange brown stain, 20th century | negative | negative | ND | 4.7–5.0 | 4.54 |
| 2 | Cotton, pillow case, orange brown stains, 20th century (Figure 5, right) | negative | negative | ND | 5.3 | 5.18 |
| 3 | Cotton, table cloth, foxing spots, 20th century | negative | negative | ND | 3.5 | 3.98 |
| 4 | Cotton, dyed with madder and iron mordant, rinsed, 2004 | negative | 50 | Trace | 5.8–6.I | 6.74 |
| 5 | Cotton, flannel, iron stained, 20th century | 25 | 25 | Minor | 5 | 5.98 |
| 6 | Cotton, mud cloth, good condition, 20th century | 10 | 25+ | Trace | 4.2 | 4.49 |
| 7 | Cotton, black print, fragmenting, 19th century (Figure 1) | 25–50 | 50+ | Trace | 3.9 | 4.27 |
| 8 | Cotton, printed floral pattern, good condition, 19th century (Figure 3) | 25+ | 50+ | Trace | _ | 4.59 |
| 9 | Cotton, dyed with madder and iron mordant, unrinsed, 2004 | 25–50 | 25-50+ | Trace | 4.7 | 4.95 |
| 10 | Linen, prepared stain, rusty nails, 2003 | negative | negative | Major | 5.3 | 5.27 |
| 11 | Linen, homespun, brown dyed, 19th century (Figure 4) | <١ | I–10 | Minor | 5 | 5.91 |
| 12 | Linen, dark orange brown stain, 20th century (Figure 5, left) | 25–50 | 50 | Trace | 4.5–5 | 5.26 |
| 13 | New Zealand flax, black dyed, fragmenting, unknown date | 25–50+ | 25–50+ | Trace | 3 | 3.65 |
| Prot | ein fibres | | | | | |
| 14 | Silk, dyed with madder and twice amount of iron mordant, rinsed, 2004 | negative | negative | Trace | 5.3–5.5 | 5.01 |
| 15 | Silk, prepared stain, rusty nails, 2003 | negative | negative | _ | 5.3 | 5.38 |
| 16 | Silk, black-dyed yarn, 19th century | ĬO | 10–25 | Minor | 3.9 | 3.87 |
| 17 | Silk, prepared iron stain, 2001 (Figure 6) | 1–10 | 10+ | Trace | 3.9-4.7 | 5.11 |
| 18 | Painted silk banner with Union Jack, circa 1812 | | | | | |
| | Undyed silk | negative | negative | | 3.8 | 3.9 |
| | Red dyed silk: Cochineal (water soluble dye) | inconclusive | | Trace | 3.5 | 3.9 |
| | Blue dyed silk: Prussian blue | 10-25 | 25–50 | Major | 4.3 | 4.5 |
| | , Dark blue paint: Prussian blue + Indigo (Figure 7) | 1–10 | 10-25 | Major | _ | |
| 19 | Wool, prepared stain, rusty nails, 2003 | negative | negative | Minor | 5.3 | 5.83 |
| 20 | Wool, dyed with madder and twice amount of iron mordant, rinsed, 2004 | negative | negative | Trace | 5.3-5.5 | 5.03 |
| 21 | Wool, dyed with black walnut, madder, cochineal, American saffron or tansy; | negative | negative | | 4.2-4.5 | _ |
| | with iron mordant. 1980s | 0 | | | | |
| 22 | Opera cape, black-dyed fur-skin trim, circa 1870 | | | | | |
| | Black-dyed skin (collagen): 6000 ppm of copper ^b | 1–10 | 10-25 | 20,000ppm ^b | Trace | 3.7 (s) |
| | Black-dyed hair | negative | negative | ···· | | 3.9 (ex) |
| 23 | Black alpaca fibres (keratin) from a Nasca textile, 300–500 AD | 10-25 | 25+ | _ | _ | |

a, Fe(II) and Fe(III) values are according to the colour code developed at CCI

b, Quantitative results of black dyed skin were obtained by atomic absorption spectrophotometry

Iron detection with bathophenanthroline test strips

The samples were all tested using the commercial iron test strips. The test for Fe(II) ions was carried out by adding a drop of deionized water to the test strip, blotting it, covering it with Mylar, and pressing it against the textile for 2–3 min. Magenta colour on the dried test strip indicated a positive result (Figure 1). The colour intensifies upon drying. Although the magenta coloured iron complex has

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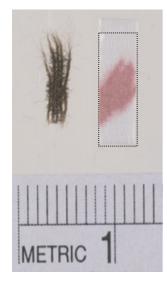


Figure 1. Positive iron test (outlined on strip) from severely deteriorated printed cotton textile fragment. Scale is in centimetres

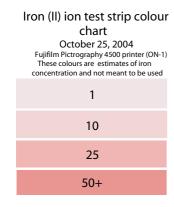


Figure 2. Colour code developed at the CCI for recording iron test strip results



Figure 3. Positive iron test from printed cotton with no symptoms of damage. The shape of the dark brown printed area is mimicked in the magenta on the test strip (sample 8)

low water solubility, excess moisture can cause migration of the colour onto the textile, especially if the test area has a low pH. It is very important, therefore, to use minimal moisture for the test.

To test for the presence of Fe(III) ions, a drop of 1 per cent (w/v) aqueous solution of ascorbic acid was added to the test strip after it was removed from contact with the textile. The test strip used for the Fe(II) ion test was cut in half before adding the ascorbic acid to one half. Thus results for both tests could be compared and retained.

Colour code

To facilitate recording the intensity of the magenta colour in positive tests, a colour code was developed at the CCI similar to those used with non-bleeding pH strips (Figure 2). Test strips were immersed into aqueous ferrous sulphate (FeSO₄.7H₂O) solutions, with 1, 5, 10, 25, 50, 100 and 1000 parts per million (ppm) of Fe(II) ions and the resulting colour noted. Because the colour intensity did not change above 50 ppm Fe(II) ions, the range adopted for the colour code was 1, 10 (mildly positive), 25, 50+ (very positive). The colour on the strips was visually matched to the printout of colour strips created in Microsoft Word and printed with Fujifilm Pictography 4500 printer (set at ON-1) onto photographic papers. The resulting colour code is not intended to be used quantitatively. It permits recording the colour intensity and allows comparison of results done by others.

pH measurement

Non-bleeding pH indicator strips (colorpHast, narrow range pH 2.5–4.5 and 4.0–7.0; manufactured by Merck) were used to estimate the surface pH of textiles tested in the field. The pH of those textiles from which fibre could be removed was also determined by micro extraction (Vuori and Tse 2004). Samples weighing either 0.001 g or 0.004 g were extracted using 0.1 M NaCl in a 50:1 (v/w) ratio, for 2–72 h. The pH of the extract was measured using pH strips and an IQ portable pH meter with micro probe. The pH of a sample of black-dyed fur (sample 22) was determined by surface measurement and by cold extraction (Binnie 1995).

Analysis

Most of the samples were analysed for total iron (Fe(II) and Fe(III)) using scanning electron microscopy–energy dispersive X-ray microanalysis (SEM–EDS). The detection limit is 0.1 per cent by weight. The samples were carbon coated, and iron concentrations were reported as major (greater than 10 per cent by weight), minor (1–10 per cent), trace (less than 1 per cent) or ND (not detected). Atomic absorption spectrophotometry (AAS) was previously done on the black-dyed skin (sample 22). Dyes and pigments from two objects (samples 18 and 22) were previously analysed by SEM–EDS, Fourier transformed infrared spectroscopy (FTIR), microchemical tests and high-performance liquid chromatography photodiode array detection (HPLC–PDA) (Moffatt 1996, Moffatt and Corbeil 1995, Sirois 1996).

Results

Results from Fe(II) test strips, SEM/EDS, and pH measurements of selected samples are summarized in Table 1.

Cellulosic textiles

Very positive results were obtained using the iron test strips on very small fragments from a number of severely deteriorated printed cotton textiles. A representative sample is shown in Figure 1 (sample 7). However, very positive results were also obtained from other printed cottons displaying no signs of damage. An example is shown in Figure 3 (sample 8).



Figure 4. Brown-dyed homespun linen (circa 19th century), with weak and missing brown-dyed yarns (sample 11)

Test strip results from a series of linen samples were provided by private researchers studying homespun, hand woven linens from eastern Pennsylvania, dating from 1750–1850 (Hoag 2003, 2004). The test strips were used on brown-dyed linen, some of which exhibited damage that is consistent with the presence of iron (Figure 4). Not all samples tested positive, but those that did produced results very similar to sample 11. Analysis of this sample by SEM–EDS confirmed the presence of iron.

The very positive test strip result from a sample of black-dyed New Zealand flax (*Phormium tenax*) (sample 13) was confirmed by SEM–EDS.

Brown stains on cellulosic textiles

Three cellulosic textiles with brown spots resembling rust stains, and one exhibiting 'foxing' were tested (samples 1–3 and 12; Figure 5). In each case, the negative iron test strip results correlated with analysis by SEM/EDS. While more testing is needed to confirm this, consistent correlation with instrumental analyses indicates the potential usefulness of these test strips for this application.

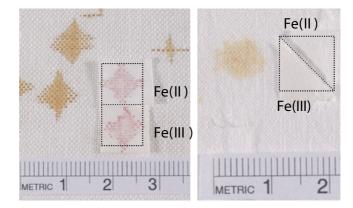


Figure 5. Test strip results from two brown-stained textiles. Left, positive Fe(II) ion and Fe(III) ion results on linen (sample 12); right, negative results on cotton (sample 2)

Protein fibres

The black skin portion of a fur trim from an opera cape, dating from 1870 (sample 22) gave a slightly positive result for Fe(II) ions, and a very strongly positive result for Fe(III) ions. Results from SEM/EDS and AAS confirmed that the skin (collagen) contained high levels of iron, 20,000 ppm. Positive test strip results were also obtained from iron stained silk (Figure 6; sample 17) and a black-dyed silk artifact (sample 16) in which iron was detected by SEM/EDS. Longer contact time is required for testing silk.

In contrast, keratin samples gave predominantly negative results. The blackdyed hair (keratin) from the fur trim (sample 22) tested negative for both Fe(II) ions and Fe(III) ions with the test strips despite the fact that SEM–EDS confirmed the presence of iron. Other samples of wool (keratin) dyed with natural dyes and an iron mordant (samples 20 and 21) gave negative test strip results. However, one test result, submitted by another researcher, proved the exception. A positive result for both Fe(II) ions and Fe(III) ions was obtained from black alpaca fibre (keratin) from a Nasca textile dating from 300 to 500 AD (sample 23). The black fibre was described as weak and brittle.

Dyes and pigments

Dyes and pigments from a painted and pieced silk Union Jack banner, dating from 1812, were tested with the bathophenanthroline strips. The results were compared to those previously obtained by instrumental analysis (sample 18). The test strip results for the undyed silk were negative for both Fe(II) ions and Fe(III) ions. The red dyed silk was slightly soluble in water and so the test strip results for this sample were inconclusive. However, dramatically positive results were consistently obtained on samples of the blue dyed silk. The blue painted silk gave a slightly



Figure 6. Positive iron test with iron stained silk (sample 17)

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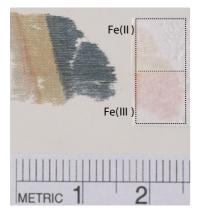


Figure 7. Positive Fe(II) and Fe(III)ion tests with Prussian blue painted silk (sample 18)

ambiguous result for Fe(II) ions (the colour on the strip was an orangey/pink) but gave a positive result for Fe(III) ions (Figure 7). The surface pH of all three colours of silk in the Union Jack (undyed, red and blue) was acidic.

SEM-EDS found high levels of iron in both the blue dyed silk and blue paint; trace amounts of iron were found in the red dyed silk. The blue dye was identified as Prussian blue (ferric ferrocyanide). FTIR indicated the blue paint was a mixture of Prussian blue and indigo in a collagen-type protein binder such as animal glue. The red dye was identified by HPLC-PDA as cochineal with a trace amount of iron (Moffat and Corbeil 1995).

Negative results from iron containing textiles

Negative results with the test strips were obtained from samples of new linen and silk that had been stained with rusty iron nails in 2003 (samples 10 and 15). SEM–EDS analysis of the rust-stained linen confirmed the presence of iron in the stains.

Discussion

The reliability of the bathophenanthroline test strips to indicate the presence of iron in textiles depends on the presence and the availability of the water soluble Fe(II) or Fe(III) ions in the fibres. Of the 23 samples that were tested with the test strips, 17 agreed with SEM/EDS results.

The test strips were found to be reliable in identifying iron in cellulosic and silk fibres. The exceptions were freshly prepared rust stains on linen and silk that gave negative results with the test strips (samples 10 and 15). A possible explanation is that the iron exists as Fe(III) hydroxide oxides, FeO(OH), which have low solubility in water making them undetectable with the test strips. The pH of the test areas may have some influence on formation and stability of these Fe(III) hydroxide oxides. Acidic pH increases their solubility (Cornell and Schwertmann 1996), making the iron more detectable with the test strips.

Except for one sample, the test strips consistently tested negative with ironcontaining wool. A reasonable explanation is that the iron ions are tightly bound to the numerous sulphydryl groups (SH) in keratin, and are therefore unavailable to react with the bathophenanthroline. The anomalous positive test strip result with aged (about 300–500 AD) keratin fibres, sample 23, can be a result of iron contamination from the surroundings. However, it can also be caused by the release of bound iron by the severely deteriorated fibres. Although more testing is required, results to date indicate that the test strips are not reliable for detecting the presence of iron ions in keratins.

The positive test strip results from samples containing Prussian blue (sample 18) are unexpected because iron ions bound in a pigment like Prussian blue are not water soluble and therefore should not be detected with the test strips (Neevel 2004). This was confirmed when Prussian blue powder pigment in deionized water and 1 per cent ascorbic acid were tested: neither sample produced a positive result with the test strip. The positive results we observed may be due to the age of the Prussian blue sample. As some ferric ferrocyanate complexes deteriorate and break down, iron ions can become available giving a positive test with bathophenanthroline. Further tests with new and artificially aged Prussian blue containing textiles are needed before reaching any conclusion.

Determining the pH of the test area can provide valuable information about the condition of the textile. Acidic pH indicates that the textile, especially if it is cellulosic, is at risk of acid catalysed hydrolysis, and that remediation is needed. When pH of the substrate is below 6, water-soluble Fe(II) ions can remain stable for extended periods (Selwyn 2004), increasing the potential to catalyse oxidation. With acidic pH, there is also a danger of the magenta indicator complex bleeding back onto the textile. In this case, a barely dampened test strip helps minimize the risk (Barker 2004).

The iron test strips are extremely easy to use even within the confines of a museum storage area. In many cases it is possible to conduct the tests without removing the textile from its storage container.



Figure 8. Transfer of water-soluble red dye onto test paper – scace marked in mm

The use of iron test strips may be problematic for extremely brittle textiles or those prone to forming tidelines or containing water sensitive dyes and finishes. The tests should be done in an unobtrusive area, or, if possible, on a loose yarn or detached fragment. Water-soluble red dyes or inks can be mistaken for a positive result on the test strip (Figure 8). To eliminate this possibility, fugitive dyes can be rinsed out of the test strip with deionized water, leaving the coloured bathophenanthroline/iron complex, which has very low water solubility.

Maintaining a clean work area is essential, as iron is a common component of dust. To identify and eliminate common sources of iron contamination, controls are needed. These include testing deionized water, ascorbic acid, ambient dust on the surfaces of storage shelves, blotting papers and Mylar pieces with the Fe(II) test strips prior to testing on samples.

Conclusions

The bathophenanthroline iron test strip is an extremely useful addition to the textile conservator's toolkit. It provides an easy way of identifying and prioritizing those textiles that are most at risk of iron-catalysed degradation. Confirmation of the presence and concentration of iron can be verified only with instrumental analyses.

The test strips were found to be effective for identifying the presence of Fe(II) ions and Fe(III) ions in most cellulosic textiles. They can detect iron in protein fibres such as silk and collagen, but require slightly longer contact time than cellulosic fibres. They are not reliable for detecting iron in keratin fibres such as wool and hair. The strips detected iron ions in an aged sample of Prussian blue (ferric ferrocyanide) dyed and painted silk.

A negative result with the test strip, in the absence of ascorbic acid, can indicate the absence of iron or it can mean that no water soluble Fe(II) ions are available. A positive result, either high (25-50+) or low (1-10) on the colour chart, indicates the presence of free Fe(II) ions, which means that the textile is at risk of iron-catalysed degradation.

A very positive result (25–50+), with low pH, indicates a higher degree of risk. Such textiles should have high priority for remediation. In practical terms this means that any form of humidification treatment should be avoided to prevent spreading of Fe(II) ions within the textile. However, if the textile can be safely wet cleaned, washing can remove soluble Fe(II) ions and acids, which will benefit the textile. High-risk textiles that cannot undergo immediate remediation should be kept in a low temperature and low RH environment to delay degradation.

A mildly positive result (1-10) on a non-acidic textile indicates that the textile contains very little iron or that the iron is present in a more stable form. This means that the textile is at lower risk. If the textile is acidic, the removal of acids and ongoing monitoring would be recommended.

Further study is required to confirm the test strip's reliability to detect iron in keratin fibres and in iron-containing pigments such as Prussian blue.

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Notes

1 The Textile Conservation Workshop in South Salem, New York, uses the test strips in pre-treatment documentation on textiles exhibiting signs of iron-catalysed degradation.

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Materials

ColorpHast indicator strips (pH 4.0–7.0 and 2.5–4.5) EM Science 480 Democrat Road Gibbstown NJ 08027, USA

Indicator Paper for Iron Ions Product Code 539-3000 Preservation Equipment Vinces Road Norfolk IP22 4HQ United Kingdom Tel.: +44 (0)1379 647400 Fax: +44 (0)1379 650582 Web site: www.preservationequipment.com