6. THE DYE ANALYSIS: PART 2

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6.1 Summary

A further seven woollen textiles recovered from Siller Holes were analysed to identify dyes. High-performance liquid chromatography with photodiode array detection revealed detectable dyes in four of the seven samples. In two cases, components related to natural dyestuffs (indigotin, purpurin and an unidentified anthraquinone) were identified, while two other cases were most probably synthetic dyes. No dyes were detected in the remaining three samples.

6.2 Introduction

Striped coarse wool fragments were recovered from the lead mining site at Siller Holes, West Linton. Eleven of these wool textiles had already been analysed for dyes by photodiode array high-performance liquid chromatography (see Part 1). A further set of seven fragment samples, mostly black, were subsequently submitted for dye analysis and their results are reported here.

6.3 Experimental

6.3.1 Sample descriptions

Fibres were viewed under a light microscope at × 80 magnification to describe the colours given in Table 5.

6.3.2 High-performance liquid chromatography with photodiode array detection

Samples were prepared and analysed as described in Appendix 1. Full results can be consulted. They will be held with the original data, currently at the National Museums of Scotland Collections Centre.

The acidified methanol extraction solution for Sample 15 turned from clear to bright red then muddy red-brown almost instantly upon heating (the fibre remained black). This rapid and distinctive extract colour change has not been observed with

Table 5 Sample descriptions

Sample no.	Find no.	Description	Colour × 80 magn	Sample weight
1	SH 77	s-spun yarn	blue-green	3.1mg
2	SH 118	warp	dark green-brown	2.1mg
3	SH 118 (1)	weft	dark green-brown	4.9mg
4	SH 118 (2)	weft	brown	1.9mg
5	SH 118 (3)	weft	dark green-brown	3.0mg
6	SH 121.5	weft?	red-brown	1.7mg
7	SH 134	weft	mid-brown	1.7mg
8	SH 139	weft?	dark green-brown	2.3mg
9	SH 234 (1)	weft?	red-brown	1.8mg
10	SH 234 (2)	weft?	black	3.3mg
11	SH 256	weft?	mid-brown	0.5mg
12	SH 99.1	z-spun yarn	black	2.6mg
13	SH 107.3	z-spun yarn	black	2.6mg
14	SH 405	s-spun yarn (probably weft)	green-brown	1.4mg
15	SH 412	s-spun yarn	black	3.0mg
16	SH 452.2	s-spun yarn	brown	4.3mg
17	SH 452.1	z-spun yarn	black	4.0mg
18	SH 663.1	s-spun yarn	light brown	1.3mg

any natural dyestuff samples or references so far analysed in the NMS laboratories.

When water was added to the extracts from Samples 16 and 17 after first reconstituting them with methanol, the solutions turned from golden yellow to black with small black particles. The black solution was not produced with methanol alone, indicating that something in these extracts was water-insoluble. This again had not been observed with other natural dye extracts.

6.4 Results

Component retention times and spectra were compared to those for reference dyes. Table 6 summarises the data.

6.5 Discussion

6.5.1 Samples 12 and 13

The areas of textiles analysed as Samples 12 and 13 did not appear to be dyed or, if they had been originally, dye components were no longer detectable. It is possible that the dark colour is natural pigment in the wool.

6.5.2 Sample 14

Indigo (natural or synthetic) and woad are the expected sources for indigotin in Sample 14. Synthetic indigo was commercially available in the 1890s, but it is not possible to analytically differentiate between synthetic and natural indigo.

6.5.3 Sample 18

Cultivated madder and similar dye plants contain two major anthraquinone compounds, purpurin and alizarin. Wild madder, however, contains almost exclusively purpurin with only a trace amount of alizarin, a distinguishing feature between it and related dyestuffs (Schweppe 1989; Cardon 1990: 37–41). Only purpurin was detected in Sample 18, suggesting that wild madder (*Rubia peregrina*) rather than cultivated madder (*Rubia tinctoria L.*) or related *Galium* plants was possibly used. Whether alizarin would degrade in preference to purpurin on archaeological textiles is doubtful as the compounds are so chemically similar, but no extensive research on buried textiles has been done to test this supposition.

6.5.4 Samples 15, 16 and 17

The analytical results for Samples 15, 16 and 17, coupled with their unusual behaviour during sample preparation, suggested synthetic dyes. Conclusive proof through their identification with known references is needed, but little analytical information or published data on synthetic dyes applicable to historical textiles exist at present to enable this. Until further information comes to light, it is proposed that synthetic dyestuffs are present in the sampled areas of these textile fragments.

6.6 Conclusions

The continued study of textile fragments from Siller Holes has revealed that two further samples analysed contain traces of natural dyes, and another two are likely to have been dyed with synthetic dyestuffs.

The natural dyestuff sources can only be speculated upon because additional characteristic compounds were not detected, but the suggested sources were widely used for many centuries in Europe.

Two more samples have been submitted for analysis (SH 97.1.2 and SH 412), and it is intended that selected parts of SH 118, SH 121.5 and SH 234 will be re-analysed to try to identify the dye sources.

A summary of results for all the Siller Holes textiles analysed for dyes is given in Table 7.

Table 6 Summary of data

Sample no.	Find no.	Colour	Identified components	Possible sources
1	SH 77	Blue-green	indigotin	woad (*Isatis tinctoria*) or indigo (*Indigofera tinctoria L.*)
2	SH 118	Dark green-brown	indigotin	woad/indigo
3	SH 118 (1)	Dark green-brown	indigotin	woad/indigo
4	SH 118 (2)	Brown	luteolin	flavonoid-containing dye plant, possibly weld (*Reseda luteola L.*)
5	SH 118 (3)	Green-brown	(i) indigotin; (ii) carminic acid	(i) woad/indigo, (ii) anthraquinoid dye insect
6	SH 121.5	Red-brown	no dyes detected	
7	SH 134	Brown	no dyes detected	
8	SH 139	Green-brown	(i) indigotin; (ii) possible flavonoid	(i) woad/indigo, (ii) possible flavonoid-containing dye plant
9	SH 234 (1)	Red-brown	carminic acid	anthraquinoid dye insect
10	SH 234 (2)	Black	(i) indigotin; (ii) carminic acid-like anthraquinone	(i) woad/indigo, (ii) possible anthraquinoid dye insect
11	SH 256	Brown	(i) anthraquinone	anthraquinoid dye plant
12	SH 99.1	Black	no detectable dye components	
13	SH 197.3	Black	no detectable dye components	
14	SH 405	Green-brown	Indigotin	woad (*Isatis tinctoria*) or indigo (*Indigofera tinctoria L.*)
15	SH 412	Black	>10 unidentified chromophoric components	probably synthetic
16	SH 452.2	Brown	4 unidentified chromophoric components	probably synthetic
17	SH 452.1	Black	1 unidentified chromophoric component	probably synthetic
18	SH 663.1	Light brown	anthraquinone; purpurin	red from plant source, possibly wild madder (*Rubia peregrina*)

Table 7 Summary of dye analysis results

*Blue. Indigo could be natural or synthetic.

Sample no.	Find no.	Description	Possible dye source
1	SH 77	blue-green, s-spun	woad or indigo*
2	SH 118	dark green-brown, warp	woad/indigo*
3	SH 118 (1)	dark green-brown, weft	woad/indigo*
4	SH 118 (2)	brown, weft	plant yellow
5	SH 118 (3)	dark green-brown, weft	woad/indigo + insect red
6	SH 121.5	red-brown, weft?	no dye detected
7	SH 134	mid-brown, weft	no dye detected
8	SH 139	dark green-brown, weft?	woad/indigo* + plant yellow
9	SH 234 (1)	red-brown, weft?	insect red
10	SH 234 (2)	black, weft?	woad/indigo* + insect red
11	SH 256	mid-brown, weft?	plant red
12	SH 99.1	black, z-spun	no dye detected
13	SH 107.3	black, z-spun	no dye detected
14	SH 405	green-brown, weft?	woad or indigo*
15	SH 412	black, s-spun	probably synthetic dye
16	SH 452.2	brown, s-spun	probably synthetic dye
17	SH 452.1	black, z-spun	probably synthetic dye
18	SH 663.1	light brown, s-spun	plant red, possibly wild madder