

Taking historical chemistry to the bench: A new perspective for modern chemists through the re-creation and analysis of 19th-century Scottish Turkey red dyed textiles

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The art of textile dyeing depends entirely on the chemistry of the process, from the type of fibre to the solvent effects of each ingredient in the dye bath. As the field of modern chemistry developed during the 18th century, a significant amount of research was conducted through experiments in textile dyeing or for the purpose of improving various dyeing practices. In their textile dyeing treatises, Claude-Louis Berthollet and Jean-Antoine-Claude Chaptal emphasise the connection between dyeing and chemistry¹, yet today it is often viewed as a craft or hobby rather than the practical applied chemistry. Significant chemical developments related to dyeing continued in the 19th century with the development of ‘synthetic’ dyes from coal tar by-products, and in the 20th century with fibre-reactive dyes. As progress fostered new innovations, old dyeing methods and techniques were abandoned and sometimes forgotten.

This is especially true for process called Turkey red, which was legendary for its lengthy and repetitive series of steps, taking at least a month to complete into the mid-1800s. Despite this inconvenience, the quality of the colour and its durability were unsurpassed. In the words of Dr Andrew Ure, a pioneering Scottish chemist, ‘this is the most complicated and tedious operation in the art of dyeing, but it produces the fastest colour which is known.’² Far from being immune to technical developments, Turkey red dyeing encouraged the development of Turkey red oil, the first anionic surfactant,³ and the quantity of ground madder roots required to satisfy the industry’s needs propelled efforts to synthetically produce alizarin, its primary colouring molecule. Dyeing Turkey red was a major industry in the West of Scotland from its establishment in 1785 and it continued until the mid-1930s, when the quality of single-application red dyes was finally enough to compete with Turkey red on the market and its production ceased.⁴ Although synthetic alizarin and Turkey red oil made it possible to dye Turkey red in a matter of

days rather than weeks, the steps to accomplish it and the unusual process remained largely unchanged.

Project overview

Throughout history, dyers and chemists endeavoured to understand the chemistry of this unique process,⁵ but classical or ‘wet bench’ analytical chemistry was unsuitable to characterise Turkey red. This is because Turkey red is not a dye, but a prescribed series of treatments to cotton fibres resulting in a fast, bright red shade that exists only on the fibre. In the nineteenth century, unscrupulous merchants sold inferior synthetic red dyes as ‘Turkey red’; these betrayed the deception by fading quickly.⁶ The quality of Turkey was such that in 1875, the American catalogue retailer Montgomery Ward sold linen ‘Table Damask’ at 50 cents per yard, ‘Turkey Red Table Damask’ 80 cents per yard, and ‘Imported Turkey Red Table Damask, fast colours’ for 95 cents per yard.⁷

Nevertheless, interest in the process waned after production ended, well before modern analytical instruments became available. Research at the University of Glasgow takes a fresh look at the chemistry of this distinctive dyeing practice through a process-based approach by reviewing historical Turkey red processes and the contemporary research on the topic. For this project, 21 historical Turkey red methods in English and French published or documented between 1765 and 1893 were reviewed; material was published in German and other languages as well, though not accessed. Nine volumes of historical textile sample books from the United Turkey Red Collection in the Glasgow University Scottish Business Archive (SBA) (Glasgow, Scotland, UK) as well as Turkey red at the Victoria & Albert Museum (London, England, UK) were viewed and analysed. Documents relating to internal dyeing practices for J&P Coats in the Paisley Central Library (Paisley, Scotland, UK) and Archibald Orr Ewing and Co in the SBA show consistency between published dyeing texts and actual Turkey red production.

This cross-disciplinary project combines the historical material with modern analytical chemistry to dye pieces of Turkey red for analysis in order to understand the chemistry of the process. The aim of this research is to find non-invasive and microanalytical techniques to learn about Turkey red and aid in the preservation of historical Turkey red textiles for the appreciation of future generations. The project is part of the Lord Kelvin Adam Smith Scholarship scheme at the University of Glasgow, which promotes novel cross-disciplinary research, and works

between the Centre for Textile Conservation and Technical Art History (CTCTAH) and the School of Chemistry.

Turkey red dyeing in Europe

The name ‘Turkey red’ refers to both process and product, a series of steps that results in exceptionally bright red woven cotton fabric noted for its resistance to fading from light exposure and washing. Its exact origins are unknown, but generally attributed to India, which is reflected in one of its French names “*rouge des Indes*”.⁸ Each set of directions varies, but all follow the same series of treatments. First, cotton yarn or fabric is scoured and dried, then prepared with oil. It was then treated with a solution of aluminium, and finally dyed with alizarin (synthetic or from madder roots) and chalk or calciferous water.⁹ Similar techniques still exist in Indonesia, where nut and seed oil, *Symplocos* leaves (aluminium), and *Morinda citrifolia* (alizarin) are used to dye cellulose fibres, corroborating a southeast Asian origin for Turkey red.¹⁰ The practice was eventually established in the Levant using rancid olive oil, alum, and ground madder roots. The textiles were sold to Western Europe, where it was known as Turkey red for its place of production.



Fig. 1: Printed Turkey red sample c. 1860s. University of Glasgow Archive Services, Records of United Turkey Red Co Ltd, GB248 UGD13/8/5.

European dyers were desperate to manufacture it themselves but found it difficult to successfully replicate. The process was dependent on the climate and the available water supply, for if it did not contain calcium naturally then chalk must be added to the dye bath to achieve the colour.¹¹ The water supply must be abundant, because thorough washing was required after each step. It required space as well to spread the cotton to dry after each treatment. The process was time-consuming, with multiple repetitions of the oil treatment needed to ensure a quality product. Until technological improvements in the mid-nineteenth century, the process generally took around a month; these developments reduced it to around three days.¹² This was called the ‘new’ Turkey red process, in contrast with the longer ‘old’ process.

<i>Marche en jaune.</i>	<i>Translation</i>
Débouilli.	Scouring.
Bains de fiente.	Dung baths.
Bains blancs.	White baths.
Sels.	Salts.
Dégraissage.	Degreasing.
Engallage.	Galling.
Alunage.	Aluming.
Lavage d'alun.	Washing the alum.
Bains blancs.	White baths.
Sel.	Salt.
Dégraissage.	Degreasing.
Engallage.	Galling.
Alunage.	Aluming.
Lavage d'alun.	Washing the alum.
Garañage.	Maddering.
Avivage.	Brightening.
Rosage.	Clearing.

Fig. 2: Summary of the ‘old’ Turkey red process from *Cours élémentaire de teinture sur laine, soie, lin, chanvre et coton, et sur l’art d’imprimer les toiles* by Jean Baptiste Vitalis, 1823 (author’s translation).

In 1747, Greek dyers from Smyrna took their knowledge of the process to France and helped establish Turkey red dyeing at Darnétal and Aubenas.¹³ In 1753, a dyer named John Wilson in Ainsworth, England sent an agent to Turkey to learn the process, but was disappointed that it was not suitable for his business as piece dyer, or dyer of fabric.¹⁴ Supposedly at the time dyers could only make Turkey red on cotton yarn, which was then woven into fabric. The dyeing of woven fabric is generally credited to Daniel Koechlin of Mulhouse in 1810,¹⁵ but an 1803

reprint of the 1786 Journals of the House of Commons mentions dyeing Turkey red ‘both in the Hank and the Piece’, pre-dating Koechlin by 25 years.¹⁶ A comparison of historical methods from before and after this date reveals few differences to the modern reader, so the nature of his contribution is unclear.¹⁷ It may be the manner of handling the fabric, which is infrequently described—indeed, many dyeing ‘receipts’ are frustratingly vague to the modern reader. Whether Koechlin developed a new practice or independently developed one similar to the one mentioned in by the House of Commons remains unknown.

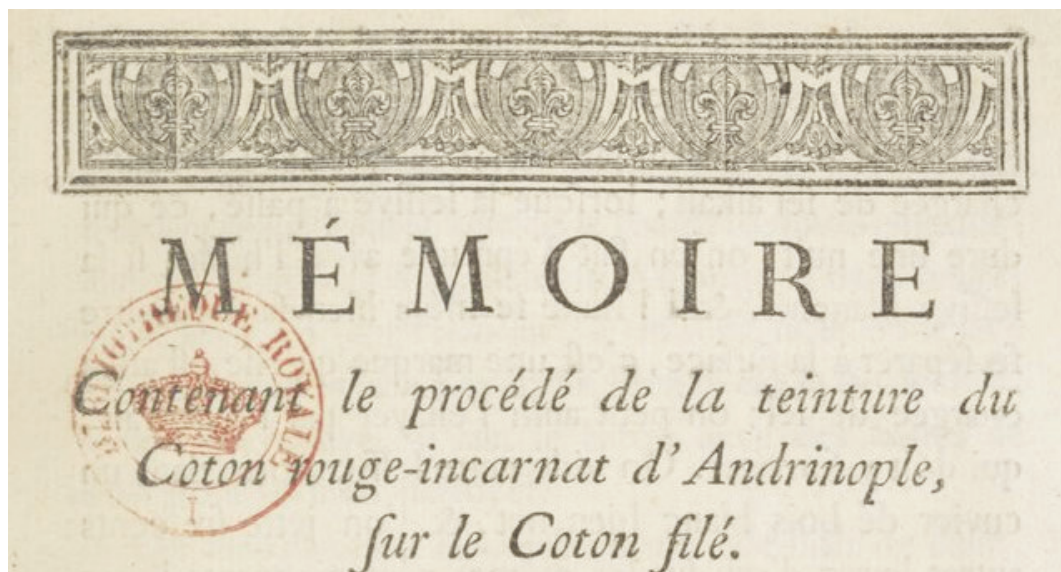


Fig. 3: Title page from *Mémoire Contenant le procédé de la teinture du Coton rouge-incarnat d'Andrinople, sur le Coton filé*, 1765. French government publication of Turkey red method.

The lack of detail raises the question of whether information was being omitted to protect knowledge of the process. Secrecy was always important in Turkey red dyeing, but in order to learn and improve the process dyers and chemists sought to understand its chemistry, an endeavour that would be improved by the sharing of knowledge.¹⁸ The desire for secrecy appears to have had little effect on the dissemination of printed directions. Publishing directions for Turkey red was evidently not sufficient to spread the practice, however. The first European publication was in 1765 by the French government, making the knowledge available to the public.¹⁹ This was not enough to transfer production to Britain and Parliament offered a prize of £2,500 (about £150,000 in 21st-century currency²⁰) for the successful demonstration of Turkey red dyeing. It was claimed by the French dyers Louis Borelle and Abraham Henry Borelle, who made their case in Manchester and received the award in 1786.²¹ Another Frenchman, Pierre Jacques Papillon,

tried to claim the prize after it had been awarded to the Borelles and was turned down. He met a Glasgow dyer and businessman named George Mackintosh who persuaded Papillon to come to Scotland to set up production with Mackintosh's partner, David Dale. The Turkey red works of Dalmarnock, located on the River Clyde near Glasgow, opened in 1785.²² Although it was first successfully dyed in England, Scotland was where Turkey red was first produced in Britain.

Turkey red dyeing in Scotland

The work of George Mackintosh changed the course of the textile industry in the West of Scotland. Born in 1739 in the parish of Rosskeen, near Inverness in the Scottish Highlands, he moved to Glasgow and worked as a clerk in a tannery and as head of a shoe factory. In 1777, he purchased land near the Cathedral in what is now the East End of Glasgow and established a works for dyeing with lichen, called cudbear or *orseille*, which he surrounded with a ten-foot wall to protect his interests. The operation consumed 250 tons of lichen annually, quickly exhausting the local supply and relying on expensive imports from Sweden and Norway. Cudbear only dyed wool and silk, however, making it useless for the growing printed cotton industry.²³

David Dale was another giant of Scottish industry whose connections to cotton made him an ideal partner for Mackintosh. He began his career as a herder and peddler of yarns and cloth, finally opening a yarn dealership on High Street in Glasgow in 1763. His business interests expanded, eventually leading to the foundation of New Lanark Mills with his son-in-law Robert Owen and the inventor Richard Arkwright in 1783.²⁴ Arkwright's contribution to the Turkey red is less direct but no less significant. The rigors of Turkey red dyeing were harsh on cotton yarn, but Arkwright's improved spinning machinery produced stronger, even yarn that could endure the process.²⁵

Mackintosh, Dale, and Papillon founded the Dalmarnock works for Turkey red dyeing in Glasgow in 1785, announcing they dyed hanks of yarn for three shillings per pound. Papillon had 'an unhappy temper' according to Mackintosh and parted ways by 1787, leaving his partners to continue producing Turkey red and improving the process. In 1790, Papillon received a fee from the Commissioners and Trustees for Manufactures in Scotland for his Turkey red method, which was disclosed to Dr James Black, Professor of Chemistry at the University of Edinburgh. It was kept secret for an agreed-upon number of years and then published in 1804.²⁶ Another account, published by Thomas Henry of Manchester, appeared

in the *Memoirs of the Literary and Philosophical Society of Manchester* in 1790.²⁷

This time, unlike with the French government publication of 1765, the industry expanded as knowledge of the process was disseminated. The First and Second Statistical Accounts of Scotland document the flourishing of the industry over the first half of the nineteenth century. The First Statistical Account was organised and compiled by Sir John Sinclair of Ulbster. It documents Scottish life in the 1790s through a collection of reports from parish ministers who describe the geography, industry, environment, society, and history of their communities. The Second Statistical Account, published in 1845, similarly documents Scottish life in the 1830s-40s.²⁸ Two Turkey red dye works are recorded in the First Statistical Account, Dalmarnock and the one founded by Papillon after his split from Dale and Mackintosh.²⁹ By the Second Statistical Account, Turkey red dyeing was documented at Blantyre, Old Kilpatrick, and Rutherglen, with many cotton printing works established west of Glasgow in the parish of Bonhill as well.³⁰ As the city of Glasgow grew, the banks of the River Leven in Bonhill parish became the primary seat of the Turkey red industry due to the abundance of space and water.

The second half of the nineteenth century was a heyday for the Scottish Turkey red industry.³¹ Increasing foreign competition and the growth of domestic textile production in India, one of the biggest markets for Scottish Turkey red, threatened the industry. In 1898 the largest firms, William Stirling and Sons, John Orr Ewing and Co., Archibald Orr Ewing and Co., and Alexander Reid and Sons amalgamated to form the United Turkey Red Company, Limited (UTR).³² The development of better azo and naphthol red dyes was a further blow to the industry and United Turkey red ceased production in 1936.³³

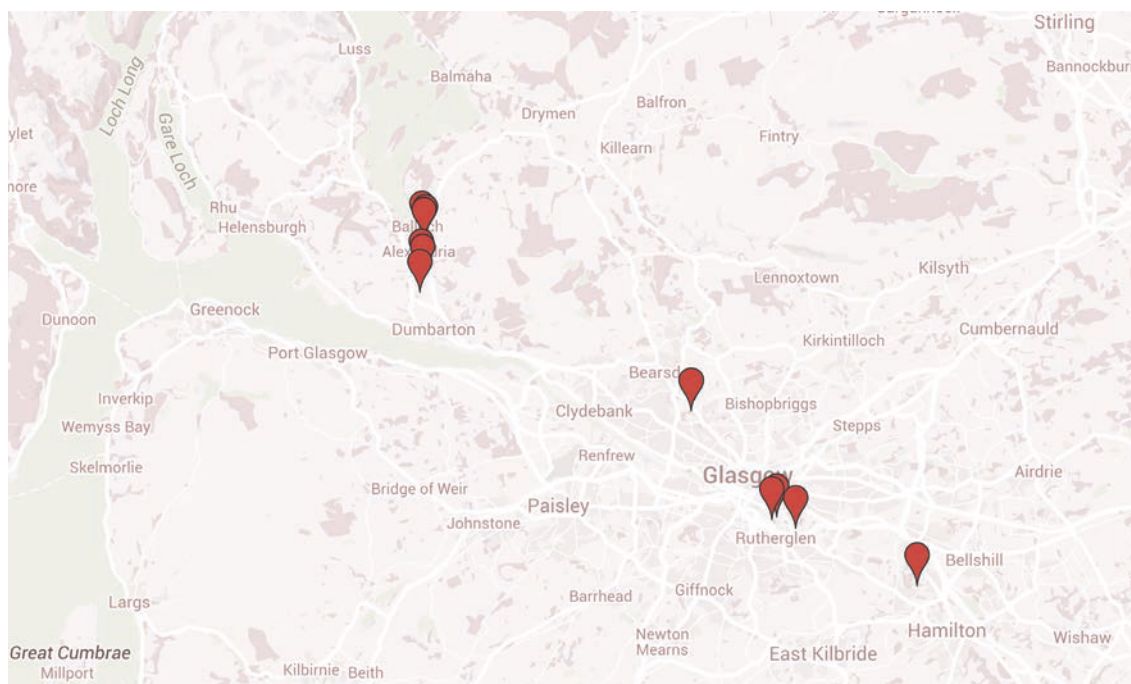


Fig. 4: Locations of some former Turkey red dye works in the West of Scotland. Vale of Leven sites centre left.

The old Turkey red process and chemistry

Turkey red is unlike any other known textile dyeing process in terms of its complexity and for its ability to impart such a vibrant, fast colour to cotton. In contrast with wool and silk, which are proteinaceous fibres, cellulosic fibres like cotton and linen are more crystalline, making them more resistant to dyeing. Historically, a Turkey red process could be described as having five ‘operations’: oiling, sumaching, aluming, dyeing, and clearing.³⁴ A modern definition from the mid-twentieth century is ‘a fiery red colour lake formed on vegetable fibre by combination of a fatty acid with calcium aluminium alizarinate’.³⁵ This recognises the essential ingredients in Turkey red, leaving out auxiliaries such as sumach. Textile dyeing is influenced by a variety of factors, and it was known that even an experienced Turkey red dyer could expect the occasional failed batch.³⁶

In the ‘old’ process, used until the 1870s, cotton was soaked in a mixture of rancid olive oil, weak sodium carbonate solution, and sheep or cow dung. After soaking it was wrung and dried; this treatment was often repeated up to eight times.³⁷ Various purposes were attributed to the dung, including the power of ‘animalisation’, or imparting the animal nature of the dung to the vegetable fi-

bres.³⁸ By the mid-1800s, ‘dung substitutes’ in the form of silicates, phosphates, and arsenates were used in dyeing and printing to fix the colour.³⁹ Modern research shows adult cattle and sheep excrete inorganic phosphorous in their dung in quantities that would have been useful to textile dyers as an auxiliary agent, giving it a purpose more concrete than ‘animalisation’.⁴⁰ The purpose of the oiling was to imbue the cotton with fatty acids in the rancid oil;⁴¹ this was necessary to get a good, fast colour and therefore repeated to ensure success.

After the cotton was oiled, washed, and dried, it was often treated with a warm brew of sumach leaves or oak gall nuts, both sources of tannins. They were also thought to ‘animalise’ the fibre,⁴² but some dyers discounted their usefulness completely.⁴³ Over the course of the nineteenth century, tannin use varies but generally declines in Turkey red processes.

In the ‘aluming’ step, the oiled cotton is soaked in a warm solution of aluminium salt, gradually taking up aluminium ions. For most of history, alum (potassium aluminium sulfate, $KAl(SO_4)_2 \cdot 12 H_2O$) was used, but only the purest kind. Turkey red dyeing is incredibly sensitive to trace contamination and the deleterious effect of iron was strong enough that no vessel or implement used in the process could contain it, not even an iron nail in a bucket.⁴⁴ Alum must be iron-free as well;⁴⁵ some texts specify ‘Roman alum’ in reference to alum mined at Tolfa, near Rome, which had a low iron content.⁴⁶

After the alumed cotton was washed and dried, it was dyed. This was done in a bath with ground madder roots (*Rubia tinctorum*), chalk, if necessary, and usually some cattle blood. Schaefer writes dyers in the Levant believed the blood had magic properties, and possibly as well that the colour enhanced the final colour.⁴⁷ Others said it coagulated with impurities in the dye bath,⁴⁸ or that it had no purpose at all.⁴⁹ One treatise says that while the dung and blood are often omitted, when used the colour was deeper and faster,⁵⁰ which accurately describes their role. They are not part of the Turkey red dye complex, but likely assisted the process, especially in the pre-industrial era. *Rubia tinctorum* contains hydroxyanthraquinone dyes, of which alizarin (1,2-di-hydroxyanthraquinone) and purpurin (1,2,4-tri-hydroxyanthraquinone) are the major products.⁵¹ Ground madder also has a large quantity of woody debris whose affect on the dyeing may have been reduced by the blood in the bath.

The final step was called ‘clearing’, and involved boiling the cotton with olive oil soap and sodium carbonate, often in a closed vessel, for an extended period of time. This was called *avivage* in French, for the brightening effect it had on the

colour.⁵² Clearing the Turkey red made the colour fast, though it would have to be nearly indestructible to have survived the clearing process.

The ‘new’ Turkey red process and chemistry

Advances in the field of chemistry during the years 1868-1875 prompted modifications in the Turkey red process that made it less time-consuming but did not fundamentally alter its chemistry. The first was the development of Turkey red oil, the first anionic surfactant, which came onto the British market from 1870-1875.⁵³ Preparing the cotton with oil is a characteristic of Turkey red dyeing and fundamental to its success. In the ‘old’ process the cotton was repeatedly soaked with rancid olive oil to make sure the fibres adsorbed enough fatty acids. Rancid olive oil, also called *huile tournante* or Gallipoli oil, was often produced in Italy and imported to Britain.⁵⁴ Looking to increase its usefulness for dyeing, research on treating olive oil with sulfuric acid began in the 1830s.⁵⁵ Turkey red oil is castor oil treated with sulfuric acid and neutralised. The reaction releases fatty acids from triglycerides, making the concentration in Turkey red oil higher than in rancid olive oil, which in turn increases the quantity of fatty acids adsorbed by the fibre with each application. Turkey red oil is also water-soluble, so it was much easier to wash away the excess than it was with olive oil. In this ‘new’ process, one application of Turkey red oil was often all that was needed, vastly reducing the duration of the process. Published ‘new’ recipes leave out the sheep or cow dung,⁵⁶ but it was said to assist in fixing the aluminium to the fibre and was in continued use at the Archibald Orr Ewing firm into the 1890s.⁵⁷ Based on this, the dung appears to have had a non-essential but useful auxiliary role in the process.

The second technical advancement was the successful commercial synthesis of alizarin, the primary colourant in madder. Turkey red dyeing required a lot of madder, which was imported to Scotland from Holland and France.⁵⁸ After the success of Perkin’s synthetic *mauveine*, chemists sought to save cost, freight, and time by finding a way to synthesise alizarin. Although ‘new’ process refers to the adoption of Turkey red oil, it is useful to include the concurrent transition to synthetic alizarin as well. Madder in the dye bath was replaced with alizarin powder or paste, with the quantity adjusted for the much higher concentration of colourant. As with the dung, dyers began to leave out cattle blood, though not universally.⁵⁹ In some cases they substituted blood albumen instead, a practice documented in the literature and in archive records, but it was considered somewhat less effective than whole blood.⁶⁰

Low end and Works Post 6 months 1875
Cost of Dyeing 112 lbs of Cloth Turkey red from dealer of

17 1/2	lb	Alumina	c	1/1	per lb	0.19	-
29	"	Pine oil	c	45	"	11	8
2 1/2	"	Sulphuric	c	1/4	"	0.2	2 1/2
23 1/2	"	Carbonate of Potash	c	2 1/2	"	4	4
18	"	Soda	c	4 1/2	"	-	7
11	"	Lump Alum	c	8 1/2	"	-	9
4	"	Corn Dyeing	c	9	"	-	1 1/4
	"	Whitening	c		"	-	1/4
6	"	Crystalline Soda ash	c	9 1/2	"	-	6
1 1/2	gallon	Bleach	c	2 1/2	gallon	-	4
2 1/2	"	Red liquor	c	6	do	-	1 1/2
	"	Indigo	c			-	0 1/2
6	oz	Crystals of Soda	c	5 1/2	per lb	-	2 1/2
2 1/2	lb	Vitriol	c	3 1/2	"	-	9
7	"	Muriatic acid	c	2 1/2	"	-	3
8	oz	Blanching powder	c	6 1/2	"	-	10 1/2
1 1/2	lb	White soap	c	2 1/2	"	-	6 1/2
1 1/2	oz	Zinc oxide	c	2 1/2	"	-	1
1 1/2	lb	Phosphate of Soda	c	2 1/2	"	-	4
8	oz	Patent dyes	c	3 1/2	"	-	0 1/2
4	"	Ground chalk	c			-	0
		Mathew's L670				-	1 1/2
		Mathew's at 694 gallon				-	
16	rod	Costs	c			5	
		Wages					10 8
		Iron & iron stand					10 1
		Sole cost per pound					3.67
		Cost per lb 7 1/8					

Fig. 5: Dyestuffs ledger from the Archibald Orr Ewing firm, 1873-1892. University of Glasgow Archive Services, Records of United Turkey Red Co Ltd, GB248 UGD13/4/1.

Synthetic alizarin

The synthesis of alizarin in 1868 by German chemists Graebe and Liebermann made it the first naturally-occurring dye created in a lab.⁶¹ It was commercially available not long after and between 1865 and 1870 the cost of a *quintal* of ground madder fell from 200 francs to 25 francs, destroying the French madder industry.⁶²

To synthesise alizarin, chemists sought to understand its structure. This was confusing for a number of years based on the mistaken assumption that naphthalene was the base structure of alizarin.⁶³ Adolf Baeyer, running a laboratory at the Gewerbeinstitut in Berlin, established the practice of breaking down molecules of interest and then designing ways to create them. Charles Graebe and Charles Liebermann, chemists in the Baeyer lab, isolated alizarin from madder and reduced it with zinc to discover the base hydrocarbon was anthracene and announced their discovery at a meeting of the Deutsche Chemische Gesellschaft in January 1868.⁶⁴ That summer, they devised a synthetic pathway, but it was cost-prohibitive due to the price of bromine and the yield was low. Nevertheless, bidding began for rights to the process and the chemists, along with Heinrich Caro at Badische Anilin und Soda Fabrik (BASF), continued to search for a better route.⁶⁵

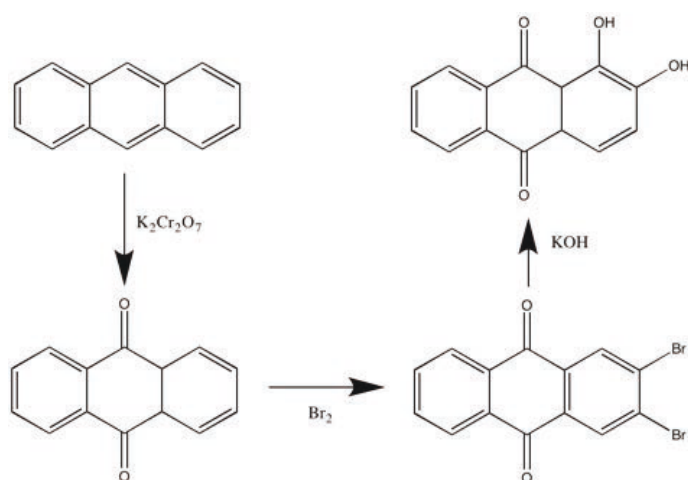


Fig. 6: Pathway for the first synthesis of alizarin via oxidation of anthracene, bromination, and hydroxylation.

Today, anthraquinone dyes are synthesised from anthraquinone sulfonic acids;⁶⁶ this was what the dye chemists were looking to achieve during the year 1869. Now that the structure of alizarin was known, Caro, Graebe, and Liebermann were not the only ones attempting to synthesise it. They were successful that summer; remarkably, so was another chemist at very nearly the same time. This was none other than William Henry Perkin, who invented *mauveine*, the first coal tar dye. By heating anthraquinone with sulfuric acid, anthraquinone sulfonic acids were obtained which could be further hydroxylated. The Germans filed for a British patent on 25 June 1869 and Perkin filed for his on the 26th, but his was sealed first due to revisions required in the Germans' application thought to be the result of unfair bias in the London patent office.⁶⁷

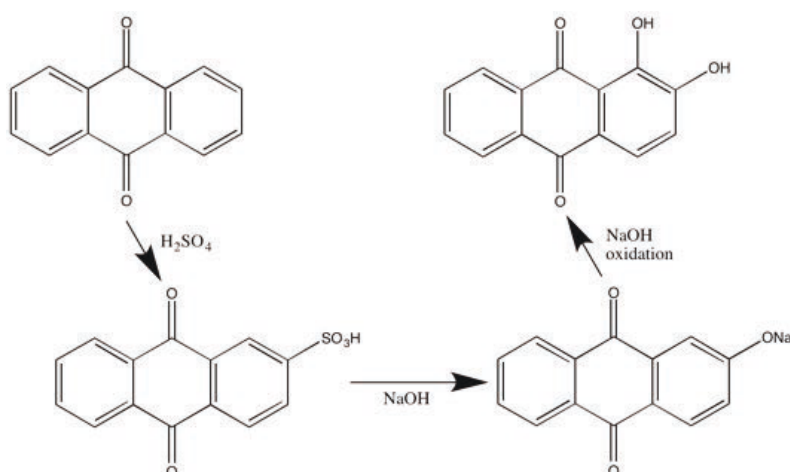


Fig. 7: Pathway for anthraquinone sulfonation, the synthetic route developed independently by Perkin and Caro, Graebe, and Liebermann in 1869.

Perkin continued his research and filed another patent in November 1869 for a process where anthracene was chlorinated, then sulfonated, before being oxidised to anthraquinone sulfonic acid and hydroxylated as before.⁶⁸ Due to the identical and almost simultaneous discovery by Perkin and the Germans, in 1870 they agreed to divide the market with Perkin keeping the British share while BASF had Europe.⁶⁹

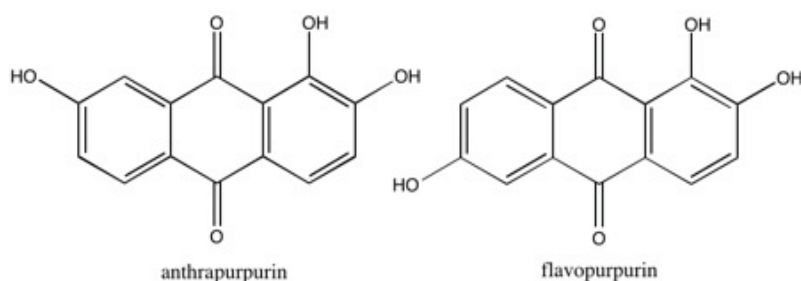


Fig. 8: Structures of anthrapurpurin and flavopurpurin, anthraquinone dyes found in late-19th c. synthetic alizarin.

Chemists soon realised synthetic alizarin was not pure and that side reactions during synthesis generated other hydroxyanthraquinone dyes. Two previously-unknown dyes in the mixture, anthrapurpurin (1,2,7-tri-hydroxyanthraquinone) and flavopurpurin (1,2,6-tri-hydroxyanthraquinone), were identified as major components of synthetic alizarin.⁷⁰ As a result, dye manufacturers sold different shades of alizarin that were mixtures of these compounds in various proportions. 'Blue' shades were mostly alizarin and 'yellow' ones were more a mixture of anthrapurpurin and flavopurpurin. Purpurin dyed a bright red, but was less fast than

the other isomers and more costly to manufacture.⁷¹ Despite the colours being slightly different to those produced by madder, dyers willingly adopted the alizarin products and found they could be used much the same as madder had been.⁷²

Legacy and collections

Turkey red was dyed in the West of Scotland for 150 years and yet very few traces of this once-great industry remain. To give an idea of its magnitude, the William Sterling and Sons firm was valued at approximately £152,000 in 1881, around £7.3 million today. Records from the United Turkey Red firm show the finished textile was exported to Calcutta, Madras, Rangoon (Yangon), Constantinople (Istanbul), Bombay (Mumbai), Singapore, Java, and Manila, to name a few from one ledger. In the eighty years since the end of Turkey red dyeing, knowledge and awareness of the process have largely faded into history. There are no former dye works still standing, though their sites can be visited on a walking tour of the Vale of Leven.⁷³ Because so many decades have passed there are no longer any dyers still living who could share their knowledge and experience, so the process had to be studied through published texts and archival records.

Fortunately, a wealth of dyed and printed Turkey red calico pieces have been preserved, often in the form of ‘sample books’. Their purpose is undocumented, but the vague notations and haphazardly cut samples indicate they were for internal company records rather than sales or display. Nine of these volumes dating from the 1850s-1900 are located in the Glasgow University SBA and were the primary source of historical Turkey red for this project. It can also be found, as sample books, miscellaneous cut pieces, and articles of clothing in the collections of National Museums Scotland (Edinburgh, Scotland, UK), Glasgow Museums (Glasgow, Scotland, UK), the Victoria & Albert Museum (London, England, UK), the Manchester School of Art (Manchester, England, UK), Bradford College Textile Archive (Bradford, England, UK), the Musée des tissus (Mulhouse, Switzerland), the Museum of Natural History (New York City, USA), the Cooper Hewitt Smithsonian Design Museum (Washington D.C., USA), and the Los Angeles County Museum of Art (Los Angeles, USA), the Technisches Museum (Vienna, Austria), and more. Other collections, especially for quilts, may have Turkey red pieces that have not been identified as such. The reputed fastness of Turkey red could have implications for how these pieces are displayed. Historical textiles are susceptible to fading from light exposure, one of the biggest issues in their conservation and display. Understanding more about its fastness and whether it is

more robust to display will make these beautiful, vivid Turkey red collections more accessible to the world.

In order to understand how and why Turkey red is fast, it is necessary to understand the chemistry of the dye complex. Furthermore, a means to test whether an identified piece is Turkey red will determine which objects are more suitable for display. These questions could not reliably be answered prior to the development of modern analytical techniques. Empirical analytical techniques in the nineteenth century could not characterise Turkey red because it exists only on the fibre and must be analysed there without extraction. Historically, Turkey red was identified by testing whether the colour on a thread persisted after an hour in dilute nitric acid.⁷⁴ This is neither in accord with modern conservation practices and nor a reliable means of assessment. The aim of this project was to learn more about the Turkey red complex and determine a microanalytical or non-invasive means of identifying textiles through a process-centred re-creation and analysis. Hopefully this will improve conservation and display practices for Turkey red textiles and perhaps revive a historical practice in the modern day.

Re-creation and analysis of Turkey red

Turkey red textile samples were dyed for this research following an 1886 method from J.J. Hummel, a professor at Yorkshire College in Leeds (England, UK) and dyer with practical experience in the Turkey red industry.⁷⁵ These replicas could be sacrificed while testing analytical techniques and also allowed the study of ‘intermediate’ samples from various stages of the dyeing process. Only finished Turkey red is preserved in the historical record, but analysis of replicas at each stage (oiling, aluming, dyeing) make it easier to identify changes after each treatment. Analysis of the replica and historical Turkey red indicates the pieces have similar characteristics that are consistent with the dyeing process. The UTR collection does not contain any dyer’s notes, only the ledger that tracks ingredients used. The directions from the J&P Coats collection are from the early 1900s, later than the focus of this project, but show the process had not changed. These documents show the methods published in dyeing manuals and treatises were the same as what textile manufacturers were using.

Dye analysis of historical textiles is most often done with high performance liquid chromatography (HPLC), a microanalytical technique in which a small fibre sample (~1 cm) is taken and the colorant extracted and separated. Recently, ultra

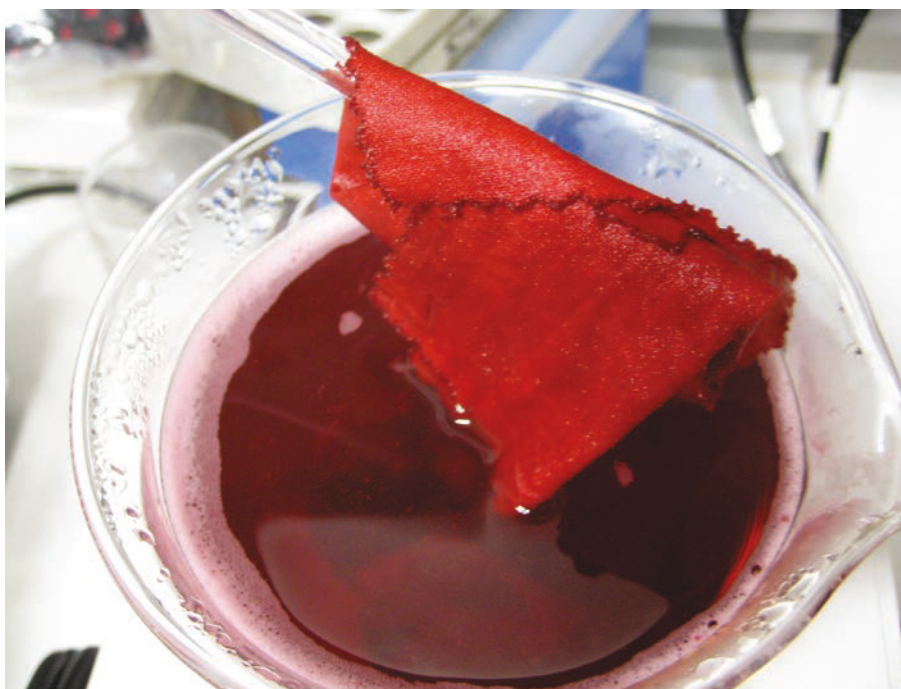


Fig. 9: Checking replica Turkey red sample for colour during dyeing, August 2015.

high performance liquid chromatography (UHPLC) instruments have permitted better separation and detection of components from even smaller sample sizes. Although the analysis requires sampling the object, the information gained is well worth the sacrifice if the thread can be taken without harming the item. Many HPLC studies of madder and madder-dyed textiles have been published, but no large study yet using UHPLC and nothing specifically on Turkey red.^{76, 77, 78, 79} Textiles dyed with madder are typically characterised by the presence of alizarin and purpurin, and possibly lesser components pseudopurpurin and munjistin. For Turkey red dyed with synthetic alizarin, anthrapurpurin and flavorpurpurin should also be detected, which would assist in determining the date of manufacture for some pieces. In this project, approximately 100 Turkey red pieces were sampled and analysed. Chromatograms were also taken of replica Turkey red samples, madder-dyed cotton, and a variety of madder extracts. References in the form of a set of historical synthetic alizarin samples provided by the Historisches Farbstoffsammlung at Technische Universität Dresden complemented the madder samples. The ongoing analytical work has identified anthrapurpurin in many historical Turkey red samples whose date of manufacture is consistent with the use of synthetic alizarin, as well as some whose date of manufacture is unknown, narrowing down when they could have been dyed. Flavorpurpurin is unavailable to purchase as a reference standard, so identification must be done by mass spectrometry.

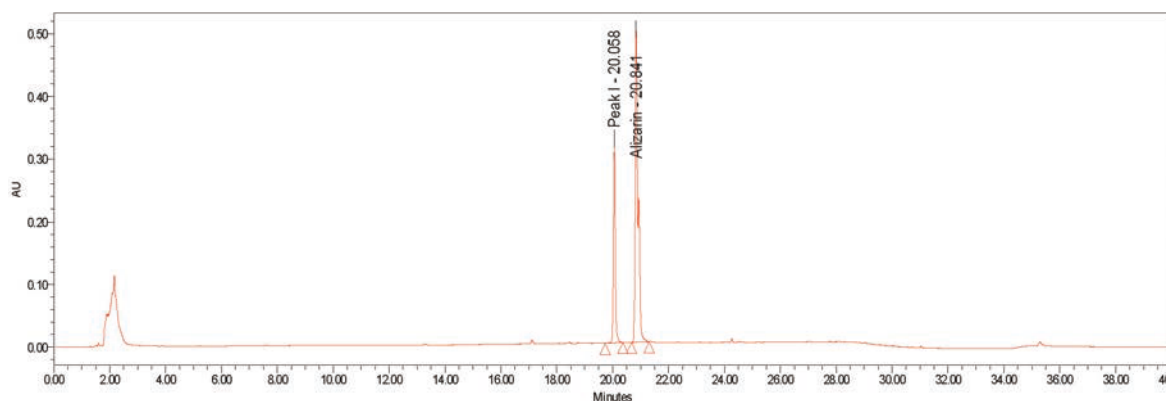


Fig. 10: uHPLC chromatogram of thread sample from German Turkey red handkerchief, item 1803-1899. Image courtesy of the Victoria & Albert Museum.

The identification of alizarin or purpurin on a cotton textile is not itself sufficient to characterise an object as Turkey red. The initial preparation of the cotton with oil is fundamental to dyeing Turkey red and should be present as well. Turkey red oil is still commercially available and was purchased for comparison to replica Turkey red oil. This was re-created for this project from descriptions and directions published by Knecht in 1893.⁸⁰ Turkey red oil was made by reacting castor oil slowly with sulfuric acid, then neutralising it. Historical samples were given by Ms Debbie Bamford, a natural dyer in Sheffield (England, UK), and the Society of Dyers and Colourists (Bradford, England, UK), allowing a comparison of historical, replica, and commercial oils. Along with castor oil and olive oil, the samples were analysed by ^1H and ^{13}C NMR. The spectra confirmed a similar composition for all Turkey red oils, which is primarily a mixture of free fatty acids (FFAs) with some glycerides. Confirmation of its chemistry narrows down potential reactions during the oiling process to those between FFAs and cellulose and makes FFAs a useful marker for identifying Turkey red.

Historically, researchers were aware the oil was present on the fibre because they could extract it, but it was never used as a means of identification. They were also unable to characterise its role in the dye complex because in extracting it from the fibre it was no longer Turkey red. *In situ* analysis is the only means to study the oil in Turkey red. This is in accord with modern heritage object research practices, which discourage any changes or damage to the item. Surface analysis by Fourier transform infrared spectroscopy (FTIR) was identified as a potential analytical tool, though with some complications. The instrument available at the CTCTAH is an FTIR-ATR instrument, a version where the sample is clamped in place to ensure contact with the crystal through which the beam is emitted.

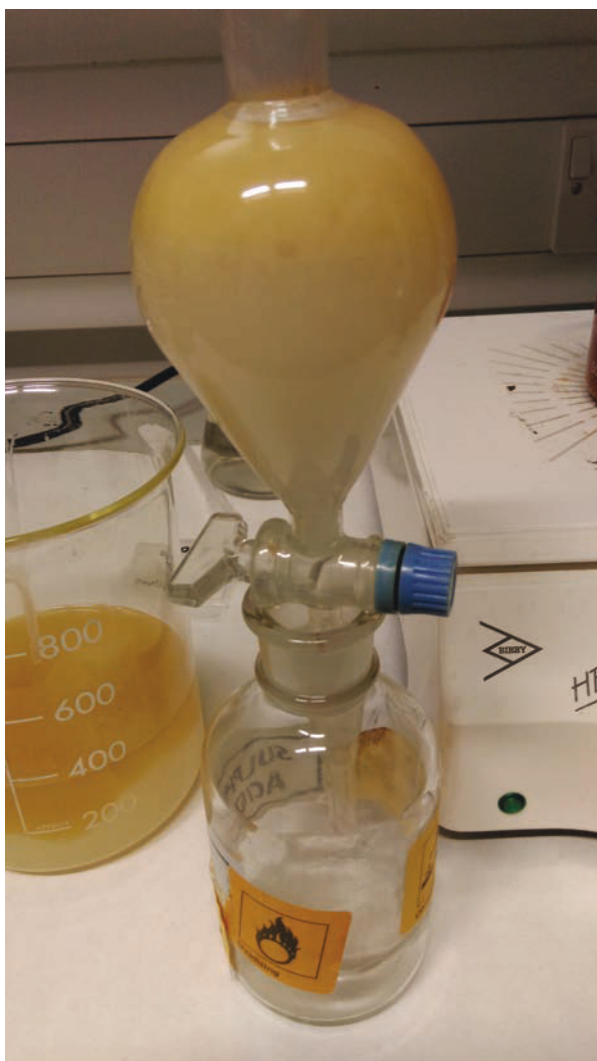


Fig. 11: Washing excess acid from castor oil treated with sulfuric acid to make Turkey red oil.

The cumbersome and fragile SBA sample books could not be manipulated onto this instrument without serious damage. The analysis was still possible thanks to recent developments in portable, hand-held FTIR instruments, which can be brought to a fragile object rather than bringing it to the instrument. Another FTIR technique, diffuse reflectance (DRIFTS), provides more information about uneven surfaces like textiles than ATR does and requires only contact with the item. Using a portable, hand-held instrument with a DRIFTS accessory, the Turkey red sample books were easily and non-destructively analysed. Comparison of the spectra to spectra of plain and oiled cotton and replica Turkey red showed distinct bands arising from the oil treatment, showing FFAs can be detected on the fibre and used to identify Turkey red.



Fig. 12: Diffuse FTIR analysis of United Turkey Red sample books, February 2015.

Alizarin synthesis

In addition to re-creating Turkey red, part of this project involved attempting to synthesise alizarin according to an 1870 patent of the process.⁸¹ Anthraquinone sulfonic acid is obtained either by the sulfonation of anthraquinone, or by sulfonating anthracene and oxidising it.

Re-creating the process was challenging and insightful, though ultimately unsuccessful. The route chosen was the sulfonation and oxidation of anthracene because it requires lower temperatures than the direct sulfonation of anthraquinone, which requires temperatures around 260-280 °C. The specialised equipment and precautions required to safely heat sulfuric acid this hot are beyond the scope of this research, but to sulfonate anthracene only 100-150 °C is needed. The final hydroxylation step occurs between 180-200 °C, the upper limit of what could be accomplished with available resources.

The first step was to translate the patent specification into a useful set of directions for synthesis. This proved difficult because the size and composition of the

vessel are not described. Inferences could be made from further literature on early alizarin synthesis,⁸² which is why this project takes a comprehensive look at the process from a historical and chemical perspective in order to gain a more comprehensive understanding of all aspects of Turkey red. To oxidise the sulfonated anthracene, the specifications say to ‘boil the whole strongly for some time’ with manganese oxide and that completeness of the reaction can be tested by taking an aliquot and carrying on with the synthesis to see if alizarin is produced. These kinds of directions are heavily dependent on the chemist being able to identify whether the reaction is proceeding and when it is complete. Very few physical descriptions of the reaction at various stages are provided, probably the result of the inventors not looking to disclose any more information than necessary to obtain a patent.

For this research, the complete synthesis was attempted once and a second started to try and identify anthraquinone sulfonic acid in the intermediates. The work was done in a glass, round-bottom flask heated by an oil bath. Anthracene is a pale greenish-white crystalline powder that turns yellow, then black, when sulfuric acid is added. During heating, the mixture continues to darken and becomes a dark greenish-black liquid with thick sediment. Heating the mixture with manganese dioxide produced no visible changes to the reaction. The excess acid was neutralised as directed and precipitated out as calcium sulfate, in theory leaving a solution of anthraquinone sulfonic acid sodium salts. This was evaporated by distillation, leaving a brownish-purple crust in the flask. It was difficult to safely obtain and maintain the minimum 180 °C required for hydroxylation and may be why the synthesis was unsuccessful. The reaction was heated with sodium hydroxide and a small quantity of water, becoming a thick, deep purple mass. This is described by Perkin in another patent, who says it should become blue or bluish violet.⁸³ The completeness of this reaction was also tested by taking an aliquot through the final phases, which were dissolution in water and precipitation of alizarin by sulfuric or hydrochloric acid.

Carrying out these steps yielded a precipitate that initially appeared to be alizarin, however after washing and collecting it on a filter it was clear the precipitate was white, not orange. Analysis by NMR was unsuccessful because the precipitate did not dissolve enough in water, chloroform, DMSO, acetic acid, methanol, or sulfuric acid to generate a useful spectrum. A second batch of anthracene was heated with sulfuric acid and the reaction stopped for analysis. NMR spectra indicated a complicated mixture of aromatic compounds that could not be distinguished without further purification. Separation was attempted using silica TLC plates but these were unsuitable for this analysis. TLC on reverse-phase C18 plates was more successful at moving the spot depending on the solvent conditions, but ade-

quate separation was never achieved. Purification was attempted using reverse-phase column chromatography that separated the mixture somewhat but complicated analysis by NMR due to the aqueous solvent used. Further work would have detracted from the textile focus of this project so the synthesis was left incomplete. Following a historical method to synthesise alizarin, although it yielded no alizarin or other dyes, was a useful way to gain insight into the experiences of the chemists who researched the process and emphasises the connections between the early synthetic chemical and textile industries.

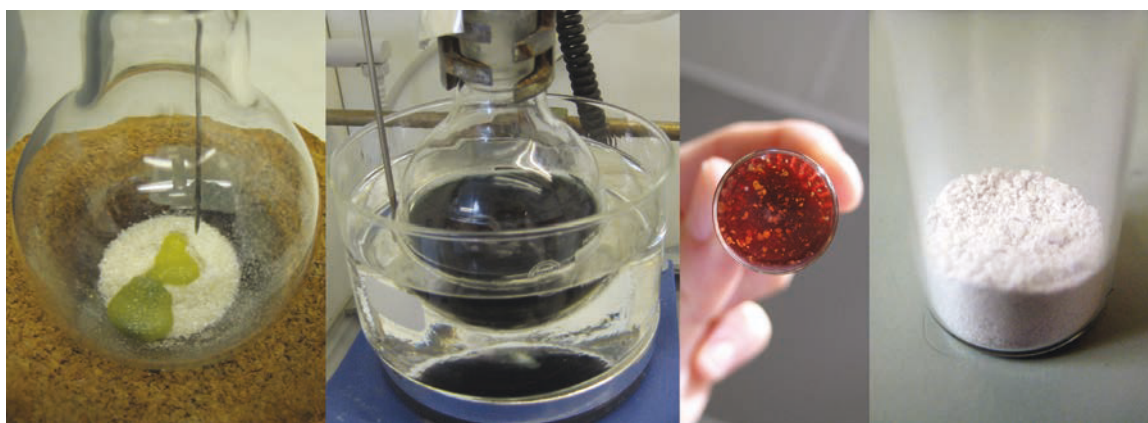


Fig. 13: From left: Adding sulfuric acid to anthracene; blackened anthracene/sulfuric acid mixture after heating; precipitated final product in solution; final product after washing and drying.

Conclusion

The development of the modern chemical industry grew from the search in the mid-nineteenth century for new synthetic dyes and during this period many texts on chemistry and dyeing were published. Today, dyeing and chemistry are no less linked. Modern analytical techniques allow researchers and conservators to gather more information from historical textiles to improve their preservation and accessibility for future generations. These objects are a valuable part of human history and give us a personal, relatable connection to the past.

This research on Turkey red at the University of Glasgow begins to fill in the long-standing gaps in our understanding of the process, paving the way for future research and re-creation. By studying the process and its chemistry, analytical techniques were identified which can identify Turkey red and possibly reveal more about when it was produced. This information also helps to finally charac-

terise the dye complex, an understanding that eluded dyers and chemists throughout history. Knowledge of the complex allows its fastness properties to be explored and hopefully quantified using modern parameters, part of a continuing project at the CTCTAH. This may have positive implications for the display conditions of these textiles, making them more available to the public to appreciate and experience. The multi-disciplinary approach this research uses was essential to the understanding of Turkey red. Studying the historical context and previous research on its chemistry set a useful context in which to understand how it was made. The hands-on experience of making Turkey red oil, synthesising alizarin, and dyeing samples was critical to fully understand the process and gives the modern researcher an appreciation for the work required.

Hopefully the success of this project will encourage the examination of other historical practices and processes, not just in textiles. The knowledge gained (or regained) may be useful in the preservation of heritage objects and reviving historical practices for our cultural heritage. Additionally, in a world of diminishing resources and increasing population, there may be useful things we can learn from practices abandoned in the name of progress. This project finished at the end of 2016 and the findings are published in the author's doctoral dissertation. To view a digitised United Turkey Red sample book from the SBA, please visit https://issuu.com/glasgow_dyes/docs/tr_print_book_6f9bac4d9674b8.

For colour images, which cannot be rendered in the printed issues, compare the electronic version of the *Mitteilungen* at <https://www.gdch.de/netzwerk-strukturen/fachstrukturen/geschichte-der-chemie/mitteilungen-der-fachgruppe-online.html>.

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