

Chromatographic analyses of selected historic dyeings from ancient Israel

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ABSTRACT Archaeological textiles excavated from ancient Israel attest to the artistic and scientific skills of the ancient dyer, spinner, and weaver. The textiles investigated in this study were excavated from the Dead Sea area sites of 'En Raḥel, Masada, the Cave of Letters, and 'En Boqeq, and date from the Roman period of two millennia ago to the late Byzantine period of the early 7th century CE. The analytical HPLC–PDA chromatographic and spectrometric results show that red alum-mordanted dyeings were predominantly produced from the plant roots of dyer's madder, *Rubia tinctorum*, which are rich in the orange-red dye alizarin. Purple colors were fashioned by double-dyeing with blue indigotin, probably derived from the woad plant, *Isatis tinctoria*, and with a purpurin-rich madder species, probably wild madder, *Rubia peregrina*. Two separate discoveries of the use of the Ararat cochineal and oak kermes scale insects on textiles from ancient Israel are singular finds. The new analytical dye investigations performed in this study have also clarified and corrected certain reported results that have been based on analyses from as far back as four decades ago.

Keywords: HPLC, madder, indigo, cochineal, kermes, archaeological textiles

Introduction

The study of ancient organic and inorganic colorants opens a historical window to one of the most fascinating chapters in the field of ancient technologies. Such an investigation of the coloring matters of ancient peoples involves an interdisciplinary approach that integrates history, archaeology, religion, botany, entomology, marine zoology, geology, and forensic-style analytical chemistry. The development of, for example, the 'scientific art' of textile dyeing through the course of time can be analyzed from the various archaeological textiles of different eras that have survived the ravages of time. The fashionable color preferences of ancient peoples can be better visualized by studying the physical and chemical makeup of these colors. The investigation of the natural colorants of historic importance leads to a better understanding of international commerce in antiquity, especially in the flow of pigments, dyestuffs, dyed goods, and of dyeing technologies from one geographical region to another. Appropriate conservation and restoration methods can be developed that would be suitable for the types of dyes used in the historic textile. Further, a determination of an artifact's coloring substance, and its light-fast properties and behavior to other climatic factors, will lead to better curatorial decisions as to whether – and for how long – to exhibit that object and under what conditions. Finally, the development of new and improved micro-scale methods of ancient-dye analyses may be transferable to other modern areas of analysis, such as in the determination of minute quantities in the pharmaceutical, forensic, and food areas and in the general quality and quantity control sector.

This study discusses the botanical and entomological dye-stuff sources of the organic colorants found on archaeologi-

cal textiles from the Judean Desert and northern 'Aravah in ancient Israel (Fig. 1). These four sites are west and south of the Dead Sea, and less than 30 km separates the northernmost site from the southernmost location. The focus of this paper will be on red, blue, and purple hues and the physical and chemical means by which they were produced.

Archaeological sites and their historical significance

It is important to understand the historical context of the analyzed textiles so that the impact of their finding, examination, and interpretation is better appreciated – not just on the scientific level, but also on the emotional and human scale. The accounts associated with the first two archaeological sites mentioned below – Masada and the Cave of Letters – are well known in Jewish history as they represent national tragedies during the Roman occupation of Judea. The second two sites are associated with other ethnic and religious groups that have settled or passed through this part of the world and have left their historical imprints.

Masada

The saga of Masada still resonates in the psyche of the modern state of Israel. King Herod built the Masada palatial fortress (*Metzadah* is Hebrew for 'fortress') in the 1st century BCE on an imposing Judean Desert cliff overlooking the Dead Sea (Yadin 1980). The Roman period Jewish historian, Flavius

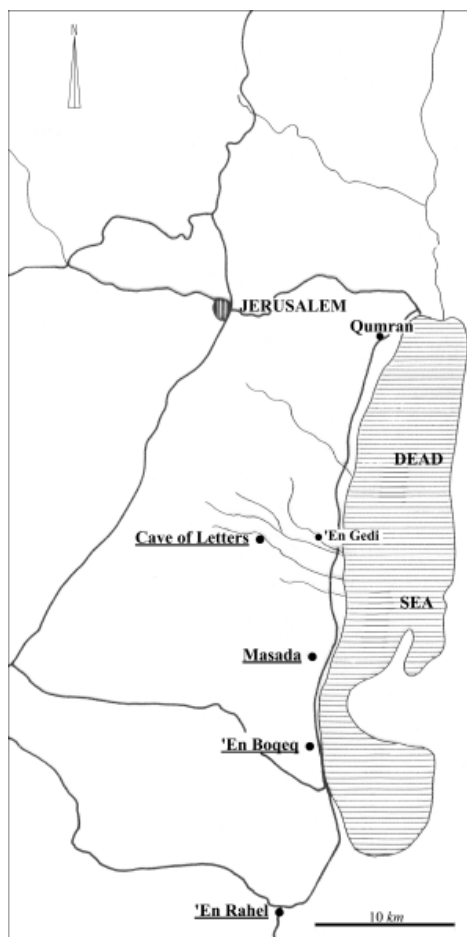


Figure 1 Dead Sea area archaeological sites (underlined) discussed.

Josephus, relates how a century after Herod's Masada was constructed, it became the last stronghold of those Jews who were militarily opposed to the Roman rule in ancient Israel (Josephus 1995). When defeat became imminent, the nearly thousand men, women, and children atop this fortress mountaintop executed mass suicide rather than await their fate at the hands of the Roman captors. Most of the textiles excavated at this site belong to those 1st-century CE rebels and their families (Sheffer and Granger-Taylor 1994).

The Cave of Letters

The last wave of rebellion against the Roman occupation of Judea was waged by Bar-Koziba (literally 'Son of *Keziv*', which probably referred to his native town) in the 2nd century CE. As history recounts, he was a charismatic leader and to some a messianic figure, who was renamed Bar-Kokhba (literally 'Son of a Star') by Rabbi Akiba, a well-known contemporaneous Talmudic figure who believed that Bar-Kokhba would be the 'Star' to redeem the Jews from all of their troubles caused by the Romans. This Judean Desert site has been named the 'Cave of Letters' as a number of letters written on scroll parchments and attributed to Bar-Koziba and his fellow rebels have been found. The textile fragments found at this site belong to these rebels and their followers (Yadin 1963).

'En Raḥel

Literally meaning 'Springs of Rachel', 'En Raḥel was a Nabatean fort in the 'Aravah valley, southwest of the Dead Sea. It probably served as a way station along the famous spice route from the Nabatean capital of Petra (now in Jordan) to the Gaza port and on to further points of commerce. Textiles, basketry, cordage and related items were found at this 2000-year old site (Shamir 1999).

'En Boqeq

This was also a way station, close to one of the spice routes and very close to the Dead Sea. The textiles excavated at the site belong to the late Byzantine period of the early 7th century CE (Sheffer and Tidhar 1991). Today, it sits a short walk across a highway from a luxury hotel located on the shore of the salty sea.

HPLC analytical methodology

Various analytical techniques have been employed for the study of textile dyes and pigments (Koren 1993a). The methods that supply the maximum information require that the colorant be extracted – stripped off – the textile substrate by means of an appropriate solvent system in order to dissolve the dyes. This colored solution can then be examined via various analytical methods. In the beginning of dye analyses, the most popular techniques were ultraviolet-visible (UV-vis) spectrophotometry, thin-layer chromatography (TLC), and infrared (IR) spectrometry. One of the best methods in use today is high-performance liquid chromatography (HPLC) followed by a spectrometric detection via a photodiode array (PDA) detector (also abbreviated as DAD), which is the method utilized in this study.

The data produced by the HPLC–PDA method are depicted as a chromatogram (Fig. 2), which is a graph of the relative amounts of light absorbed by the eluting substances at a particular wavelength as a function of time. This information yields the retention time, t_R , which is the amount of time a component is retained in the separation column (stationary phase) before eluting out by the mobile phase, and it is an important factor in the identification of the dye. Processing of the data can also produce contour plots, which are topographical-type mappings showing a 'bird's-eye view' of the light absorption intensities as 'mountains and hills' that are encountered at different times for each eluting component and at different wavelengths (Fig. 3). These contours are often invaluable in the identification of concealed component peaks. In addition to a chromatographic factor (t_R), the HPLC–PDA method also produces a spectrometric property, the substance's UV-vis spectrum, which supplies information regarding the wavelengths at maximum light absorption, λ_{max} , which will be shown later.

This study utilized various reverse-phase gradient elution methods with a polar acidic methanolic mobile phase and a

nonpolar C-18 stationary phase column, the details of which have been published elsewhere, as were the dye extraction procedures (Koren 1994a,b, 1995a, 1999).

Archaeological textiles

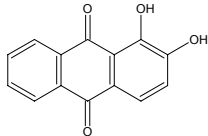
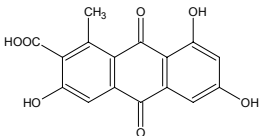
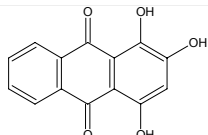
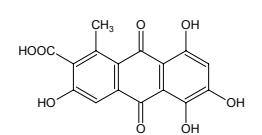
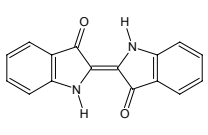
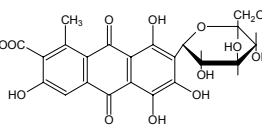
The ancient textiles discussed in this study are composed of fleece and weaves, and are catalogued below according to the main flora and fauna sources of the detected dyes (Fig. 4).

Plant sources for dyes: madder and/or indigo

From the dye analyses performed by the author on historic textile dyeings from ancient Israel (Koren 1993b, 1994b, 1995b, 1999), a picture of the botanical expertise of the ancient dyer becomes apparent. Results show that almost all of the red dyeings from about two millennia ago were produced by means of the use of the inner root parts of the plant known as dyer's madder, *Rubia tinctorum* L. During the Roman period, this plant was grown in the Levant as well as in Europe, and a related species was also native to India and its environs. The various madder species of plants from around the world contain a combined total of about 20 different colorants that belong to the hydroxy-antraquinone chemical grouping (Schweppe 1989). The main component produced from dyer's madder, however, is alizarin (Table 1), an acidic methanolic solution of which yields a wavelength at maximum absorption, λ_{\max} , of 430 nm, characteristic of its visible orange-red hue (Fig. 5). Alum-mordanted dyeings with the madder plant can yield various hues or shades of red from salmon-orange to brick-red colorations.

The dyeing with plant-extracted blue was effected by means of a fermentative hydrolysis or reduction process followed by

Table 1 Molecular structures of some plant and insect dyes.

Plant dyes	Insect dyes
 Alizarin	 Flavokermesic acid
 Purpurin	 Kermesic acid
 Indigotin	 Carminic acid

air oxidation. In the Levant and Europe of the Roman period, the source available was undoubtedly the leaves of the woad plant, *Isatis tinctoria* L., whereas *Indigofera tinctoria*, the indigo plant native to India and its environs, as its name implies, was another source for the blue pigment in that far-flung part of the world. Both plants can produce the blue pigment referred to as indigotin (Table 1, Fig. 5) so as not to confuse it with its plant source.

Masada red and purple

A beautiful example of the use of alizarin-rich dyer's madder in Masada textiles can be found in a number of dyeings (Koren

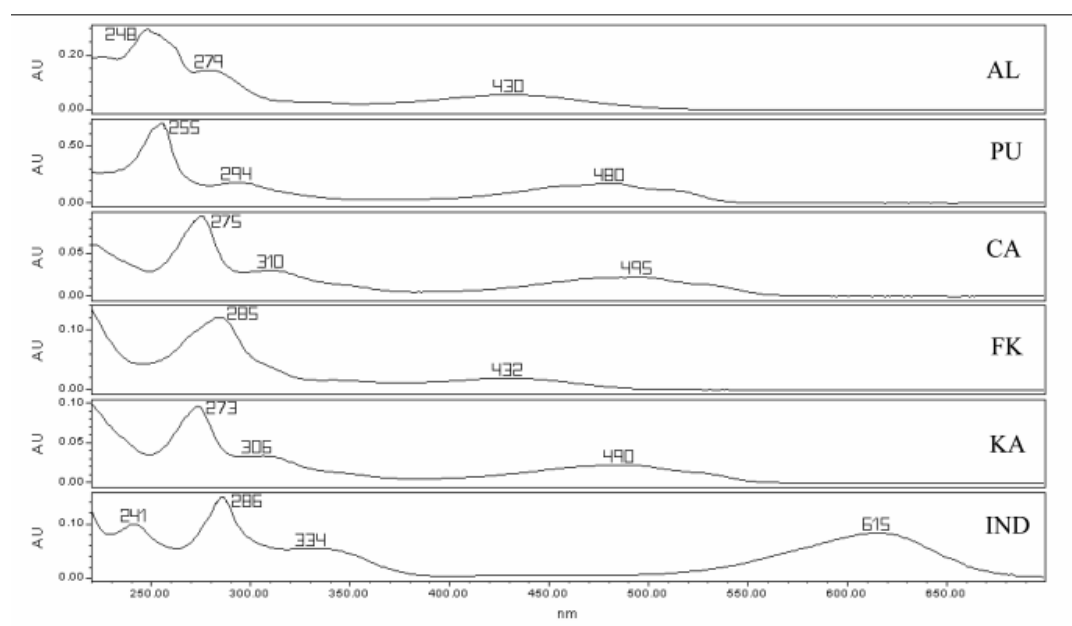


Figure 5 PDA-UV-vis spectra of acidic methanolic solutions of alizarin (AL), purpurin (PU), carminic acid (CA), flavokermesic acid (FK), kermesic acid (KA), and indigotin (IND) solubilized in dimethyl sulfoxide.

1994b; Koren *et al.* 1994), such as on the salmon-colored ground of Figure 4(a), which was probably from a garment – perhaps a mantle (Sheffer and Granger-Taylor 1994).

In addition to reds, purple dyeings were also the haute couture color of that period. The most noble of these was Tyrian Purple, which was produced from the hypobranchial glandular extracts of various *Murex*-type sea snails (Koren 1995c). The chemical ‘trademark’ of such a purple pigment produced from these and other species of mollusks is a dibrominated indigotin molecule substituted in the 6 and 6’ positions (see Table 1). The violets and purples produced from these mollusks were the most expensive, complex, sacral, and royal of all the dyes used in antiquity, and adorned the textiles of emperors, caesars, kings, military generals, Israelite high priests, and temples. Various edicts were enacted to ‘persuade’ the common folk that it would not be advisable for them to wear such real purple-dyed garments. All of these technical and legislative obstacles precluded the dyeing of *Murex*-purple by those who were on the lower rung of the socioeconomic scale. Nevertheless, the clever dyers found a means of circumventing these difficulties by developing a double-dyeing process to produce these popular hues. This ‘poor people’s purple’ was fabricated by mixing a red and a blue dye, the combination of which, as even the ancient color theorist realized, yielded purple colors whose desired hue could be fashioned by controlling the relative quantities of the red and blue components.

An example of a ‘fake purple’ can be seen in the same Masada textile fragment discussed above. Dye analyses determined that the purple of the notched band was produced by double-dyeing the blue indigotin pigment with madder red (Koren 1994b). The surprising discovery, however, was that although alizarin was detected, the red dyestuff used was rich in purpurin (Table 1). Analyses of the roots of the plant known as wild madder, *Rubia peregrina* L., have shown that they are rich in purpurin, and some varieties even have no alizarin content whatsoever (Wouters 1985). This indicates that such a species – instead of dyer’s madder – would probably have been used for the purple at Masada and other sites. This botanical choice is highly plausible and it seems quite likely that the efficient ancient dyer, knowing that the final hue desired was to be a double-dyed purple, would want to use a botanical source that was already rich in a red-purple component. Purpurin is a purplish dye, as its name implies, and obviated by its visible λ_{max} at 480 nm when dissolved in acidic methanolic solutions (Fig. 5).

‘En Raḥel shaded band

Another example of the use of the red-blue double-dyeing process to produce purple is seen in the 2000-year old weave from ‘En Raḥel (Fig. 4b). The upper band consists of indigotin-dyed fibers, while the lower one is an intricate mix of double-dyed fibers (Koren 1999). A microscopic examination has revealed the method by which the dyeing, spinning, and weaving were integrated in designing the final product. The transition from the blue area to the purple one is not sharp, as there is no exact border demarcation. The gradual shading pattern between adjoining bands was a design deliberately

commissioned by the weaver. In the ‘En Raḥel example, while the warp consisted of undyed (and now yellowed) wool, it is obvious that in order to form the intricate weft pattern it was necessary for the dyeing to have been performed on the sheared and washed fleece, then spun into yarns, and finally woven to the desired design. Three dyed fleeces – a blue only, a red only, and a purple double-dyed one – were spun, alone and together, to produce the following four differently colored weft yarns: (i) blue only yarns, (ii) mostly blue yarns, consisting of spinning together much blue fleece with some of the red and purple fleeces, (iii) blue-purple yarns, consisting of spinning together purple and blue fleeces, with some red, and (iv) violet yarns, consisting of spinning together mostly purple fleece with some blue and a trace of red.

‘En Boqeq plant blue

A beautiful navy-blue (violet) patch on an undyed textile (Fig. 4c), dating from the late Byzantine period, was found at ‘En Boqeq. Previous examinations of the colorant conducted in the early 1970s detected the presence of both indigotin and bromine, which would imply that this was a most important (and rare) molluskan blue-purple (Sheffer 1993)¹; this reported finding was also cited in a review (Koren 1993b). The methodologies used were TLC and visible spectrophotometry, and elemental analysis for the detection of bromine was performed via X-ray fluorescence (XRF). Subsequently, this violet patch was examined by the author via the HPLC method. The result obtained was that only a plant-based blue indigotin pigment was present without any brominated indigotin, which, as stated above, is a necessary ingredient of a real molluskan purple. Based on geopolitical and scientific considerations, this new result is not surprising. The early 7th century CE would be a rather late period of finding real purples as dyeing with *Murex* sea snails in the Levant died out with the onset of the Islamic conquests of this part of the world, which occurred at about this time. Additionally, elemental analyses are inappropriate for the determination of dye molecules because the element must be detected as an integral part of the overall molecule, and not just by itself, in order to implicate the presence of the whole dye. Elements belonging to different compounds are often present on the same substrate as the dye molecule. This is such a case, for it is not surprising and is in fact expected that bromine was found on this textile as subsequent XRF analyses on other dyed and undyed textiles from this site have all revealed the presence of some bromine. The ‘En Boqeq site is located very near to the Dead Sea, whose major industrial resource is bromine!

A similar problematic result was encountered with the analyses of traces of a blue pigment on the edge of a linen burial shroud from Bani Hasan (Egypt), an important archaeological site dating from 1900 BCE (housed in the British Museum in London). Previous examinations identified indigotin, and elemental analyses detected the presence of bromine, which led to the (incorrect) conclusion that the dye was a brominated indigo, indicative of the rare *Murex* purple dyeing.² HPLC analyses performed by the author on a sample from this textile showed, however, that only a plant-derived indigotin

was present. In the author's professional opinion, this nearly 4000-year old blue-dyed linen sample is one of the oldest, if not the oldest surviving indigotin dyeing.

The Cave of Letters purple balls of wool

The dean of Israeli archaeologists was unquestionably Yigael Yadin whose excavations and findings in the early 1960s in the Judean Desert led to major breakthroughs in our understanding of ancient Israel during the Roman period. He supplied Abrahams and Edelstein with textile samples from the Cave of Letters and they were the first to analyze dyeings from ancient Israel (Abrahams and Edelstein 1963, 1964). Their pioneering analytical efforts employed infrared (IR) spectrometry, which required a painstaking sample preparation method to purify each major component via a selective precipitation and extraction technique, and then producing classic KBr pellets of these samples for analysis. In one sample from a bundle of purple wool (Fig. 4d, left) they reported that this fleece was double-dyed with indigotin and a red dye produced from a scale insect, as carminic acid (Table 1) was detected. The use of an insect dye – a rare and high-status source – lent utmost importance to that dyeing, and, partly based on this analytical result, Yadin extrapolated that it could be an imitation to a Biblical blue-purple color known as *Tekhelet*, an authentic sample of which must be derived from a *Murex trunculus* mollusk.

These results were re-examined by the author via HPLC analyses on six purple fleeces with hues ranging from reddish to bluish, all found at this site (Fig. 4d, right). In none of these samples was an insect dye found; instead, the clear use of purpurin-rich madder together with indigotin was discovered in all, as in the Masada example discussed above. The contradictory results can be explained in three ways. One possibility is that the samples recently analyzed were not the same as the one examined about 40 years ago. While this can be theoretically correct, it is not highly probable as the purple fleeces recently analyzed run the gamut of all possible hues that would have been possessed by the Yadin sample. The second more probable explanation is that in the double-dyeing that was examined recently, the major component from the red dyestuff was purpurin, with alizarin playing only a secondary role. Unfortunately, the ancient sample's IR spectrum produced four decades ago was not compared with purpurin (or a combination of purpurin and alizarin), but with alizarin alone and with carminic acid. An additional difficulty with the 40-year old result is that the reported finding of carminic acid is historically and geographically problematic. This dye is the major component of a cochineal-type insect found in Turkey and known as Ararat cochineal (see also below). The breeding grounds of this insect are quite a distance away from the 2nd-century CE Cave of Letters archaeological site located near the Dead Sea. Much shorter distances would have been traversed within ancient Israel between the Dead Sea and the Galilee region, in which scale insects breeding on oak trees have been found. These Galilean insects were discovered recently to possess the chemical signature of the red dye produced by kermes,³ flavokermesic acid and kermesic acid (Table 1), which have noticeably different chromatographic behavior

from a cochineal's carminic acid (Fig. 2). Thus, if any insect dye was to be found in the Roman period bundle of purple wool, it would probably not be cochineal, but rather kermes (see also below).

Insect sources for dyes: cochineal and kermes

Entomological dye sources in the Levant for the production of red dyeings, alone or in combination with other dyestuffs, are the scale insects, cochineal and kermes (Wouters and Verhecken 1989), as mentioned above. These were obviously more prestigious and expensive than botanical sources for a number of reasons. A biological explanation is that while practically dye plants can be grown in one's backyard, these unique insects needed special breeding grounds and certain host plants. A chemical reason is that these insect dyes produced more vibrant reds than plant dyestuffs.

In the ancient Middle East, the obvious cochineal to be imported into this area would have been the one that goes by the popular names of Ararat, Armenian, or Turkish cochineal, as well as by *kirmiz*, and scientifically as *Porphyrophora hamelii* Brandt. This insect breeds on the ground-level upper roots of certain grassy weeds and the dried dark brown mature female insects are relatively flat with lines or scales on their shell. This cochineal produces crimson (bluish-red) dyeings on alum-mordanted wool, and the major dye component is the hydroxy-anthraquinone carminic acid, whose acidic methanolic solutions show a λ_{\max} of 495 nm (Fig. 5).

The other red-producing insect is known as oak kermes, *Kermes vermilio* Planchon, which breeds on the branches and leaves of certain oak trees at relatively high altitudes. The 'etymology of entomology' is quite interesting as the Biblical name for kermes in Chronicles II has an equivalent name – *karmil*. The round, pea-shaped, dark brown mature female insects are collected together with their larvae for the dye production. Their dye content consists primarily of the two components that constitute the 'kermes doublet' (Figs 2 and 3), the chemical 'trademark' of all modern kermes insects, flavokermesic acid (orange, λ_{\max} of 432 nm) and kermesic acid (red-purple, λ_{\max} of 490 nm), as shown in Figure 5. Alum-mordanted kermes dyeings on wool produce scarlet (orange-red) hues.

'En Boqeq cochineal

The late Byzantine period red patch from 'En Boqeq (Fig. 4e, left) was first examined in the early 1970s⁴ and found to contain an insect dye (Masschelein-Kleiner *et al.* 1991; Masschelein-Kleiner and Maes 1978), though there was ambiguity in the identification of the main component. One report identified it as kermesic acid, while another identified it as carminic acid. When the author analyzed one yarn sample from this dyeing via HPLC, it was found that madder was the source of the red dye. In order to solve these contradictory results, a small weave sample from this textile was then analyzed. A microscopic examination revealed that there were two differently colored reds; the warp yarns had a subdued dull red color, whereas

the weft yarns had a brilliant crimson red coloration (Fig. 4e, right). Chemical analyses via HPLC showed that the warp yarns were in fact differently dyed from the weft. The warp consisted of alizarin-rich madder, whereas the red component in the weft yarns was clearly carminic acid, which based on the geographic region and the archaeological time frame would have come from the Ararat cochineal previously discussed. This was the first time that a textile from ancient Israel was clearly found to be dyed with Ararat cochineal.

This red patch is an excellent example of the 'economical aesthetics' practiced by the ancient weaver. The red patch is a weft-faced tabby, so that the weft is what is mostly visible, and the warp is more or less concealed in the background. Thus, as the weaver was interested in producing the crimson look for this textile, the relatively expensive cochineal-dyed yarns took the weft center stage in this production, while the inexpensive red madder-dyed yarns were backstage in the warp. With an eye on the aesthetics of the finished product, red-dyed warp yarns were nevertheless used – not undyed ones – just in case a warp here and there would peek through and be visible to the eye of the beholder of this textile.

'En Raḥel kermes

From the 2000-year old 'En Raḥel site, a rare purple-banded weave was excavated (Fig. 4f). This purple was unlike any of the other purples previously mentioned. This was another example of a double-dyeing with a trace of indigotin for the blue, but whereas a madder plant was typically used for the red dye source, kermesic acid was found to be the main component in this purple band (Koren 1999). This colorant is clearly identified with an oak kermes insect dyestuff. As in the case of the 'En Boqeq cochineal dyeing mentioned above, this too was the first time that a kermes dyeing had been found on a textile from ancient Israel.

Additionally, the surprising find – or lack of it – was that flavokermesic acid (abbreviated as FK), always found together with kermesic acid (abbreviated as KA) in modern kermes dyeings, was decidedly absent. The absence of the FK component is most probably due not to the use of a specific kermes species that only consists of the KA dye constituent, but to the fact that

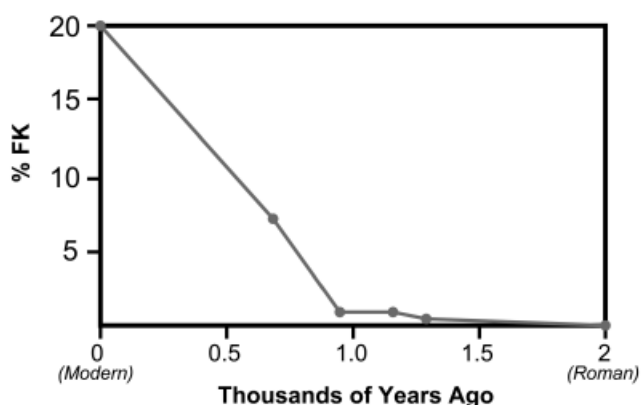


Figure 6 Relative quantity of FK detected today in ancient kermes dyeings (four middle points are from Wouters and Verhecken 1989).

FK must degrade over the archaeological time frame. A plot of this proposed degradation theory can be seen in Figure 6. In modern dyeings, FK is about 20% of the overall dye content; however, in Roman period textiles, there is no detectable FK present 2000 years later.

The modern-day implication of ancient kermes dyeings is that 'what-you-see-today' is not 'what-you-had-yesterday'. Thus, as the FK component has degraded over two millennia, it is then obvious that the original hue of the now purple band had a hint of orange to it. This is an important fact in attempting to reconstruct the original color of this band. Further work will be performed in order to properly study the kinetics of the ageing of kermes-dyed textiles.

Conclusions

This study has shown that preliminary microscopic examinations of both the warp and weft in ancient weaves are indispensable for the determination of the physical construct of the color seen on such artifacts, as in the red 'En Boqeq patch and in the 'En Raḥel blue-to-violet shading. Chemical analyses must employ the appropriate method for the determination of the dye components as in the purple balls of wool from the Cave of Letters. Additionally, elemental analyses are inappropriate for dye identifications as in the case of the blues from 'En Boqeq and Bani Hasan, as they can only detect atomic entities that may or may not be parts of the whole molecular dye species. This study has shown that whereas all natural dyestuffs contain various coloring substances, the most efficient analytical method for the determination of these dye components is one that leads to a separation (and thus purification) of the components, such as chromatography, followed by detection of each component via, for example, spectrometry. It is thus advisable to recheck ancient-dye analyses that were performed 30 and 40 years ago when such investigations were in their infancy and the appropriate techniques were not yet developed.

From the textiles examined in this and other studies, a global picture of the fashionable colors of ancient Israel becomes clearer. Alizarin-rich dyer's madder dominates in reds and a purpurin-rich madder species – probably wild madder – double-dyed with indigotin from woad produced purple imitations to those obtainable from molluscan sources. In two unique finds, two scale-insect dyes were discovered on two separate textiles. Kermes was found (with and without traces of indigotin) in some 2000-year old purple dyeings at one Roman period site, and six centuries later, the cochineal also enters this area from the base of Mount Ararat.

The dyeings discussed in this study demonstrate the chemical, physical, economical and artistic skills of the ancients in textile design, and their ability to integrate those fields into an aesthetically harmonious product. As a testament to their creations of the hoary past, many of their dyeings 'do not look their age' as they have maintained much of their vibrancy throughout the ravages of time. Their appearance today is not of a product that was chemically dyed and physically structured two millennia ago, but rather a design fashioned 'only yesterday'.

Acknowledgements

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Notes

1. A. Sheffer, personal communication.
2. H. Granger-Taylor, personal communication.
3. Koren, unpublished.
4. A. Sheffer, personal communication.

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