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DYESTUFF AND COLOUR ANALYSES OF THE SELJUK CARPETS IN KONYA ETHNOGRAPHY MUSEUM

R. Karadag and T. Yurdun

ABSTRACT
Reversed-phase high performance liquid chromatography with diode-array ultraviolet–visible spectrophotometric detection was used to identify dyestuffs in carpets of the Seljuk period. The objects that were examined originate from the thirteenth century and belong to the collection of the Konya Ethnography Museum in Konya, Turkey. Extraction from fibres was carried out with a 2:1:1 solution of hydrochloric acid, methanol and water. The study identified the main individual chemical components of natural anthraquinone, flavonoid and other dyes, including alizarin, purpurin, indigotin and ellagic acid, indicating that the most important classes of natural red and yellow dyes were used in these historical carpets. Colour measurements were made on the pile of the carpets and are reported as CIELAB L^*, a^* and b^* values. The data from the dyestuff analyses and colour measurements were used to inform conservation and restoration.

ÖZET
Konya Etnografya Müzesinden alınan 13th yüzyıl Selçuklu Dönemine ait halıların boyar madde analizleri UV-Vis detektorlü HPLC (yüksek basınçlı sıvı kromatografisi) cihazı ile analiz edildi. Renkli halı ilmeleri ve atkı ipliklerinin ekstraksiyonu HCl/metanol/su (2:1:1) karışımı ile yapıldı. Tarihi halılarda kırmızı ve sarı doğal boyalar bulundu. Bu çalışmada, başlıca antrakinon ve flavonlara (alizarin, purpurin, indigotin, ellajik asit) ait doğal boya mddeleri bulundu. Halı örneklerinin renk ölçümleri CIELAB cihazı ile L^*, a^* ve b^* değerleri ölçüldü. Boyalara ait bilgiler ve renk analizleri restorasyon ve konservasyon için kullanılması önerildi.

INTRODUCTION

The collection of the Ethnography Museum in Konya, Turkey contains carpets that came from the mosque and the tomb of Sultan Alaaddin Keykubad in Konya and are considered to be the earliest group of surviving knotted pile carpets produced under Seljuk rule in the first half of the thirteenth century on the Anatolian peninsula. They were woven in the Gordes knot with rich colours, warm tones and a vast variety of designs, proving the resourcefulness of the weavers (Figs 1 and 2). The central field of these large carpets is an overall geometric repeat pattern while the borders are ornamented with a large-scale, stylized, angular calligraphy termed Kufic, pseudo-Kufic, or Kufesque.

In this study, the colour of the piles and weft fibres of the carpets from the Seljuk period in Konya Ethnography Museum were first measured and reported using the CIELAB method (CIE is the Commission Internationale de l'Eclairage and the method measures the so-called L*, a* and b* parameters). Secondly, dyestuff analysis was conducted on extracts from the piles and weft fibres, which were analysed by high performance liquid chromatography with diode-array detection (HPLC–DAD). Thirdly, new fibres (piles and weft) were dyed with the same dyestuffs and same methods using historical recipes as a guide. The colours of the newly-dyed fibres were measured using the CIELAB method for comparison.

The colour of the fibre is the result of three combined factors: the spectrum of the light source, the spectral reflectivity of the fibre colour, and the spectral sensitivity of the eye. The CIELAB (1976) system was introduced to describe the colour that results from these three factors. The system is a three-dimensional space, with coordinate axes L*, a* and b*. The L* axis denotes the lightness of the colour (an L* of 0 corresponds to black, while an L* of 100 denotes white), a* represents the green–red axis (a* negative: green, a* positive: red), and b* represents the blue–yellow axis (b* negative: blue, b* positive: yellow). Each fibre colour can be represented as a set of values for L*, a* and b*, and consequently as a point in this colour space [1].

Fig. 1 Seljuk carpet (inventory No. 841) from the Konya Ethnography Museum.

The difference between two colours measured in the CIE L*a*b* colour space is calculated using the equation:

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

If ΔE is equal to zero there is no difference between two colours. Small values of ΔE indicate that there is little difference in colour, with greater values of ΔE signifying larger colour differences.

The identification of material in carpets that are significant for cultural heritage has received significant attention because it is important for the development of appropriate conservation and restoration strategies. Natural dyes have the advantage that their production implies the use of renewable resources, causes minimum environmental pollution and has a low risk factor in

Fig. 2 Seljuk carpet (inventory No. 862) from the Konya Ethnography Museum.

relation to human health. They have been analysed, initially by thin layer chromatography (TLC) and more recently by high performance liquid chromatography (HPLC) with diode array detection (DAD) [2–4] and mass spectrometric detection (MS) [5, 6], which has been utilized successfully to identify the active colouring principals in historic yarns and textile fibres [7].

Fig. 3 The roots of *Rubia tinctorum* L., which were used for red and violet colours in the rugs examined.

Historical textiles were dyed with natural dyes that were mainly derived from plants or insects. Dye identification for historical textiles is usually based on comparison with known references. In this study, the dye sources that were used for reference are as follows.

Cochineal (*Dactylopius coccus* Costa): The dye insect is one of the most important natural dyes used to produce fast reds. The dye was discovered by Mexican dyers in about 1000 BC. It is native to Mexico, but the cochineal insect was brought to Europe by the Spanish in the early sixteenth century after the European discovery of America.

Kermes (*Kermes vermilio* Planchon): In the antique period, kermes dyeing was of great importance in the Near East and southern Europe. The origins probably go back to the Sumerians, that is, to the third millennium BC. The insect was the source of the most important red dye known to the ancient Mesopotamians.

Ararat kermes (*Porphyrophora hamelii* Brandt): This insect is native to the area of Mount Ararat in Turkish eastern Anatolia and in Armenia. They flourish in the salt marshes on both sides of the River Aras (Araxas), which flows past the north side of Mount Ararat and forms the border between Turkey and Armenia. Armenian sources cited their usage in dyeing silk and for the colouring of miniature paintings in the fifth century AD.

Lac (*Kerria lacca* Kerr): is produced from an insect found in India and Southeast Asian countries such as Thailand, Malaysia, Cambodia, Laos, and Indonesia. Around 1500 bc, lac was mentioned in the *Atharvaveda*, one of the oldest Indic texts. The insect was imported from India to the Near East more than 2000 years ago, but after the introduction of the cochineal, the use of lac in dyeing decreased.

Madder (*Rubia tinctorum* L.): Madder is one of the most important dye plants used to produce fast reds [8]. It has been used from 3500–4000 BC to the present for red dyeing, Fig. 3.

Gall nut (*Quercus infectoria* Oliv.): The plant was used both for dyeing and mordanting, Fig. 4 [9].

Indigo: There is dispute about whether dyeing with indigo was first developed in Indian or in Egypt. The name *indigo*

Fig. 4 The galls of *Quercus infectoria* Oliv., which were used to produced the black colour in the rugs examined and also as a mordant.

for the most important dye of the ancient world suggests India. For hundreds, perhaps even thousands, of years India was the greatest producer of indigo. The most important indigo plant is indigo (*Indigofera tinctoria* L.) while the woad plant (*Isatis tinctoria* L.) was another important source of indigo in Greece, in the Roman Empire and, from the Middle Ages, throughout Europe [10].

MATERIALS AND METHODS

Mordanting and dyeing procedures

The mordanting and dyeing of wool fibres were performed as described below [9]. Traditionally, a mordant was applied prior to dyeing (called pre-mordanting) to assist the adsorption of the dye and to promote good bonding of the dyestuff and the fibre. Mordants are generally metal salts that hydrolyse in hot aqueous solution; the most commonly-used mordants are alum [potassium aluminium sulphate ($KAl(SO_4)_2{\cdot}12H_2O$)], iron [iron (II) sulphate ($FeSO_4{\cdot}7H_2O$)] and tin [tin(II) chloride ($SnCl_2{\cdot}2H_2O$)]. Fibres with an animal origin (such as silk and wool) have basic chemical groups (mainly amino and carboxyl groups) to which the hydrolyzed metal ions bond. During dyeing, the basic groups on the dye molecules then bond to these metal ions that are attached to the mordanted fibres, so that a strong chemical bond is formed from the fibre to the dye molecule via the metal ion.

To begin the dyeing process, the fibres are immersed in an aqueous solution of the appropriate metal salt (mordant bath) and heated to 90°C for one hour. The mordanted fibres are removed from the mordant bath and left to dry in air. The dyeing procedures were performed in accordance with historical dyeing methods cited in the literature [10]. In the dye bath, the ratio of the fibres to dye extract is 1:25; so that, for example, if 1 g of wool is to be dyed, 25 g of dyestuff is used. To prepare the dye bath, insect or plant dyestuffs is extracted with water at 90°C for one hour and then resultant solution is filtered. Mordanted fibres are immersed in the dye bath and the temperature is gradually raised to 90°C and is kept at this temperature for about 30 to 60 minutes. The dye bath is allowed to cool to around 30°C and the dyed fibres are then removed, squeezed, rinsed thoroughly with water and dried in the shade.

Table 1. Colour measurements in the CIELAB system for thirteenth-century Seljuk carpets in the Konya Ethnography Museum.

Inventory number	*Colour*	*L**	*a**	*b**
841	Blue	46.82	–4.61	–6.98
	Light red	65.04	17.24	16.23
862	Green	52.06	–3.67	5.69
	Dark blue	38.2	–4.58	–5.44
	Red	41.09	32.96	18.86
	Yellow	73.89	6.5	29.15
	Blue	57.33	–6.27	–4.06
1033	Green	46.01	–4.32	3.56
	Red	39.13	23.03	13.57
	Violet	38.2	5.21	6.9
	Blue	23.37	–2.85	–3.8
1034	Light green	78.49	–2.72	3.82
	Green	66.46	–1.7	12.52
	Red	52.57	20.74	10.83
	Black	36.98	5.75	12.61
	Blue	60.33	2.82	11.04

In this study, wool samples were dyed with five dye plants: madder (*Rubia tinctorum*); weld (*Reseda luteola*); gall nut (*Quercus infectoria*); acorn cups (*Quercus ithaburensis* Decne); and indigo (production from *Isatis tinctoria* L. or *Indigofera tinctoria* L.). As mentioned above, the methods used were those given in historical recipes [10].

CIELAB measurements

The L*, a* and b*values for the Seljuk carpets were measured with a Konica Minolta CM 2003d spectrophotometer/colorimeter using CM-S100w *SpectraMagic NX* software to process the colour data, which are given as CIELAB values in Table 1.

Reagent and standards

All the reagents were analytical grade unless stated otherwise. High purity water was produced by passing though a Milli-Q treatment system (Millipore, Bedford MA, USA) and the HPLC mobile phase was prepared using Milli-Q water.

The following standard materials were used as references: carminic acid from Sigma Chemicals; luteolin from Roth; alizarin from Merck; and kermesic acid, pseudopurpurin and purpurin, which were synthesized by the University of Jordan for the FP6 project MED-COLOUR-TECH.To identify the dyes on the carpet textiles, dyed wool standards were prepared using the following plant dyes: madder (*Rubia tinctorum*); weld (*Reseda luteola*); gall nut (*Quercus infectoria* Oliv.); acorn cups (*Quercus ithaburensis* Decne); and indigo (produced either from *Isatis tinctoria* L. or *Indigofera tinctoria* L.). The standards coloured pieces of wool were prepared in the Laboratory for Natural Dyes in the Faculty of Fine Arts at Marmara University in Istanbul.

Extraction Procedure

The Seljuk carpets samples for this study were provided by the Konya Ethnography Museum; the carpets originated from the Eşrefoğlu Mosque in Beyşehir and the tomb of Sultan Alaeddin Keykubad in Konya. Dyestuff extraction was carried out using the method described previously [11–13]. Historical carpet samples of 0.4–1.0 mg were hydrolyzed with 400 µL of a 1:1:2 mixtures (v/v/v) of water, methanol and a 37% solution of hydrochloric acid in conical glass tubes for precisely eight minutes in a water bath at 100°C to extract the organic dyes. After rapid cooling under running cold water, the solution was evaporated

Table 2. Colorant compounds identified by HPLC-DAD and the inferred dye source for the samples investigated from thirteenth-century Seljuk carpets in the Konya Ethnography Museum.

Inventory number	*Sample colour*	*Compounds detected*	*Inferred dye source*
841	Dark blue	Indigotin	*Isatis tinctoria* L. or *Indigofera tinctoria* L.
	Red	Ellagic acid, alizarin and purpurin	*Quercus infectoria* Oliv. or *Quercus ithaburensis* Decne and *Rubia tinctorum* L.
862	Green	Indigotin and flavonoid	*Isatis tinctoria* L. or *Indigofera tinctoria* L. and unidentified plant
	Red	Alizarin and purpurin	*Rubia tinctorum* L.
	Yellow	Flavonoid	Unidentified plant
	Green	Indigotin and flavonoid	*Isatis tinctoria* L. or *Indigofera tinctoria* L. and unidentified plant
1033	Red	Alizarin and purpurin	*Rubia tinctorum* L.
	Green	Indigotin and flavonoid	*Isatis tinctoria* L. or *Indigofera tinctoria* L. and unidentified plant
	Violet	Ellagic acid, alizarin and purpurin	*Quercus infectoria* Oliv. or *Quercus ithaburensis* Decne and *Rubia tinctorum* L.
	Blue	Indigotin	*Isatis tinctoria* L. or *Indigofera tinctoria* L.
1034	Blue	Indigotin	*Isatis tinctoria* or *Indigofera tinctoria* L.
	Light green or blue	Indigotin and flavonoid	*Isatis tinctoria* L. or *Indigofera tinctoria* L. and unidentified plant
	Red	Ellagic acid, alizarin and purpurin	*Quercus infectoria* Oliv. or *Quercus ithaburensis* Decne and *Rubia tinctorum* L.
	Green	Ellagic acid	*Quercus infectoria* Oliv. or *Quercus ithaburensis* Decne
	Black	Ellagic acid	*Quercus infectoria* Oliv. or *Quercus ithaburensis* Decne

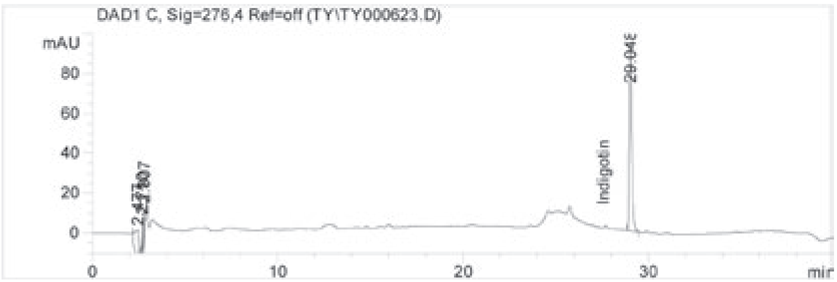

Fig. 5 Chromatogram of indigotin, which was found in rugs from the Konya Ethnography Museum (inventory Nos 841, 862, 1033 and 1034).

just to dryness in a water bath at 50–65°C under a gentle stream of nitrogen. The dry residue was dissolved in 200 μL of a 2:1 (v/v) mixture of methanol and water and was centrifuged at 2500 rpm for 10 minutes. Then 25 μL or 50 μL of the supernatant liquid was injected into the HPLC instrument. The residue was dissolved in 200 μL of N,N-dimethylformamide to dissolve any indigotin present, as this is not soluble in the methanol and water mixture; this solution was then injected into the HPLC system separately.

Instrumentation

Chromatographic experiments were performed using an Agilent 1100 series system (Agilent Technologies, Hewlett-Packard, Germany), which included a G1311A gradient delivery pump with a 50 μL loop and Rheodyne valve (7725i sample injector), a G1315A diode-array detector, a G1322A vacuum degasser and a G1316A thermostatted column compartment. A Nova-Pak C18 analytical column (3.9 × 150 mm, 4 μm particle size, Waters part No. 086344), protected by a guard column filled with the same material, was used. The analytical and guard columns were maintained at 30°C and the HPLC gradient elution was performed at a flow rate of 0.5 mL per minute using the methods of Halpine *et al*. [12] and Karapanagiotis *et al*. [14]. Chromatographic separation was accomplished using a gradient elution program that utilizes two solvents: solvent A contains water with 0.1% trifluoroacetic acid and solvent B acetonitrile (ethanenitrile) with 0.1% trifluoroacetic acid. The chromatograms were obtained by scanning the sample from 191 to 799 nm with a resolution of 2 nm and chromatographic peaks were monitored at 255, 268, 276, 350 and 491 nm; the data were recorded and analysed with Agilent Chemstation software.

Dyeing new material

The dye sources to be used to produce the new material were determined from the dyestuff analyses on the Seljuk carpets, Table 2. New wool piles and wefts were dyed with the appropriate dyestuff using historical recipes with the aim of producing materials whose colour differed from the originals by less than one ΔE unit.

RESULTS AND DISCUSSION

In this study, samples from four Seljuk carpets provided by the Konya Ethnography Museum collection were studied, Tables 1 and 2. The components in the piles and wefts that were detected using HPLC-DAD — alizarin, purpurin, ellagic acid and indigotin (Figs 5 and 6) — indicated that natural dyes of plant origin had been used to dye these carpets. The components that were identified, based on a comparison of the absorption spectra acquired for the samples with those for reference standards, are listed in Table 2. The colours of the piles and wefts of the Seljuk carpets were measured by CIELAB (Fig. 7) and are reported in Table 1.

By using colour measurement to assess the success of dyeing, it was possible to produce piles and wefts to be used at the

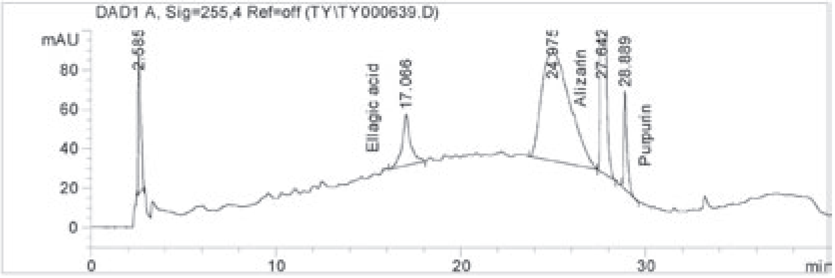

Fig. 6 Chromatogram of ellagic acid, alizarin and purpurin, which were found in rugs from the Konya Ethnography Museum (inventory Nos 841, 1033 and 1034).

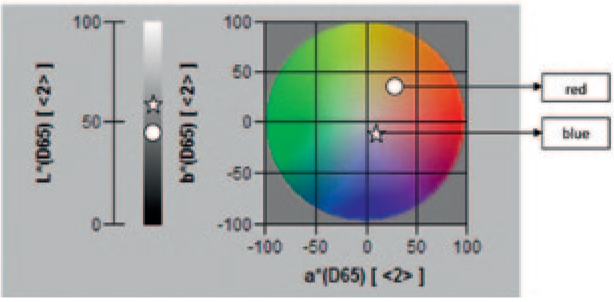

Fig. 7 Colour measurements for blue and red areas on a Seljuk carpet (inventory No. 862) from the Konya Ethnography Museum.

Fig. 8 New wool samples dyed according to historical recipes using the dye plants identified by the study.

restoration that were not only dyed according to the historical recipes (Fig. 8) using the same dyestuffs, but were also very close in colour, with ΔE values of one or less.

The results of the dyestuff analysis for the Seljuk carpet samples were compared with results of dyestuff analysis for some sixteenth-century Ottoman silk objects in the Topkapı Palace Museum [15]. It was observed that insect dyes were the source of the red colours in the silks at the Topkapı Palace Museum, while madder (*Rubia tinctorum*) rather than insect dyes appeared to be the source of the red colour in the carpets included in this study.

Generally, weld (*Reseda luteola*) is the source for yellow and green colours in the Topkapı Palace Museum silk objects, but in these carpets the source of the yellow dye has not been identified. In many of the samples residues characteristic of gall nuts (*Quercus infectoria*) or acorn cups (*Quercus ithaburensis*) were detected; *Quercus infectoria* Oliv. is a common tannin-containing plant that was used in the Seljuk and Ottoman textiles for dyeing, increasing the brightness or as a mordant.

The green colours in the Topkapı Palace Museum silk were based on a mixture of weld (*Reseda luteola*) and indigo. Only one dyestuff component (ellagic acid) was detected in the green sample from carpet No. 1034, and it is possible that this sample was dyed with gall nuts (*Quercus infectoria*) or acorn cups (*Quercus ithaburensis*). Indigotin and a flavonoid were detected in the green samples from carpets No. 862 and No. 1033. Although the same flavonoid was also found in the yellow sample from carpet No. 862, it was not possible to identify the source of the flavonoids found in these Seljuk carpets.

For the textiles in the Topkapı Palace, indigotin was used in the blue colours, while the greens were produced using indigotin and weld and the violet shades by a combination of, indigotin and insect dyes [15].

Finally, gall nuts or acorn cups were the source both for the black colours in the Topkapı Palace Museum silk textiles [15], and for the black colours in the Seljuk carpets.

CONCLUSIONS

From a stylistic point of view, the designs and themes used in these carpets are characteristic of the Seljuk era; for instance, Kufi themes, unique to the Seljuks, may be observed at the edges of the carpets.

The dyestuff sources were identified from the colorants detected in these carpets and wool fibres were dyed by using these dyestuffs according to the historical recipes to provide materials for the restoration process. The colour values of the dyed wool fibres and the original carpets were measured using the CIELAB system and the results were used to select wool fibres with the same colour values to be used in the restoration.

In the past, the restoration process at various museums was made using materials that had colours close to those of the original objects but, with time, big differences between the colour used in the restoration and the colour of the object occurred as the colours changed in a different manner. This situation arose because of the different chemical and physical properties of the materials and colours of the original objects and those used for the restoration.

Hence, it is recommended that the same dyestuffs and dyeing processes are used for the conservation and restoration, and such a procedure is proposed for future restoration. Knowing the physical and chemical properties of the dyestuffs in the objects is, therefore, a very important first step in the conservation and restoration.

To conclude, in the restoration it is important to use fibres that are dyed with the same dyestuffs to the same colour values as the original materials for two reasons: to eliminate the colour differences between the restored and original sections which may occur over time or under particular environmental conditions; and to minimize any chemical and the physical interactions that may happen over time or as a result of environmental conditions.

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