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PERIODIC COMPARISON OF TWO SELECTED HISTORICAL CAFTANS BY NON-DESTRUCTIVE AND MICRO ANALYSIS METHODS

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ABSTRACT

Identification of an art object material of cultural heritage had received significant attention, because of its importance for the development of appropriate restoration and conservation strategies. In this work, two important caftans were analysed in the Topkapi Palace Museum. The caftans were dated that one 16th century and other 18th century by art historians. But, designs of the caftans have been same features (characteristics). To compare these two art objects, the caftans were investigated by non-destructive and microanalysis methods. Multi analytical techniques were used for the analyses. These methods are high performance liquid chromatography (HPLC), scanning electron microscope energy disperse X-ray (SEM-EDX), technical analyses by optical microscopy and CIEL*a*b* for colour measurements.

KEYWORDS: Caftans, historical art object, HPLC, SEM-EDX, optical microscopy, CIEL*a*b*, dyestuff, metal thread.

INTRODUCTION

While numerous museums around the world hold Ottoman textiles in their collections, the Topkapi Palace Museum has the largest and most comprehensive collection in the world. These textiles often represent the highest form of cultural, artistic and socio-economic discoveries of their time periods. Information embedded in these textiles, therefore, is not only important to Turkey's textile sector, but is of utmost value for the world's textile heritage. Ottoman textiles reached their peak in the 16th century. In this work, two caftans were examined belongs to Topkapi Palace Museum collection.

Identification of an art object material of cultural heritage had received significant attention, because of its importance for the development of appropriate restoration and conservation strategies. Natural dyes have advantages since their production implies renewable resources causing minimum environmental pollution and has a low risk factor in relation to human health. Some of natural dyes are used by pharmaceutical industry as a basis for drug products and by the food industry [1]. The identification of dyes is one of the most important targets aimed for in the scientific examination of paintings, textiles [2], illuminated manuscripts and other historic and archaeological materials. Thus, several analytical techniques have been used, for example thin layer chromatography [3], high performance liquid chromatography [4] gas chromatography/mass spectrometry, UV-visible spectrometry [4-8] reversed phase liquid chromatography and capillary with electrospray mass spectrometric detection, FTIR spectroscopy and Raman spectroscopy [9]. These techniques, high performance liquid chromatography (HPLC) using a diode-array detection (DAD) is ideally suited to the identification of dyes sampled from museum collections especially [10].

Characterization of metal threads on historical textile materials is important for preservation of valuable cultural heritage. Obtained results dictate decisions on cleaning, conservation and restoration steps[11]. Scanner electron microscope energy disperse X-ray (SEM-EDX) has used most commonly method analysis of the metal threads in the textile in the museum collection [12].

The CIEL*a*b* (1976) - system was introduced to describe colour as a result of these three factors. This system is a three-dimensional space, with coordinate axes L*, a* and b*. L* denotes the brightness of the colour (L*=0: black, L*=100: 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 colour can be represented as a set of values for L*, a* and b*, and consequently as a point in this colour space [13].

EXPERIMENTAL

In this work, two caftans were examined belongs to Topkapi Palace Museum collection (Fig. 1-2).

Figure 1. Caftan of Prince (Inventory Number 13/738).

Figure 2. Caftan of Prince (Inventory Number 13/739).

The caftans were dated that one 16th century (Inventory No. 13/739) and other 18th century (Inventory No. 13/738) by art historians. But, designs of the caftans have been same features (characteristics). Many analytical techniques were used for determining the periods of the caftans. One of these techniques is high performance liquid chromatography (HPLC).

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

HPLC Instrumentation

Chromatographic measurements were carried out using an Agilent 1200 series system (Agilent Technologies, Hewlett-Packard, Germany) including G1322A Degasser, G1311A Quat pump, G1329A autosample, G13166 TCC, G1315D Diode Array Detector. DAD detection is performed by scanning from 191 to 799 nm with a resolution of 2 nm and the chromatographic peaks were monitored at 235, 255, 268, 276, 350, 491, 520 and 580. Column: A Nova Pak C18 analytical column (3,9 x 150 mm, 4 µm, Part No WAT 086344, Waters). Analytical and guard columns were maintained at 30 ºC and data station was a Agilent Chemstation. Two solvents were utilized for chromatographic separations of the hydrolyzed samples. Solvent A: H_2O - 0.1% TFA and solvent B: CH_3CN- 0.1 % TFA. The flow rate was 0.5 mL/min. and following elution program was applied (Table 1).

Table 1. HPLC analysis is performed using the gradient elution.

Time (min.)	Flow rate (mL./min.)	H_2O - 0.1% TFA	CH_3CN- 0.1 % TFA
0.0	0.5	95	5
1.0	0.5	95	5
20	0.5	70	30
25	0.5	40	60
28	0.5	40	60
33	0.5	5	95
35	0.5	5	95
40	0.5	95	5
45	0.5	95	5

EXTRACTION PROCEDURE FOR HPLC ANALYSIS OF FIBERS IN THE HISTORICAL TEXTILES

The extraction of historical textile samples were performed with a solution mixture of %37 HCl:MeOH:H_2O 2:1:1; v:v:v) for 8 minutes at 100 oC in open small tubes to extract dyestuffs. After cooling under running cold tap water, the solution was evaporated just to dryness in a water bath at 65 ^oC under a gently stream of nitrogen. The dry residue was dissolved in 200 µl of the mixture of MeOH:H_2O (2:1; v:v) or 200 µl dimethyl formamid (DMF) and was centrifuged at 4000 rpm for 10 min. 50 to 100 µl supernatant was injected into the HPLC apparatus.

HPLC with diode array detection (HPLC-DAD) method was utilized fort he identification of dyestuffs dyed samples with different dye insect and different dye plants which are the most important natural dyes found in historical textiles. Dyestuffs in the caftans examined in this work were detected using HPLC-DAD (Figure 3-11).

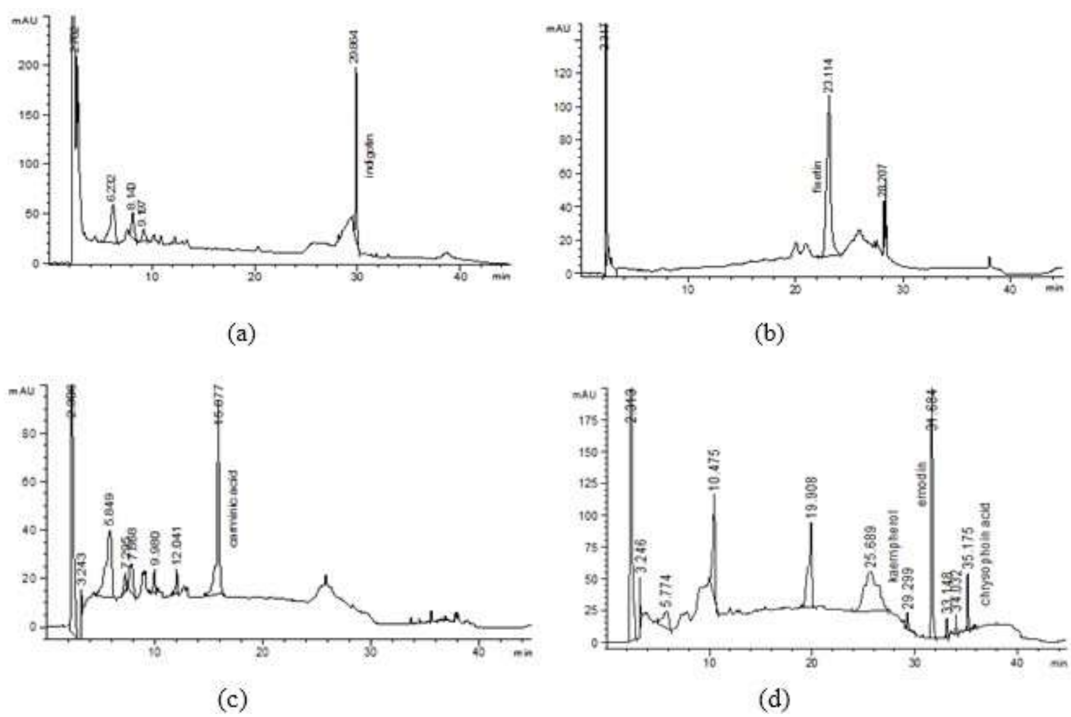

Figure 3. HPLC chromatograms of historical art object (Inventory number 13/738). (a) blue sample, (b) yellow sample , (c) red sample and (d) green sample.

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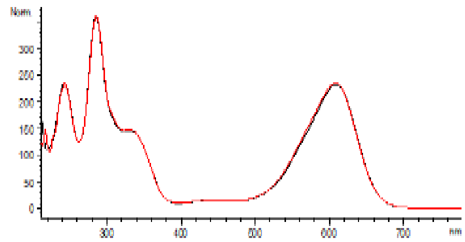

Figure 4. Spectra of blue sample (Inventory number 13/738), spectra of sample together with indigotin standard.

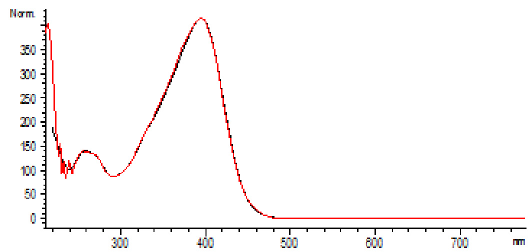

Figure 5. Spectra of yellow sample (Inventory number 13/738), spectra of sample together with fisetin standard

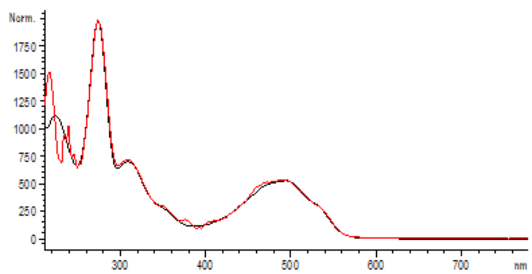

Figure 6. Spectra of red sample (Inventory number 13/738), spectra of sample together with carminic acid standard.

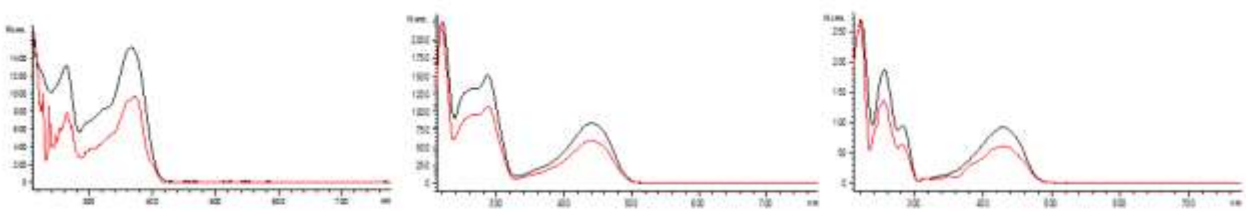

Figure 7. Spectrum of green sample (Inventory number 13/738). (a) spectra of sample together with kaempherol standard, (b) spectra of sample together with emodin standard and (c) spectra of sample together with chrysophoin acid standard.

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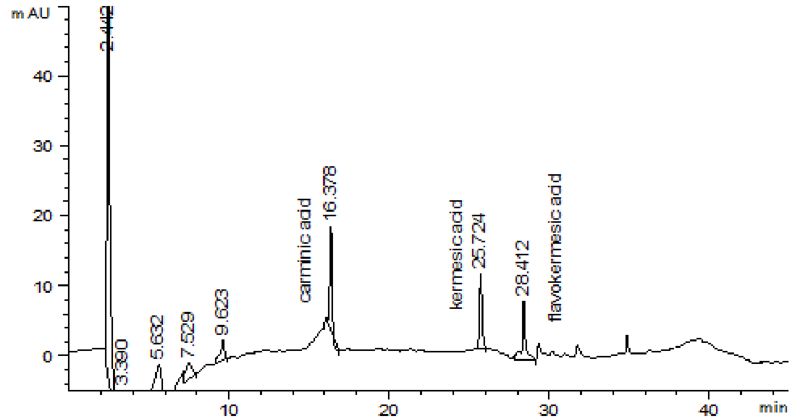
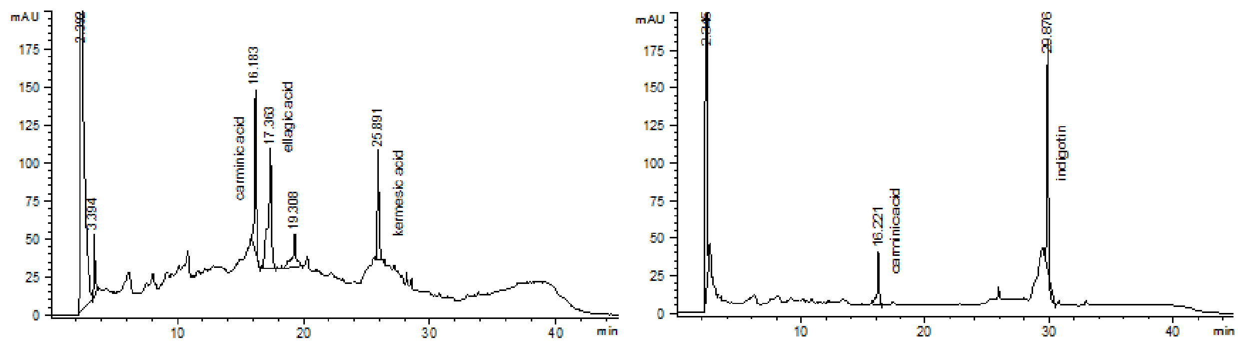
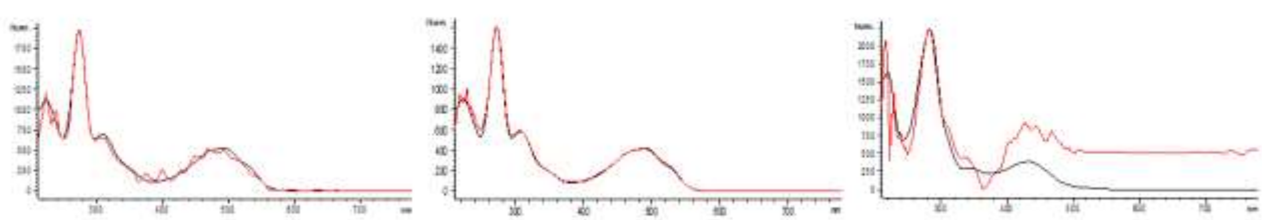


Figure 8. HPLC chromatogram of the red sample of historical art object (Inventory number 13/739).



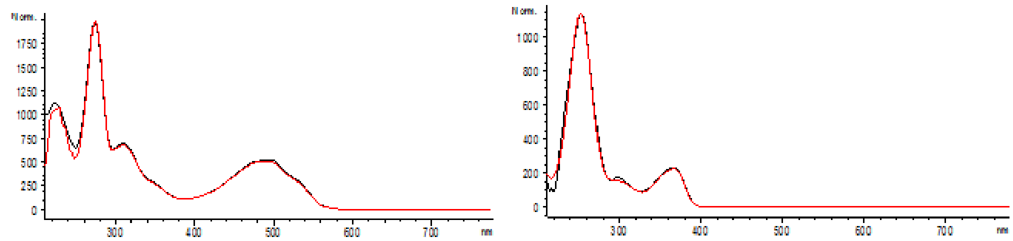
(a) *(b)*

Figure 9. HPLC chromatograms of the purple sample of historical art object (Inventory number 13/739). (a) chromatogram of 255 nm and (b) chromatogram of 276 nm.

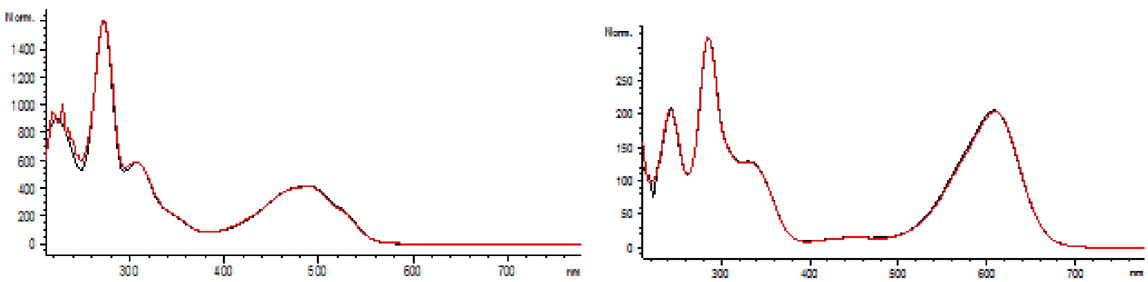


(a) *(b)* *(c)*

Figure 10. Spectrum of red sample (Inventory number 13/739). (a) spectra of sample together with carminic acid standard, (b) spectra of sample together with kermesic acid standard and (c) spectra of sample together with flavokermesic acid standard.



(a) (b)



(c) (d)

Figure 11. Spectrum of purple sample (Inventory number 13/739). (a) spectra of sample together with carminic acid standard, (b) spectra of sample together with ellagic acid standard, (c) spectra of sample together with kermesic acid standard and (d) spectra of sample together with indigotin.

SEM-EDX Analysis

One of the most useful procedures for fast and simple determination of specific metals of interest is scanning electron microscopy equipped with EDX detector (SEM-EDX) since SEM-EDX was a simple and non-destructive method which provided information on chemical composition of sample surfaces and chemical analysis of metal threads.

In this work, the samples were investigated using a TESCAN EasySEM Scanning Electron Microscope (SEM) equipped with energy disperse spectroscopy (EDX with detector Bruker X-Flash 410-M). Metal fibers collected from historical textile materials were characterized (Figures 12-13 and Tables 2-3).

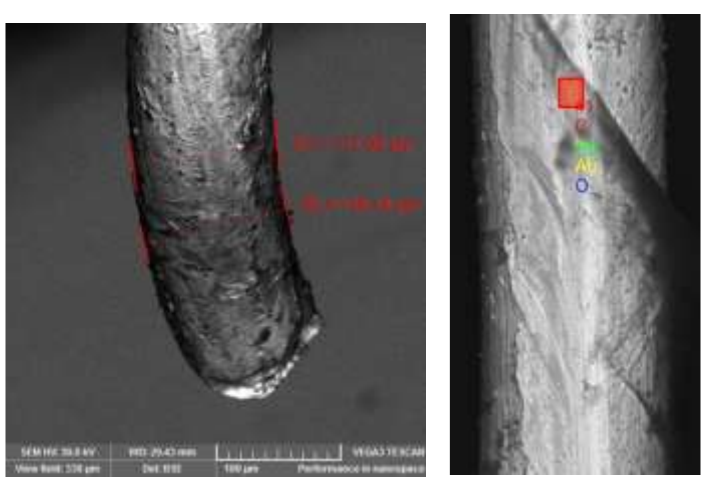


Figure 12. The SEM- EDX images of historical textile (Inventory number 13/738).

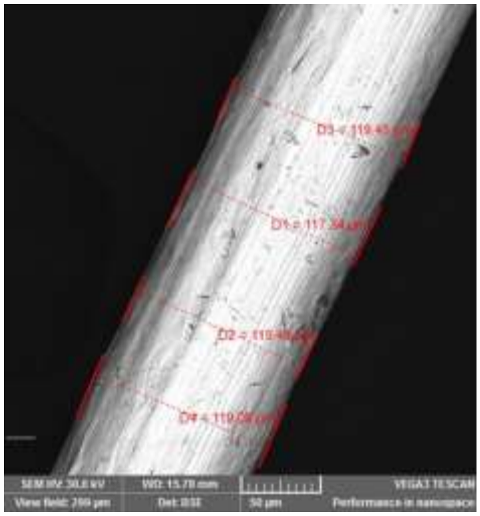


Figure 13. The SEM- EDX images of historical textile (Inventory number 13/739).

Table 2. The metal analysis results of historical textile (Inventory number 13/738).

Inventory number	Identified Elements	% Wt	% At
13/738	C (K)	35.76	74.83
	O (K)	9.42	14.81
	Ni (K)	0.31	0.13
	Au (L)	23.43	2.99
	Ag (L)	31.07	7.24

Table 3. The metal analysis results of historical textile (Inventory number 13/739).

Inventory number	Identified Elements	% Wt	% At
13/739	C (K)	22.55	73.92
	O (K)	3.16	7.78
	Au (L)	53.43	10.68
	Ag (L)	20.87	7.62

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Colour Measurement and Technical Analysis

L*, a* and b* values for historical textiles and reproduced silk brocades were measured with Konica Minolta CM-2300d Software Spectra Magic NX (6500 K, 45º). CIELAB graphs and L*, a* and b* values were shown Figures 14-15. Optical microscope is used for fibre characterization of historical textiles. In this work the historical samples were investigated using a OLYMPUS SZ61 (SZ2-ILST, Camera C18U) (Figure 14,15).

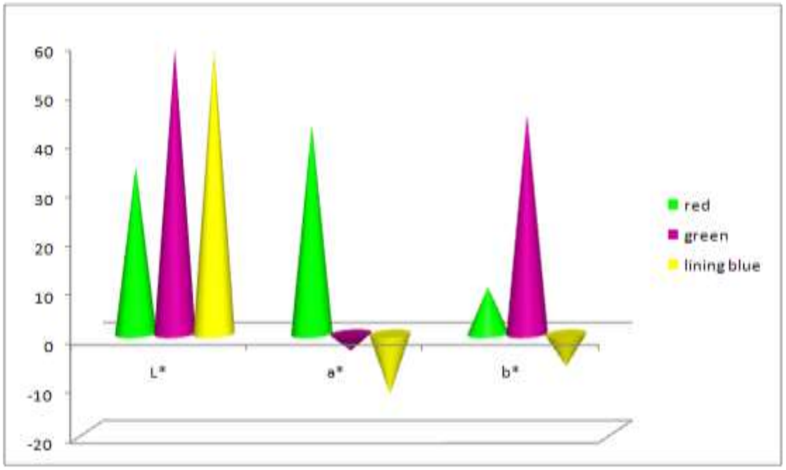

Figure 14. The CIEL*a*b graph of historical art object (Inventory Number 13/738).

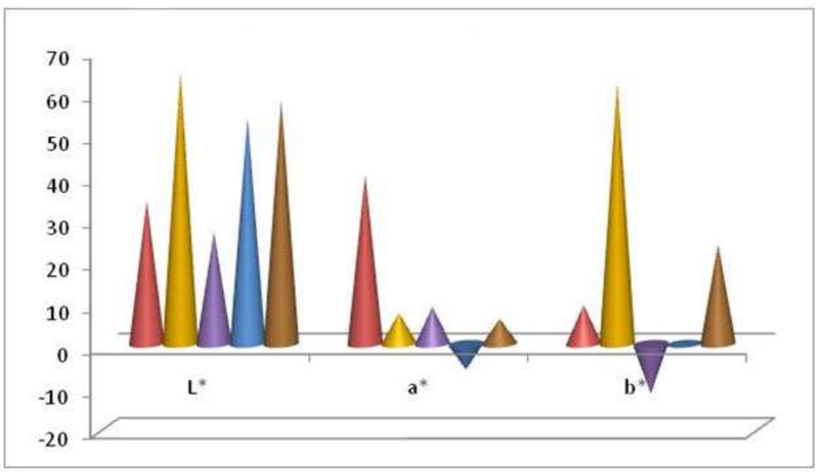

Figure 15. The CIEL*a*b graph of historical art object (Inventory Number 13/739).

RESULT AND CONCLUSION

Ottoman textiles reached their peak in the 16th century. The wide use of textiles in furniture, home textiles and clothing displayed the wealth of the Ottoman court. While Ottoman textiles were produced in multiple locations, Istanbul and Bursa were the only centers to supply the Topkapi Palace (Ottoman court). Textiles produced for the palace were subjected to strict quality controls.

While numerous museums around the world hold Ottoman textiles in their collections, the Topkapi Palace Museum has the largest and most comprehensive collection in the world. These textiles often represent the highest form of cultural, artistic and socio-economic discoveries of their time periods. Information embedded in these textiles, therefore, is not only important to Turkey's textile sector, but is of utmost value for the world's textile heritage.

Textiles were the cornerstone of the art of Ottoman fashion and clothing. Embellished textiles of highest quality made for the sultan for special occasions functioned as status symbols.

The samples belong to two caftans were provided from Topkapi Palace Museum collection. The caftans were dated that one 16th century (Inventory No. 13/739) and other 18th century (Inventory No. 13/738) by art historians (Figures 1-2). But, designs of the caftans have been same features (characteristics). In this work, the dye analysis, the technical analysis, the elemental analysis and colour measurements were analyzed for two caftans.

According to HPLC-DAD analysis dyestuffs and their biological sources in the inventory number 13/738 and 13/739 are shown Tables 4-5. Main coloring compounds are detected.

Table 4. Table of the dyestuff analysis result of historical textile (Inventory Number 13/738).

Inventory Number	Colour of Sample	Identified Dyestuffs	Biological Source
13/738	blue	Indigotin	Indigofera tinctoria or Isatis tinctoria
	yellow	Fisetin	Cotinus coggygria SCOP
	green	Kaempherol Emodin Chrysophoin acid	Rhamnus petiolaris Boiss
	red	Carminic acid	Dactylopius coccus Costa or Porphyrophora hameli Brand

Table 5. Table of the dyestuff analysis result of historical textile (Inventory Number 13/739).

Inventory Number	Colour of Sample	Identified Dyestuffs	Biological Source
13/739	Purple	Ellagic acid Carminic acid Kermesic acid Indigotin	Quercus infectoria Olivier or Quercus ithaburensis + Kermes vermilio + Indigofera tinctoria or Isatis tinctoria
	Red	Carminic acid Kermesic acid Flavokermesic acid	Porphyrophora Polanica L.

SEM-EDX method is utilized for the identification of metal threads for two caftans. The surface morphology and chemical composition historical the caftans were investigated by scanning electron microscopy (SEM) equipped with energy dispersion spectroscopy (EDX). The most important part of characterization is chemical and physical analysis of originally applied materials, since this enables understanding the nature of chemical and physical degradation and helps to determine the proper restoration and conservation methods. One of the most useful procedures for fast and simple determination of specific metals of interest is scanning electron microscopy equipped with EDX detector (SEM-EDX) since SEM-EDX was a simple method which provided information on chemical composition of sample surfaces and chemical analysis of metals threads.

According to elemental analysis by SEM-EDX, metal threads are not alloys that all metal threads are gilded. Au gilded was dedected on metal threads. Contaminants in analysis results were detected in metal threads. These contaminants are S, Cl, Mg, C, O, etc. The metal threads are 5-7 micrometers thick and 115-119 micrometers wide in the caftans that wide in the 16th century while 14-17 micrometers thick and 270-580 micrometers wide in the 18th century.

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Inventory number 13/739 and 13/738 are same period (16th century). 13/739 can be dated first quater of 16th century. 13/738 can be dated second half of 16th century.

According to colour measurement results, discoloration were seen in location subject to exposure to light of historical textile expecially blue, green and purple colours. Colour differensis is very very slight in main colour (red) in two caftans.

According to technical analysis results by optical microscopy, twist (spun) metal threads are S, warps direction are Z, wefts direction are Z. As can be from the results of technical analysis similar for two caftans.

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www.turkishculturalfoundation.org, www.tcfdatu.org .

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