Characterization of Dyeing Techniques of Late 19th Century Ottoman-Style Costumes from the Jordanian Museum of Cultural Heritage

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A collection of Ottoman-style costumes belongs to the late 19th century exhibited at the Jordanian Museum of Cultural Heritage were analyzed using SEM-EDX to identify types of fibres and mordants. The study aims to obtain more and comprehensive knowledge about these costumes completing a previous study that was carried out earlier to identify types of dyes applied. The article also attempts to establish an overview of the dyeing techniques used in late 19th century in the Middle East, specifically in the Levant region which was part of the Ottoman Empire at that time.

Keywords: Ottoman textile; dyeing, recipes, Levant region, Jordanian Museum

In the Ottoman period, dyeing craft witnessed variable levels of dyeing production and importing. The dyeing industry depends on the number of dyeing workshops, which are special buildings with their personnel of dyers, washer men and scribers [1-3]. The most impressive changes in the Ottoman textiles production at the end of the 19th century have been made by the small workshops and the homemade production not the big factories, Many cities were famous in dyeing, such as: Aleppo and Damascus in Syria, Mandar, Maras, Trabzon and Diyar Baker in Anatolia (Turkey), Jerusalem in Palestine, and Baghdad and Musil in Iraq [4-7].

Local Ottoman sources had supplied most dyestuff from natural sources until the discovery of synthetic dyes during 1850s and 1860s in Europe [3, 6]. Synthetic dyes had enormous consequences on Ottoman textiles producers. The impact on natural dyestuffs production and usage was impressive; they disappeared from the world market within decades. Many of the dyestuffs were grown wild or only as a spices by First World War. But even then, Ottoman textiles makers become familiar with synthetic dyes. They also increasingly imported plain yarns and dyed them locally [6]. The Ottoman entrepreneurs had created distinguished fashions and styles for the local market, making it difficult for the Europeans to compete, because they involved an extensive use of embroidery low-wage and intensive decoration that couldn't be replicated [2, 4, 5, 8-10].

The beginnings of the 20th century witnessed the end of the Ottoman power and the Ottoman Empire broke down. The textiles industry including dyeing fell down gradually, and lost its unique styles on the expense of the regional local costumes. Finally, the dyed textiles were recognized as objects with historical value, and found their ways to the museums [11-15].

Despite of quite informative history recorded in the literature regarding dyeing industry and trading, little information in written form is kept, or documented about the details of dyeing techniques, methods and recipes used during the Ottoman era. This study aims to shed light on the traditional dyeing craft and its techniques during the late 19th century in the local regions in Levant area. The integrative approach combines analytical and historical data to characterize the basic and essential dyeing steps in which were applied on the costumes under study.

Historical review

The study mainly depends on two references written in Arabic after the second half of 19th century to provide data that will contribute, along with the elemental and spectroscopic results, in characterizing the basic dyeing techniques that were used on fabrics of that period of time.

The first source is an Arabic Book (1862) entitled*Al dur almaknun fi alsana'e wal funon* (The Hidden Secrets in Arts and Crafts) [16], it contains a complete whole chapter with detailed and rich information about types of natural colorants (dyes and pigments) and recipes including techniques of dyeing methods as well as other elements evolved in the process like types of mordants and additives.

The second source is a well known periodic Arabic magazine issued at that time and stopped in the middle of 20th century called al Muktataf (The Extract) [17, 18] contains, casually, some dyeing recipes or articles related dyes and dyeing industry. Also several researches are focused on analysis of ottoman textiles [19-23].

Samples and Methods

The collection

The samples were taken from the Ottoman collections costumes & exhibited at the Museum of Jordanian Heritage (fig. 1). Very few and small samples of millimetres length from already damaged parts, mostly from lower or side edges, were taken (fig. 2). The samples are listed in table 1.

Fig. 1. The blue dress and black cloak

Fig. 2. Damaged part from brown cloak

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Table 1
DETAILS OF SAMPLES TAKEN

NO.	NAME AND DESCRIPTION OF THE COSTUME	SAMPLES	DYE(S) DETECTED EARLIER
1	Black Man's cloak, thought to be woven from wool sheep, and embroidered with planetary decorations of golden threads, along the chest to the end of the piece, on both sides.	A - Blue fibres were taken at the end of dress. B - Small portion of the red lining was also taken (0.5 x 0.7 cm^2).	Indigo, tannins, Synthetic Alizarin
2	Man's cloak, thought to be woven from wool sheep, embroidered with golden threads on the shoulders and along the cloak in tiny lines.	A - Bright brown sample from the shoulder. B - Yellowish-brown threads from the chest.	Natural brown wool, Madder
3	Blue woman dress, made of silk fibres velvet, embroidered with heavy "crusty" golden and silver metallic threads.	A - Blue fibres were taken at the end of dress. B - Small portion of the green lining was also taken (0.5 x 0.7 cm^2).	Prussian blue, Indigo, White Lead based pigment
4	Green Woman dress, thought to be woven from silk fibres velvet, embroidered with very refined and smooth golden threads all over the cloth.	A - Small deep green portion (about 0.5 x 0.5 cm^2) from the bottom of the dress. B - Small red portion from the lining (about 0.2 x.4 cm^2).	Arsenic-Copper based pigment, Indigo, saffron, Turkey red

Experimental part

Samples of fibres were studied under Scanning Electron Microscope (SEM) coupled with an energy dispersive x-ray spectroscopy *EDX*. SEM is used to observe any surface characteristics that could indicate the fibre type (e.g., cotton or wool). EDX is used for elemental analysis to identify any possible inert materials from mordants (table 3).

The samples were pure gold-coated using sputtering method, under conditions 1200v, 20mA, 105 seconds, in a Polaron E6100 vacuum coater. Samples were viewed in a FEI Quanta 200 SEM, equipped with EDX analyzer. The collection conditions were 15 KV, 90 sec. Area scanned was full fibre width at about 500-4000× magnifications, by back scattered electrons (BSE). The samples were analyzed without washing or cleaning, they appeared to be clean, unsoiled, without staining or badly faded.

Results and discussions

Fibres and Mordants

The feasibility of defining the types of fibres and mordants taken from the costumes have been explored successfully by SEM spectroscopy (table 2), each type of fibre has a special morphology that can be easily identified under certain magnification (fig. 3).

The presence of metallic elements has been detected using the EDX to predict types of mordants. It was reported that the indication of the presence of the mordant could be identified upon element: sulfur ratio [24-32]. For Alum if the ratio of Al/S is 2:1 then the mordant is present, while ratios of 1:1 or 1:2 mean that mordant may be possibly present or probably absent respectively, as to iron mordant if the Fe/S ratio more than 2:1 or 1:1 indicates either the presence or the absence of the iron mordant respectively.

Table 2
TYPES OF FIBERS DETECTED BY SEM SPECTROSCOPY

Samples number	Name of sample	Colour	Type(s) of fibre
1a	Man's cloak	Black	Cotton
1b	Black cloak lining	Red	Silk
2a	Man's cloak	Brown	Wool
2b	Man's cloak	Yellowish Brown	Synthetic fibres
3a	Woman dress	Blue	Cotton and silk
3b	Blue dress lining	Light Green	Cotton
4a	woman dress	Green	Cotton and silk
4b	Green dress lining	Red	Cotton and silk

The data in table 3 represent the types of elements detected in the samples by EDX that might act as an element-based mordant. From the obtained elemental data analysis and according to Koestler approach, it can be concluded that the black cloak was mordanted with iron salts (ratio of 2.25:1.0); the yellowish fibres of the brown cloak (1.8:1.0) were mordanted with ferrous salts. Despite of the very high relative ratio of copper in the green woman dress (8:1) this is due to the Arsenic-copper based pigment, copper sources might used as mordant. The red lining of the green dress might have been mordanted with Alum (1:1). The high amount of iron which exists on the blue dress is due to Prussian blue dye not from Fe based mordant, since Fe element is a major constituent for Prussian blue. The green lining of the blue dress was mordanted with alum (2.7:1.0).

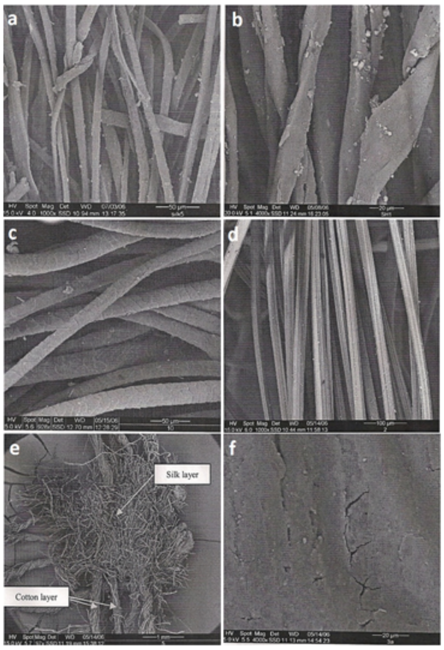

Fig. 3. SEM Images: a - Cotton fibres of black cloak; b - Silk fibres of the red lining; c - Wool fibres of brown cloak, d - Synthetic fibres, e - Layers of the blue dress, f -Dye/pigment matter

Sample wt% element	Sample number	Colour	Al	Fe	Cu	Cr	S
Mans cloak	1	Black	0.51	**1.37**	---	---	0.61
Mans cloak	1a	Red	0.32	0.22	0.00	0.08	0.58
Mans cloak	2	Brown	0.09	**0.29**	0.64	----	1.74
Mans cloak	2a	Yellowish Brown	0.78	**4.06**	1.04	---	2.29
Woman's dress	4	Blue	0.74	4.21	0.08	----	0.34
Dress lining	5	Light Green	**4.69**	2.74	0.95	0.1	1.71
Woman's dress	3	Green	0.29	0.12	**2.82**	**0.44**	0.34
Dress lining	3a	Red	**0.28**	----	----	0.03	0.27

Table 3
ELEMENTS DETECTED IN THE REPRESENTED SAMPLES

Dyeing techniques

Based on the identification of types of fibres and mordants used in the costumes as well as the types of dyes that were identified previously for the same collection, identification of possible dyeing techniques used on the understudied costumes can be done in accordance with the available referred historic sources.

The SEM image of the black cloak fibres appears clearly to be cotton (fig. 3), in contrast to the museum guide that mention the cloak is made of sheep wool. Ferrous salts source were added as mordant as well as a probability of the presence of alum salts as the EDX result refers. To obtain the black indigo-based colour a specific method of dyeing is needed, involves the addition of tannins as well ferrous salts as mordants to give the blue colour deeper and darker shade. Old rusty iron objects may have been used as a source of ferrous salts, which is easy to get at that time in the small dyeing workshops. Cotton is immersed and fermented in acidic water path with some additives like wheat (in form of grains, flour or dough) to accelerate the fermentation process. Cotton is fermented in this mixture before dyeing up to 45 days according to references. Tannins solution usually added after soaking and squeezing out the cotton bulk or yarns the under warm water at about 1:1 ratio, this step might be considered as a second stage of mordanting. Leaving cotton with tannins enough time in the water bath to complete absorption of tannins, cotton then taken out and dried by subjecting to air. The presence of indigo as the main dye suggests the use of one of the *Indigofera* species. To obtain the black-blue combination colour by indigo means the use of vat dyeing method in which the cotton yarns must have dipped in a water-soluble yellowish *leuco* form, then hung up to be oxidized in open air. Due to the presence of tannins and ferrous mordants the blue colour turns into deeper dark shade. If the desired results not obtained, the already dyed cotton simmered in iron oxide and tannin solution several times till obtaining the black hue.

The process of gaining black colour hue by indigo is difficult, take more time and effort than the ordinary black dyes, and cost more. The alizarin red lining of the black cloak appears to be made of silk (fig. 3b), mordanted with alum and dyed mainly with synthetic alizarin as indicated before. The use of silk fibres as a lining, cotton for making the cloak rather the ordinary weaving from wool, also gaining black by indigo vat dyeing process, refers that this cloak was designed for a wealthy man with high social rank in his committee or tribe, since the more steps, labour and time involved, the more complexity and color the fabric would have, the more expensive it would be.

The brown cloak was made of natural brown wool (fig. 3c) - which was unexpected result, since it needs a lot of effort to collect the brown sheep wool, due to their scarcity. The yellowish brown decorative fibres are made of synthetic fibres and are most likely to be viscose fibres (Fig. 3d), which are cellulose fibres immersed in an alkali solution. The reason of the yellowish brown shade of the madder dyed fibres is due to the very high relative amount of Fe element, according to EDX results, indicating the use of ferrous salts to give this hue for the madder-based dye fibres.

The SEM scanning image for the blue dress appears to be made of two layers. The inner layer of cotton, and the soft velvet-like outer layer of silk, composes a big thread of cotton as wrap and silk piles as weft (fig. 3e). The SEM scanning also reveals a combination of intensive dye/ pigment matter composed of Prussian blue pigment mixed with indigo that gives the dress its dark blue hue in addition to the bright luminescent appearance (fig. 3f).

According to historical reference [16] dyeing methods with Prussian blue for cotton and silk yarns are different only in mordanting process. The scoured silk is immersed in 5% Ferro hydrochloride solution, then, removed, and washed in soapy water at boiling point. Then washed and immersed in a cold acidic solution (hydrochloric acid or sulfuric acid) of Potassium cyanide for 15 min. then washed and dried. Cotton yarns are mordanted before dipping in the Ferro cyanide bath solution. Indigo vat dyeing might have occurred before the Prussian blue dyeing step, in order to have a preliminary judgment for the level of the blue colour obtained.

The mechanism of gaining the blue colour is by attaching the ferrous ions to the fibres, leaving the acid ions to be neutralized by soapy water. sulfuric or hydrochloric acid is reacted with potash ions, while the hydro cyanide group is attached with the ferrous ions, which exists on the fibres to form the cyan ferrous group which reflects the deep blue colour.

The chrome-based green lining of this dress might be obtained by mixing Prussian blue and lead white ($PbCrO_4$), to form what it was called chrome green. This technique was mainly used in paint as a pigment and was rarely used as a dye source for textiles. It was manufactured by adding equal amount of saltpetre KNO_3 and potassium tartar in a hot crucible. Dry powdered cattle blood was added and the mixture was heated, then washed with water and treated with a solution of alum and ferrous sulphate. A green precipitate will form and turns blue with addition of hydrochloric acid.

The SEM-EDX data analysis of the Arsenic-based green dress's sample shows the existence of two types of fibre; cotton as wrap and silk as weft. The Silk weft contains of 0.44 % Wt Chrome, while the wrap cotton sample shows no sign of chrome. Chrome salts might used as mordant for silk yarns, as for cotton there is weak evidence for using mordant. The whole sample fragment contains average amount of %Wt 4.47 cobalt, indicating the probability of using cobalt source as additional second addition of mordant to the whole piece of fabric. There is a strong possibility that the dyeing process for the silk and cotton yarns was carried out separately, after weaving the fabric another step of vat dyeing with indigo plant species might have been occurred. The other possibility is to have the horizontal fibres dyed with arsenic chrome yellow, while the vertical fabrics are copper-arsenic based pigment, and all of the fibres were over dyed with indigo, additions of small amount of saffron might have been occurred as well as the addition of Arsenic-copper based pigment perhaps by brushing. G. Tannos, in 1862 mentioned a dyeing method close to the obtained results: by adding copper sulphate as mordant, Arabic gum as additive, and washing with diluted potash alkali solution the, fabrics then are dipped in an arsenic acid path until complete absorption is taken place, the final step in to clean and squeeze the fabric from excess colour then hang to dry.

The red madder - dyed cotton lining of the green dress has a similar hue to that of the famous *Turkey red* dye obtained from cultivated madder. In the monthly magazine Al-Muktataf [17, 18], a process for dyeing cotton was mentioned for obtaining the Turkey red from madder; by washing and immersing the cotton fabrics in sodium carbonate solution. The mordant process involves the use of Alum or sumac or both. Some additives like olive oil might be used prior to mordant process. After days of preparation the fabric by washing and mordanting the dyeing process starts by dipping the cotton fibres in powdered madder solution containing some additive like calcium and blood [33]; the final step is to add some sodium carbonate and calcium chloride to remove the yellowish-red matter and purify the red colour obtained.

Conclusions

Obtaining precise dyeing recipes identical to the traditional ones, based on collected data, is fairly difficult. This is due to the fact of unrecorded information of the exact details during the dyeing process. Since dyeing industry, historically, was considered a highly skilled craft inherited from father to son through generations as trade secrets, the loss of many of these small “secrets”, but yet crucial, over time was possible.

Some descriptions of dying process in the referred historical sources may provoke questions rather than providing answers. Such as the *form* of textiles to be dyed (is it loose fibres, yarns or woven fabrics), exact proportion of textiles to colour and proportion of solution to colour, also time control of boiling, immersing and soaking, number of over dyeing repetitions to obtain certain degree of colour or shade.

Another details that play vital role in properties of colour (like shade, fastness and intensity) like *ageing* and *dunging* in which is applied to firmly fix the dye within the fabric, need to be accurately known. Some *additive* materials are also involved during the dyeing process like tannins, urine, wheat, blood, chalk, milk and egg albumen that act as binders or *catalysts*, as well as the amounts of adding acidic or alkali components to control the pH medium of the dye bath. These practices only come by gained experimental knowledge.

However, some general remarks and notes with the help of both analytical study and historical references of certain period of time can still be obtained to provide essential information that contribute to limit the possible used recipes, giving a glimpse of how local dyers succeeded to integrate traditional recipes with innovative techniques and new colouring materials to adopt, what will become, a very fast growing industry.

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