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Mediterranean Archaeology and Archaeometry
Vol. 20, No 1, (2020), pp. 163-171
Open Access. Online & Print.

DOI: 10.5281/zenodo.3707806

ARCHAEOMETRICAL STUDY OF A RARE EMBROIDERED AND APPLIQUED LEATHER TAPESTRY FROM THE SAFAVID ARTWORKS. PART I: WEAVING FIBERS AND DYES

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Received: 23/02/2020
Accepted: 20/03/2020

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ABSTRACT

The present work introduces a laboratory multi-technique study for a unique heritage object from the Persian Safavid art preserved in Egypt. This study is alleged to be the preliminary one of such heritage embroidered and appliqued leather tapestry. The aim of the present study is to reveal the type(s) of fibers and dyes used in crafting this rare artwork and the deterioration forms as well. The study used the Stereo Microscope (SM), the Scanning Electron Microscope coupled with EDX unit (SEM-EDX), ATR-FTIR and the MALDI-TOF MS. The object is composed of different layers; a textile substrate, a blank leather layer upon the textile substrate, a blackish leather layer, pre-cut white leather patterns appliqued upon the blackish layer using threads, the entire surface is embroidered with dyed threads. The obtained results revealed that the textile substrate is a plain cotton. The embroidery threads are also cotton, dyed with natural indigo, madder and Persian berries. Dyes' mordants were not verified, probably due to the high proportion of dust and soiling matters. The object is suffering from many deterioration forms; namely drying, brittle fabric and threads, faded dyes, lost threads and parts, stains in both the textile substrate and the embroidery threads, and much amount of dust and soiling matters.

KEYWORDS: Natural fibers, Dyes, Textile, Leather tapestry, Embroidery, Investigation, Analysis

1. INTRODUCTION

Weaving, interlacing highly flexible and long or continuous materials, requires suitable fibers and tools, without them the craft can't exist. The natural flexible fibers of wool, silk, hair, cotton, linen were generally cleaned, combined and twisted into long and strong threads. They were used to be colored for value addition by anciently using natural sources of colors/dyes (Barber, 1991). The word 'natural dye' includes all different colors derived from the animals and plants. Due to their non-substantive colors, natural dyes are commonly applied with the help of some metallic salt as mordants, which have an affinity for both the substrate fibers and dyeing matters and. Natural dyes were used in coloring of natural fibers, food substrate, leather, etc. since prehistoric times in different civilizations (Karapanagiotis & Karadag, 2015; Mantzouris et al., 2016; Samanta & Konar, 2011). The early 11th century was the beginning of a renaissance of ancient Iranian art and crafts. Between the end of the 13th and the beginning of the 18th centuries, ancient Iran was progressively ruled by three major powerful dynasties: the Il-Khanids, the Timurids and the Safavids (Shirazi-Mahajan, 1985).

The Safavid Dynasty ruled Persia (ancient Iran and Iraq) from 1501 to 1736. It extended to rule the Mongol in the East and the Ottomans in the West (Savory, 1980; Sefatgol, 2006). The Safavid period was the golden age of textiles, carpets, leathers and related art objects. The 16th and 17th centuries generally witnessed a growing interest in the arts and art collections of the three empires, Safavid, Ottoman, and Mongol (Lukonin & Ivanov, 2013; Talebpoor, 2008). Embroidery, decorating a substrate material with stitching different threads, is an ancient technique used in decorating most ancient objects. It is created using a needle and thread on leather, cloth, or any other flexible material. When stitching or sticking a pre-cut pieces of another material, rather than threads, the technique is called appliqué. It is an ancient brocading technique, that appeared during the 18th Dynasty (1550 to 1292 BC) of ancient Egypt. Tutankhamun's treasures included tapestry weave, loop-weave, appliqué, sewn-on beads etc. These crafts are found in almost each country, but using different materials, methods, ornaments, and styles, according to the national traditions of the country (Dross-Krüpe & Paetz, 2014; Riefstahl, 1944).

Ancient textiles and leathers were embroidered, dyed, colored, ornamented, etc. in many different forms of crafts, e.g. hangings, tapestry, curtains, garments, caskets, saddles, pouches, gloves, boots, shoes etc., preserved in international museums (Dross-Krüpe & Paetz, 2014; Göksel & Kutlu, 2016; Watt et al., 1997). These crafts were developed by applying an upper paint or gilt layer, and using gold or silver threads for embroidery. The most common craft was what so called 'wall hangings' or 'leather tapestries'. These embroidered and appliqued art-works were widespread particularly in ancient China and East Asia around the 10th century. The Chinese technique influenced the West Asian art, specially Persia during the Mongol period (13th-14th century AD) (Moroz, 1993; Watt et al., 1997). With time, the art of leather tapestry had transformed from only hanging on part of the wall to covering a whole wall, especially in Europe. By the mid-18th century AD, leather wall hangings were supplanted by wallpaper (Moroz, 1993; Nimmo et al., 1993; Andersen, 1995).

The case study object (Fig. 1) dates to the 16th-17th century and is related to Persian Safavid art. It is 65 x 97 cm and is preserved in The Cotton Museum in Egypt. It is a unique piece of craftsmanship, as it is more than textile or leather, not only embroidered or appliqued; it is an incredible combination of all of them. It is composed of a plain blank textile substrate, upon which a blank beige leather layer is identified. On top of that, a blackish-colored leather layer is also seen, upon which pre-cut and appliqued leather patterns are sewn to the ensemble. The entire object is embroidered and fixed, with chain stitches using textile threads dyed in different colors. The decorative elements include a Persian female face, four birds, modified plant flowers and leaves, arranged in a symmetric design, all surrounded by three decorated outer frames. The frames' decorative elements are perfectly integrated in the corners. All the decorative elements are corresponding to the ancient Sasanian art, affected by the ancient Chinese art (Watt et al., 1997; Riddell, 1946).

One limitation of this study is due to the lack of relevant bibliographical reference. They were all focused on the separate techniques, namely painted or gilded leather (Moroz, 1993; Nimmo et al.1993; Andersen, 1995), appliqued and metal-threads embroidered leather or textile fabric separately (Dross-Krüpe & Paetz, 2014; Riefstahl, 1944; Watt et al., 1997; Göksel & Kutlu, 2016), that's why, no results concluded from a similar investigated case study objects. Moreover, to the author's knowledge, no similar object is known in other Egyptian museums; there are only few other pieces in the same museum, with no further reference. For this reason, there is no information available for comparison. This study focuses thus on this unique case, providing new data to conservators, archaeologists and historians. Another difficulty lies in the fact that The Cotton Museum in Egypt, where the object is preserved, has no in house archaeologist, conservator, laboratory, or instruments. This museum is owned and managed by

The Egyptian Ministry of Agriculture, and completely free from any material or human resources related to conserving its heritage objects. Moreover, according to the law, there is no way to transport any heritage object out of the Museum for investigation and analysis; it was thus necessary to collect micro-samples, or to use handheld instruments when possible. A relevant study of treatment and conservation of archaeological textile of Coptic period, waved by tapestry technique, has been reported using SEM and FTIR (Amin 2018).

Figure 1. The case study object; (a) Obverse, (b) Reverse.

The Stereo Microscope (SM) and Scanning Electron Microscope (SEM) were physically used to identify the types of textile fibers, while Attenuated Total Reflection-Fourier Transformer Infrared spectroscopy (ATR-FTIR) was used to chemically distinguish between cellulosic and proteinous fibers. EDX unit, coupled with the SEM, was commonly used to identify the inorganic elements in archaeological objects, it indicates the possibility of using mordants to fix dyes. The Matrix-Assisted Laser Desorption/Ionization-Time-Of-Flight Mass Spectrometry (MALDI-TOF MS) was used to identify the natural dyes used in coloring the natural textile fibers in ancient times (Uring et al., 2018; Dellaportas et al., 2014; Cooper, 1987, Talebpoor, 2008; Shibayama et al., 2015, Trojanowicz et al., 2004, Ilharco et al., 1997; Garside & Wyeth, 2003; Kavkler et al., 2011; Armitage, 2011; Amin, 2017; Kramell et al., 2019; Liritzis et al., 2020).

2. MATERIALS AND METHODS

2.1. *Sampling*

Five micro-samples were collected form the case study fibers to identify the fibers' type(s), dyes' source(s), mordant(s) and deterioration forms. The samples collected were; the substrate fabric (SF), the blue embroidery (BE), the orange embroidery (OE), the green embroidery (GE), and the beige embroidery (BiE). All samples were directly investigated using all techniques in the solid state without any preparation or extraction.

2.2. *Stereo Microscope SM*

The study used a Stereo Microscope SM (SMZ800, Nikon) integrated with Canon EOS 700D camera. It was used to identify the anatomical structure of the object layers, using a cross-sectioned micro-sample without moulding in resin. It was also used to observe the object's deterioration phenomena, weaving structure, and brocading techniques.

2.3. *SEM-EDX*

The study used a Scanning Electron Microscope SEM (Quanta 3D FEG, FEI Company, USA) coupled with Energy Dispersive X-ray detector EDX. It was used in the morphological identification of the fibers' type(s) used in the substrate fabric, the embroidery threads, and the deterioration forms in the object's micro-samples. The EDX unit was used to identify the inorganic components and the dyes' mordant(s) when applicable. The operating conditions were: beam energy 20 kV, acceleration voltage with ETD detector (secondary electron mode) at 10 mm working distance and a spot size 5.5 (1kV/10pA) with a scale ranging from 10μm to 50μm. The spot size was 7 (20kV/4nA) with a scale of approximately 200μm.

2.4. *ATR-FTIR*

FTIR spectroscopy (Thermo Nicolet 6700 FTIR spectroscopy, USA) was used in the chemical identification of fibers' type(s) of the substrate fabric and embroidery threads. The spectra were obtained in the reflection mode using ATR crystal in the spectral range from 4000 to 400 cm^{-1} with 4 cm^{-1} resolution at room temperature. The FTIR results is aimed to chemically interpret the macroscopic and microscopic physical results obtained by the SM and SEM (Ebsen et al., 2019; Alexiou et al., 2015). All samples were directly investigated in the solid state without any preparation.

2.5. MALDI-TOF MS

Mass Spectrometer (Autoflex Speed MALDI-TOF-TOF, Bruker) was used to identify the dye(s) probably used in coloring the different embroidery threads. Four 3mm-lenghth fibers (BE, OE, GE, BiE) were directly examined without any processing. The fibers were fixed on steel plate. The instrument was fitted with a standard nitrogen laser (337nm), and calibrated using a commercial peptide mixture MPep. The spectra were acquired in negative mode and processed with Bruker Flex III, Bruker Xtof software and mMass 5.0.1 software.

3. RESULTS AND DISCUSSION

3.1. Macroscopic and microscopic results

The obtained macroscopic and SM microscopic results (Fig. 2) revealed that the object consists of three layers; a substrate blank fabric, an intermediate beige leather, and an upper blackish leather. Pre-cut small white leather pieces are shaped and appliqued upon the three layers. The whole layers and appliques are jointly embroidered, and consequently fixed, with different-dyed textile threads. The substrate blank fabric is a tapestry weave structure (plain weave structure 1/1). The number of warp threads is 12/cm, the number of weft threads is 14/cm, the torsion direction is Z. the dyed threads were used in two different purpose; sewing and embroidering the appliqued patterns to the upper leather layers, sewing and embroidering the whole object layers from obverse to reverse. The chain stitching style was used in the embroidery technique. The embroidery threads were dyed in different colors; dark and light blue, dark and light green, orange, and beige. The object is suffering from many deterioration phenomena, such as drying, lost parts, brittle fabric and threads, faded dyes, stains (in fabric substrate and embroidery threads), dust, and soiling matters (Uring et al., 2018; Dellaportas et al., 2014; Cooper, 1987). No notable microbial colonies were observed on either the obverse or the reverse of the object.

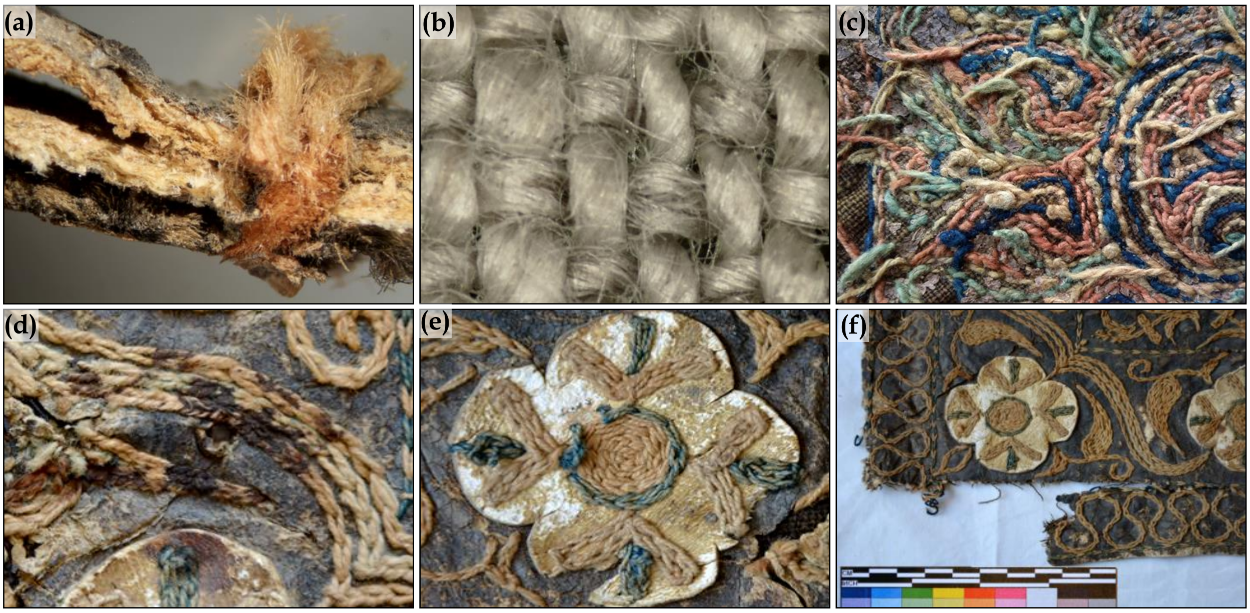

Figure 2. Macroscopic and SM microscopic results; (a) SM micrograph of the cross section of the object layers, (b) SM micrograph of the plain 1/1 weave structure, (c) the SF embroidered with dyed threads, (d, e) stains, brittleness and faded dyes, (f) lost parts

The SEM results (Fig. 3) revealed that all fibers used in weaving the substrate fabric and spinning the embroidery threads are cotton. It was expected to identify the blank substrate as cotton, but it is rare to find dyed embroideries also made of cotton. In ancient civilizations and in Persia, the wool and silk fabric and threads tended to be dyed, not cotton or linen (Talebpoor, 2008; Shibayama et al., 2015). The SEM micrographs reveals the disintegration, deformation, degradation and brittleness of all used fibers, especially those used in the SF sample, may be this substrate was attached to soiling matters in ancient times. A huge amount of accumulation, soiling matters and likely adhesive matters also cover the substrate fabric, maybe due to ancient restoration or adhering the object to a substrate surface, or adhering the substrate fabric to the leather itself. The deterioration phenomena in cotton fibers showed by SM and SEM are likely due to the ill-adapted museum environment in the Cotton Museum, where the conditions of preservation are not stable, and a humidity content is not monitored, the other internal and human factors (Elsayed, 2019; Uring et al., 2018).

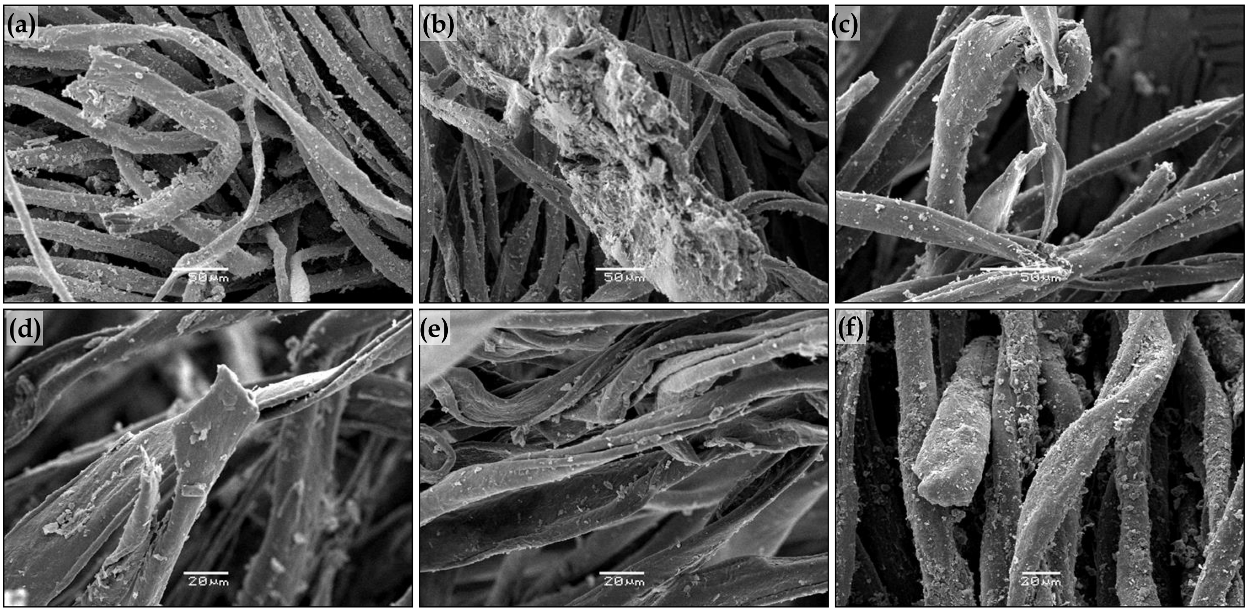

Figure 3. SEM micrographs of the investigated cotton fibers and deterioration forms; (a) SF, (b) soiling matters and adhesive-like deposits in SF, (c) BE, (d) OE, (e) GE, (f) BiE

The results of the EDX unit for the SF, BE, OE, GE, and BiE showed many chemical elements in all investigated samples (Table 1, Fig. 4). Carbon (C), nitrogen (N), oxygen (O), fluorine (F), sodium (Na), magnesium (Mg), aluminum (Al), silicon (Si), phosphorus (P), sulfur (S), chlorine (Cl), potassium (K), tin (Sn), calcium (Ca), iodine (I), and iron (Fe) elements were identified in all samples. The high intensity of C, N, and O are attributed to the major components of cotton cellulose (Uring et al., 2018, (Ibrahim et al., 2017). Other detected elements are interpreted as soot, dust deposits and soiling matters from the Egyptian environment (Elsayed, 2019). All elements are identified in blank and dyed fibers as well, this is thus attributed to the more amount of soiling and dust deposits, compared to the small amount of mordants, consequently, some mordants might have been used to improve the dyes' colors and stability, but could not be verified, e.g., Al, S, K, in in BE, OE, GE, and BiE may refer to alum ($KAl(SO_4)_2.12H_2O$), but once all Al, S, K were identified in both SF investigated samples, this confirmed that no alum used (Uring et al., 2018; Trojanowicz et al., 2004). The main threat caused by dust deposits and soiling matters is the fading of fibers and dyes' colors. It causes and catalyzes the physical and chemical degradation of the structure of dyes and cellulose (Grau-Bové & Strlič, 2013).

Table 1. The analytical results of the EDX unit for the case study macro-samples

	Wt% detected elements															
	C	N	O	F	Na	Mg	Al	Si	P	S	Cl	K	Sn	Ca	I	Fe
SF obverse	19.46	13.67	51.21	2.27	1.64	0.66	1.18	2.85	0.25	0.68	1.55	1.49	0.30	1.42	0.31	1.06
SF reverse	18.43	11.55	46.7	1.52	1.09	1.92	3.13	2.46	1.02	1.54	1.07	3.86	0.8	2.65	0.31	1.95
BE	24.47	14.7	47.62	2.34	1.76	0.47	0.35	0.87	0.28	0.73	2.25	1.07	0.28	2.06	0.30	0.45
OE	23.82	13.09	47.76	1.75	1.91	0.50	1.03	0.97	0.67	1.26	2.08	1.79	0.47	2.09	0.29	0.52
GE	22.27	13.07	52.70	2.77	1.48	0.37	0.26	0.68	0.29	0.80	1.77	1.10	0.23	1.55	0.35	0.31
BiE	18.90	13.90	52.15	3.34	1.83	0.79	1.29	2.94	0.28	0.53	1.12	0.74	0.19	1.06	0.33	0.61

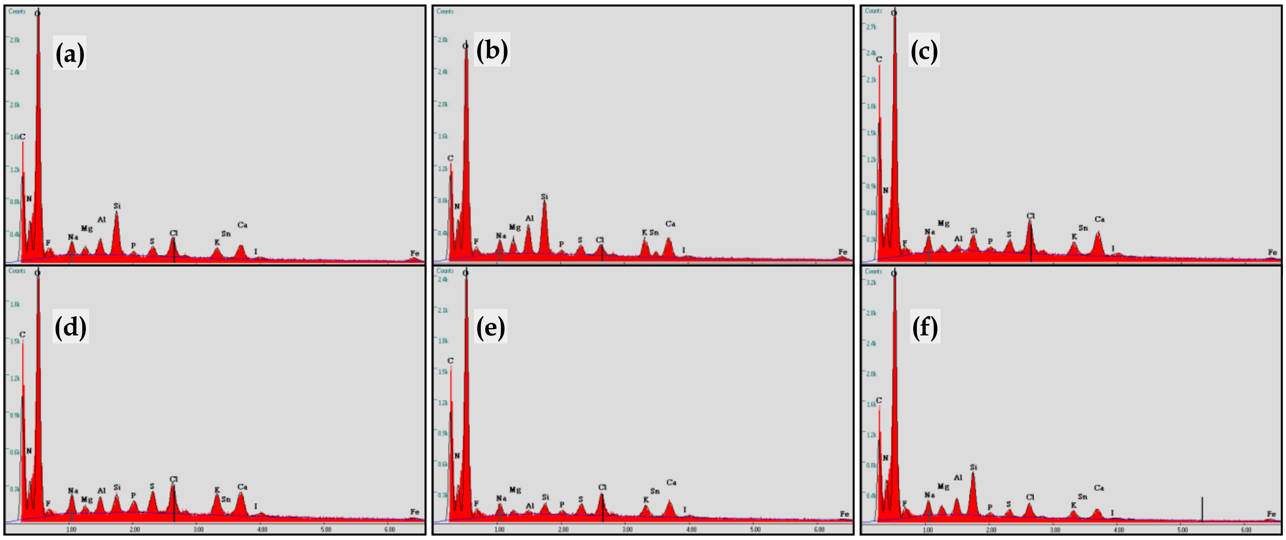

Figure 4. The patterns of EDX results; (a) SF obverse, (b) SF reverse, (c) BE, (d) OE, (e) GE, (f) BiE

3.2. *ATR-FTIR results*

The ATR-FTIR obtained spectra (Fig. 5) revealed that the samples SF, BE, OE, GE and BiE correspond to cellulosic fibers; the vibration modes at ~900 cm^{-1}, ~1375 cm^{-1}, ~1430 cm^{-1}, ~2900 cm^{-1} and ~2995-4000 cm^{-1} are characteristics for cellulose, either crystalline or amorphous. The bands at ~1280 cm^{-1} and 1430 cm^{-1} correspond to crystalline cellulose, especially in cotton spectra. These bands indicate an increased crystallinity of the cellulose in the fibers. The vibration at ~900 cm^{-1} corresponds to the amorphous phase, which represents the higher intensity in the cellulose distorted structure (Kavkler et al., 2011). The increase of spectral intensity at ~968-1050 cm^{-1} could be related to the $_{\nu}$C–O. The C-O-C glycosidic ether band at ~1105 cm^{-1} and C-C ring breathing band at ~1155 cm^{-1} refer to the polysaccharide components (cellulose). Three bands at ~1317 cm^{-1}, ~1337 cm^{-1}, and ~1372 cm^{-1} are of $_{\delta}$HCC and $_{\delta}$COH. The C=C band at ~1595 cm^{-1} and ~1505 cm^{-1} correspond to lignin. The C=O ester band at ~1735 cm^{-1} corresponds to pectin, which might also be strengthened by the carbonyl groups of oxycelluloses in degraded materials. The $_{\nu}$C-H at ~2900 cm^{-1} is a measure of the general organic content of the fibers (Ilharco et al., 1997; Garside & Wyeth, 2003; Kavkler et al., 2011). The SEM results of some fibers' identifications faced some drawbacks, due to the deformation or disintegration of the fibers' morphology, that's why, ATR-FTIR, as the best analytical technique to identify fibers, is commonly used. Moreover, the reflectance-FTIR spectroscopy (r-FTIR) is a more suitable technique to non-invasively identify the textile fibers (Peets et al., 2019).

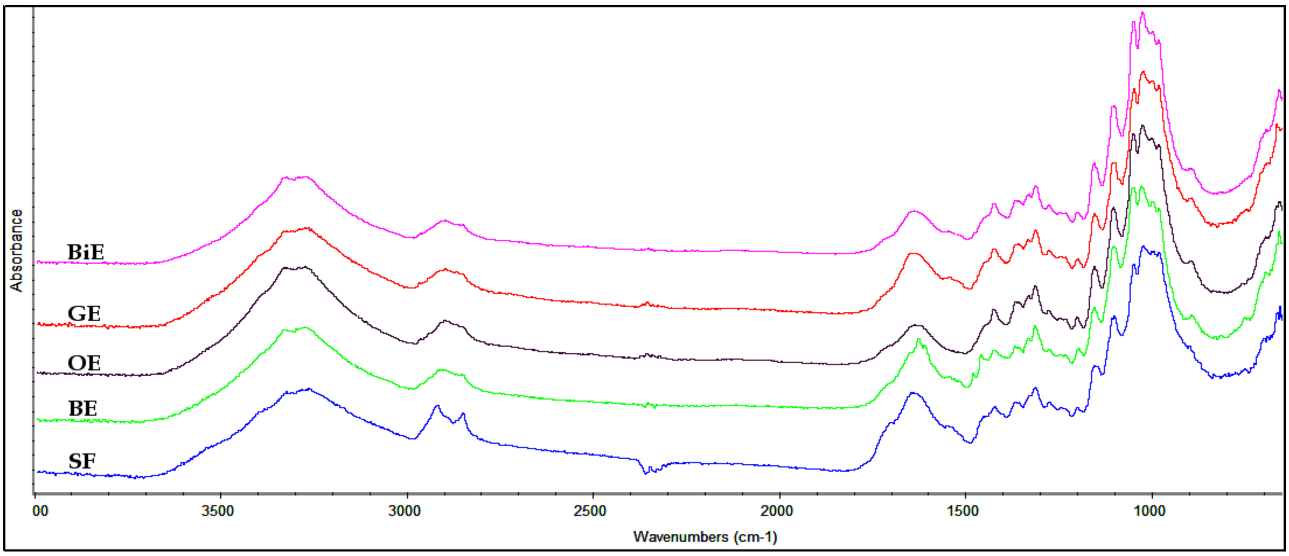

Figure 5. The ATR-FTIR results of the SF, BE, OE, GE, BiE micro-samples revealing that all are cotton

3.3. MALDI-TOF MS results

The MALDI-TOF MS obtained results of the samples BE, OE, GE and BiE, revealed that the BE spectra (Fig. 6a) is attributed to the indigo dye, owing to indirubin and indigotin, the main components of the indigo and woad, identified at 262.17 *m/z* and 263.32 *m/z*. Indigotin is the predominant peak in all textile blue fibers, dyed with the vat process (Kramell et al., 2016; Selvius & Armitage, 2011). It is not possible to chemically distinguish the plant species, which was used in the dyeing process, whether indigo (*Indigoferia tinctoria*) or woad (*Isatis tinctoria*). Distinguishing them is linked only to the historical evidence (Cooksey, 2001). The OE spectra (Fig. 6b) is attributed to the madder dye (*Rubia tinctorum*), due to identifying alizarin (241.96 *m/z*) and purpurin (255.06 *m/z*). Both compose the madder red dye. This means that the sample OE was originally red, but changed to orange due to the natural aging and exposure to light. It is also possible that it might have been originally orange, but the yellow dye remains unidentified (McGlinchey, 1994; Selvius & Armitage, 2011; Kramell et al., 2019). The GE spectra (Fig. 6c) is attributed to a green dye, a mixture of indigo blue dye and Persian berries' yellow dye (*Rhmnus sp.*). Indigotin, the main component of the indigo, was identified at 263.32 *m/z*. Quercetin and rhamnazin, the main components of the Persian berries dye, were identified at *m/z* 302.09 *m/z*, and *m/z* 329.26 respectively. The green dye was commonly obtained by combining blue and yellow ones (Selvius & Armitage, 2011; Degano et al., 2009). The BiE spectra (Fig. 6d) revealed that no dyes had been used. The fiber is suggested to be used blank, as it is without any dyes. The beige color is likely attributed to the pollutants and aging process (Cooper, 1987).

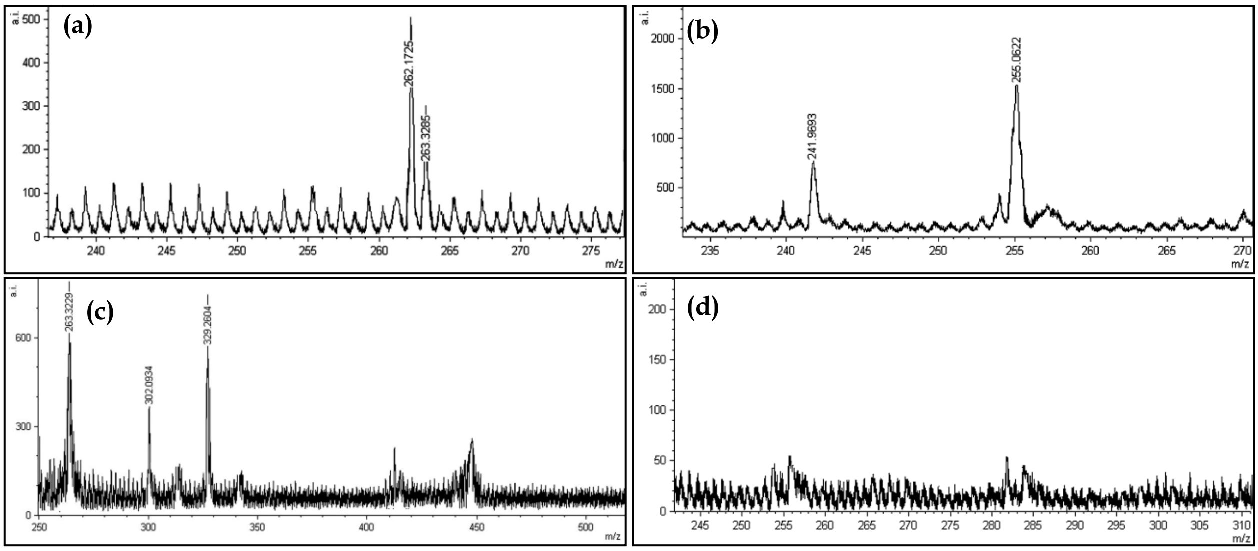

Figure 6. The MALDI-TOF MS results; a BE, b OE, c GE, d BiE samples

4. CONCLUSIONS

The present study focused on the investigation through multiple scientific techniques, in order to analyze a rare case study object. The archaeological and laboratory results revealed that the object is a unique piece dating to the Safavid era, the golden age of art in ancient Persia. The crafting process is not common, either in the materials or the methods used. Due to the textile fabric substrate, the leather layers and appliqués, the painted surface, and the dyed-threads embroidery, it is suggested to name the object as "an embroidered and appliqued leather tapestry". It is as yet unknown the reasons for this object to be crafted; however, it is likely that it was used as a wall hanging.

In the textile crafts of all ancient civilizations, it was very common to use a blank cotton and linen substrate for embroidery works, using silver and golden threads, or using a dyed wool and silk threads as well. In our rare object, it is unique to identify a dyed thread as cotton, rather than wool or silk, which was used in embroidering a leather surface; indeed, it was more customary to embroider this material with gold and silver threads, or wool and silk threads. Natural indigo, madder and Persian berries were used in dying the embroidery cotton threads, while mordants were not verified, due to the identified high proportion of dust and soiling matters.

Due to the potentially deteriorative factors of the ill-adapted museum environment, namely the high difference in temperature and humidity in Egypt during the year, the huge amount of air pollutants, the oxygen-rich and strongly lit display halls, the absence of archaeologists and conservators, all of these factors damaged the object which is in dire need of treatment and preservation. If nothing is done, the ongoing deterioration will pursue, reducing the object until its full decay. The present study highlights this unique object in the small Cotton Museum to attract the interest it deserves, from the responsible authorities, and from the heritage specialists.

ACKNOWLEDGMENTS

The author acknowledges the assistance provided by the Research Infrastructure NanoEnviCz, supported by the Ministry of Education, Youth and Sports of the Czech Republic under Project No. LM2015073. Thank to Prof. Pavel Lejcek, Institute of Physics, Czech Academy of Science, for his help in SEM-EDX analysis.

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