

THE DETERMINATION OF CONSERVATION STATE OF ARCHAEOLOGICAL MOROCCAN KILIM BY PHYSICAL ANALYTICAL METHODS

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Abstract

This paper present the strategies for identification the raw material and dyes used in old textile and deterioration and degradation statement of archaeological dyed samples by using physical techniques such as light microscopy, scanning electron microscope and X-ray diffraction. Samples were taken from morocco kilim which was suffering from several types of damage, as a result of natural ageing appearing in brittle fibres, fading in dyes in many parts of it and missing in a lot of parts in many places. Light microscope and SEM were used to identify the kind of fibres, their condition and surface morphology. XRD was used to identify mordant, impurities and dust. Various dyed samples were taken from different eight sites selected from the worst places in statement of damage in the kilim, and examined by optical microscope, XRD and Scanning electron microscope. Main results revealed that the raw material was wool with its significant morphology appearance. XRD analysis revealed that iron sulphate, Alum, copper sulphate, tin was the main mordant existed in. On one hand, these mordants were considered the most common mordants used in archaeological textiles, on the other hand it should be one of the deterioration and degradation factors for old samples. SEM showed the weakness, brittleness and friction existed in dyed wool samples, the reasons of deterioration/degradation was discussed. Finally, the previous analysis provided a suitable plan for treatment the archaeological dyed textile, as it could be helpful for conservator to have a good decision of the procedure and material should be used in the conservation process based on the previously obtained data analysis.

Keywords: *Deterioration and degradation; Physical techniques; Dyed wool; Archaeological textile; Mordant*

Introduction

Archaeological textiles, as all organic materials, are hardly preserved due to several factors, which caused different types of deterioration and degradation, depending on the nature of the fibres and environment where the objects were kept. So we have to get sufficient information about the state of damage existed in objects, how it was happen [1], by application of analytical methods on dyed samples, taken from different parts of morocco kilim. These techniques enrich conservation scientist information about the conditions of preservation and physiochemical properties of materials, in order to choose the proper treatment methods as well as the conditions of display and storage [2]. Several artistic objects, especially textile, undergo colour modify which changes their appearance which have a significant effect on aesthetic and

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iconographic interpretation of the archaeological textile [3, 4]. So, non-destructive methods have to be used for identifying the type of fibres, the state of deterioration/degradation and kind of mordant used in dyeing process [4]. Some researchers have studied application of optical microscope for characterizing the historical objects [5-7], but we present at this paper different type of optical microscope such as light stereomicroscopy, transmission light microscope and comparison light microscope [8] by which we can distinguish the surface of wool fibres, which used in making kilim, consisting of layers of overlapping scales which is a feature that is not seen in other fibres [9]. In addition, the scanning electron microscope can reveal the details of the wool fibre surface and also indicate to the damage happened in the morphological appearance [10-13]. Also, XRD is used in the identification the type of mordants [14], counting the degree of polymerization for archaeological materials [15], and evaluation the effect of selected conservation materials used in the treatment of museum arte facts [16].

Description and condition

The morocco kilim under investigation, dated to the modern period, is hold by the applied art museum (Fig. 1). It is considered a kind of nonwoven textile called tapestry, as the weft thread didn't pass through loom from the one side to width other, but the artist weaved the dyed thread in the region of the specific figures used in decoration [17]. The kilim was manufactured using the plain weave technique 1/1; it measures 93.5×167.5 cm, divided into twenty four horizontal rows. The kilim is in a bad condition because of embitter, drying and weakness of the wool fibres, which were exposed to light and heat for a long time.



Fig. 1. The deteriorated and degradation morocco kilim before conservation

Samples

Three examples were taken from several parts of the object. Then the samples were examined by three types of optical microscope: stereo-microscope, digital light microscope 2 and comparison light microscope 3 (microscope is a Nikon made in Japan, with lenses No. 83128, Mag. 10 Pi). These techniques are considered non-destructive methods for investigating archaeological wool samples. So we can use the same samples in all techniques without losing them. Moreover, applying X-ray diffraction on the obvious samples to figure out the mordant and purities existed in the samples [18].

Light stereomicroscope

The three samples from the morocco kilim under test were subjected to Stereo- microscopy (Fig. 2). The information obtained regards the type of natural fibres, type of weave, and the degree of colour of the dyed wool used in the fabric, as well as the rate of visual fading [19].



Fig. 2. The stereo-microscope, ASM, Arizona University, USA

Transmission light Microscope

Single fibres from three samples were mounted in 50% glycerine, then fixed in glass strips and covered with another one. After that the samples were examined using light microscope, type Olympus, SZ40, GSWH 10 ×122, Japan with Dino eye Eyepiece Camera, for observing their anatomical characteristic in higher magnification, in addition to detecting the damage incurred by the physical properties of dyed fibres.

Polarizing microscope

The archaeological samples were examined under several wavelengths of light, in order to obtain several figures to the same yarn in different kinds of visible and invisible light [20]. For this task there were used a polarizing microscope Olympus A229922, lens in the range of 530 nm, and a magnification lens 20 po.

SEM examination

A cross section profile of the archaeological fibres was analysed; a yarn (2cm) was cut using a sharp scalpel, and then embedded into cells for examination, in order to ascertain the quality of the wool thread as well as the damage aspects on these fibres.

XRD analysis

X-ray diffraction of threads was carried out using a SIEMENS X-Ray Diffractometer, type Philips type PW 1840, D 5000, given 40 Kv CU Ka, radiation of 20mA. This was done in order to identify the type of mordant used in the dyeing process, as well as to determine the deterioration and degradation components deposited on the surface of the fibres.

Results and discussion

Visual examination

The results show that all visual features of the yarns from different colours of the kilim are composed of wool (Fig. 3-6) [21]. The fibres were generally in bad condition for warp and weft. It was extremely brittle, weakened and so drying besides to the disfiguration of morphological properties of the wool threads.



Fig. 3. Surface appearance of dyed wool fibres taken from the archaeological morocco kilim, examined by stereo-microscope

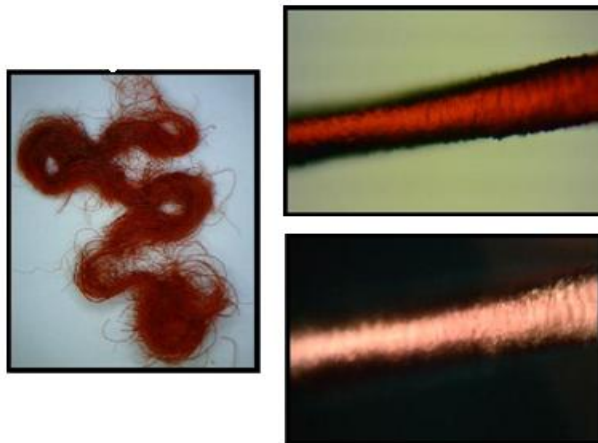


Fig. 4. The microscopic pictures taken by polarizing microscope, which identify the kind of "wool" fibres, and illustrate the damage in the surface

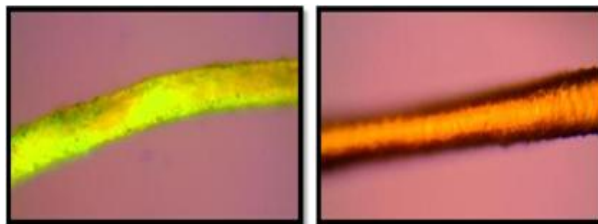


Fig. 5. The surface appearance of the yellow archaeological sample, and damage incurred, examined by polarizing microscope

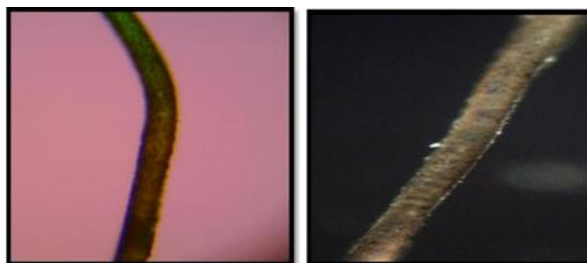


Fig. 6. The microscopic pictures, taken by polarizing microscope, of black yarn taken from the archaeological kilim, which indicate to the missing in scales of wool fibres as a result of ageing inside the museum.

SEM examination

The SEM photos of the examined morocco textile prove that the type of yarn used in decoration the morocco kilim is wool [22-23]. In addition they show the damage incurred in the surface appearance: the yarns are extremely rough, damaged, broken, with degradation products on the surface, the scales have disappeared completely in many parts all over the yarn, with characteristic transverse cracking and longitudinal splitting, as shown in Figure 7.

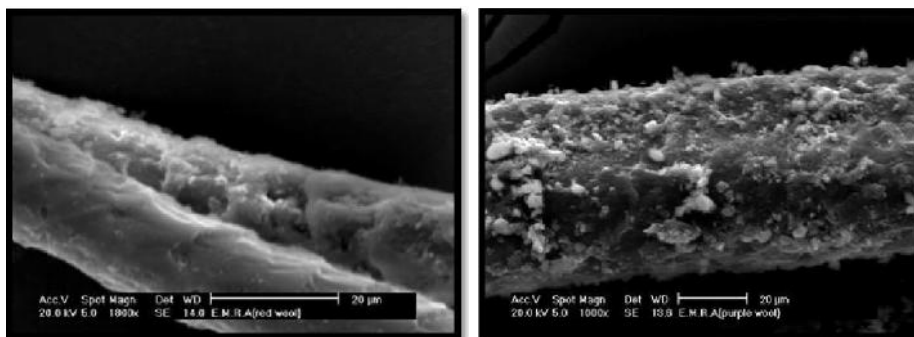


Fig. 7. SEM image of the archaeological samples which indicate the destruction of surface appearance besides to the accumulation of dust

XRD analysis

The spectra of the examined samples showed that the mineral composition of the dirt contained quartz, calcite, dolomite, albite, and clay minerals. This composition is similar to that of the common mordants used in the dyeing process of wool yarn at that period of time in Egypt (Fig. 8-10) [24-25].

The dark red wool yarn indicated as the mordant used in dyeing copper sulphate, with 67.7% of the sample, alongside iron oxide (geocite) as a result of converting the main component of fibres by chemical reactions.

The yellow wool yarn illustrates the usage of alum as the mordant in dyeing the wool fibres, besides aluminium silicate, as a kind of dust component.

The dark brown wool fibre reveals the use of ferrous sulphate as a mordant according to card (12-0068), which interpreted the sample's darkness of colour. The existence of hydrated iron oxide reveals the increase of the acidity of the wool fibres as a result of reactions with sulphuric acid produced by air pollution in the inner environment.

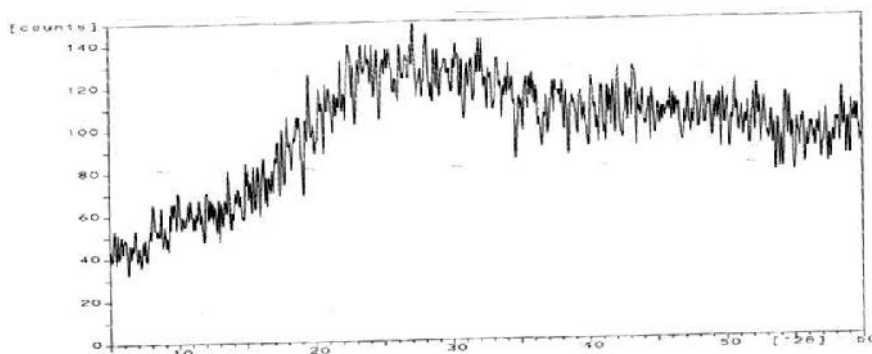


Fig. 8. XRD for dark red wool fibres , identifying copper sulphate at 67.7 % as a mordant, in addition to iron oxide (geocite) as "products of chemical reactions"

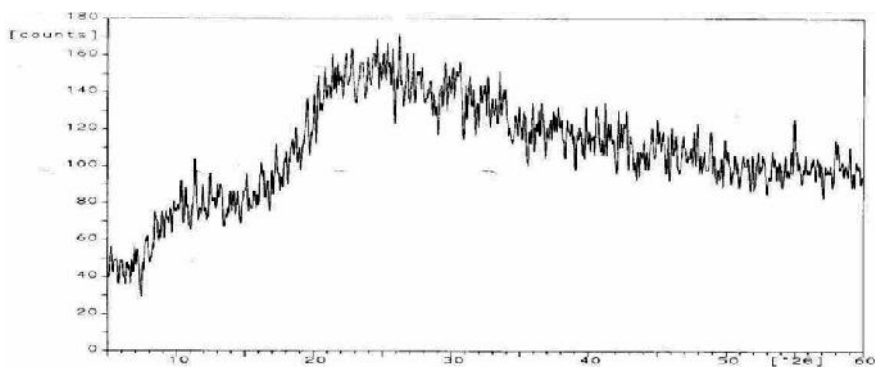


Fig. 9. XRD for yellow wool fibres, which refer to existence of alum as a mordant, and aluminium silicate as a kind of dust components

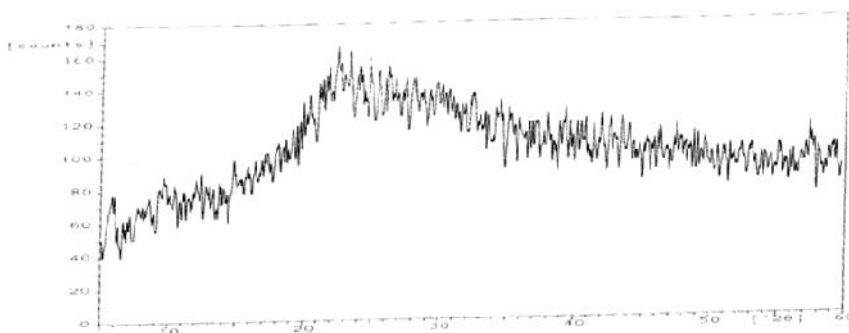


Fig. 10. XRD for dark brown wool fibre, which indicates ferrous sulphate as a mordant, in addition to hydrated iron oxide, as "degradation products"

Conclusion

This study presents the application of non-destructive physical techniques for examination and analysis of archaeological textiles. The analysis of the samples collected from the object is an extreme challenge on account of the reduced size of the collected material, the low abundance of target compounds, and the presence of ageing products.

The microscopic examination by stereo-microscopy, optical microscopy and scanning electron microscopy were sufficient to characterize the kind of fibres and the state of deterioration and degradation. XRD is useful for identifying the mordant used in the dyeing process in addition to degradation products. These techniques illustrate the state of deterioration and degradation of the morocco kilim used as case study, which help the conservator to make decision regarding the procedure of conservation. The visual examination indicated the poor condition of the kilim and the morphological appearance of the dyed wool yarn taken from the kilim.

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References

- [1] A. Bidarra, *Microscopy and Microanalysis Applications in Cultural Heritage Research*, **e-Conservation Magazine**, **22**, 2011, pp. 22-26, <http://www.e-conservationline.com/content/view/1034> USA
- [2] E.A. Verella, **Conservation Science for the Cultural Heritage**, Springer, Greece, 2013, pp. 224-230.
- [3] C. Cementi, *A spectrometric and chromatographic approach to the study ageing madder dyestuff on wool*, **Analytical Chimica Acta**, **596**, 2007, pp. 46-45.
- [4] E. Osman, Y. Zidan, N. Kamal, *Using The Microscopic And Spectroscopic Techniques To Identify And Characterize Archeological Artifacts*, **International Journal of Conservation Science**, **5**(4), 2014, pp. 459-468
- [5] O. Abdel-Kareem, Y. Zidan, N. Lokma, H. Ahmed, *Conservation of a Rare Painting Ancient Museum in Cairo*, **e- Preservation Scientific**, **5**, 2008, pp. 6-15
- [6] H.A. Ahmed, O.A. Nassef, *From Ptolemaic to modern ink via laser induced breakdown spectroscopy*, **Analytical Methods**, **5**(12), 2013, pp. 3114-3121.
- [7] J. Liu, *Identification of ancient textiles from Yingpan, Xinjiang, by multiple analytical techniques*, **Journal of Archaeological Science**, **38**, 2011, pp. 1763-1770.
- [8] W.Y. Li, J.L. Zhou, **Archaeological Finds at The Burial Site of Yingpan and Some Related Questions**. **Archaeological**, Shanghai Translation Publishing House, Hanghai, 1998, pp. 63-75.
- [9] E.S. Rodríguez, A.A. Rodríguez, M.A. García Rodríguez, M. del Egado, C. Cámara, *Identification of natural dyes in historical coptic textiles from the National Archaeological Museum of Spain*, **E-Conservation Magazine**, **15**, 2010, pp. 32-45.
- [10] C. Bergfjord, B. Hols, *A procedure for identifying textile bast fibers using microscopy: Flax, nettle/ramie, hemp and jute*, **Ultra Microscopy**, **110**(9), 2010, pp. 1192-1197.
- [11] A. Burnstock, C.C. Jone, *Scanning electron microscopy techniques for imaging materials from paintings*, **Radiation in Art and Archeometry** (Editors: D.C. Creagh and D.A. Bradley), Elsevier, London, 2000, pp. 202-231.
- [12] L.A. Donaldson, *Analysis of fibres using microscopy*, **Handbook of Textile Fiber Structure** (Editors: S. Eichhorn, J.W.S. Hearle, M. Jaffe and T. Kikutani), vol 1, Fundamentals and Manufactured Polymer Fibres, ed. 1st, Elsevier, 2009, pp. 121-153.
- [13] A. Massa, M. Masali, A.M. Conti Fuhrman., *Early Egyptian mummy hairs: Tensile strength tests, optical and scanning electron microscope observation*, **Journal of Human Evolution**, **9**(2), 1980, pp. 133-134.
- [14] J.W.S. Hearle, S.C. Simmens, *Electron microscope studies of textile fibers and materials Original Research Article*, **Polymer**, **14**(6), 1973, pp. 273-285.
- [15] M. Schreiner, B. Fruhmann, D. Jembrih-Simburger, R. Linke, *X-rays in art and archeology - An overview*, **Powder Diffraction**, **19**(1), 2004, pp. 3-11.
- [16] H.E. Ahmed, Y.E. Ziddan, *New approach for conservation treatment a silk textile in Islamic art museum*, **Journal of Cultural Heritage**, **12**, 2011, pp. 412-419.
- [17] J. Formica, *X-ray Diffraction*, **Hand book of Instrumental Techniques For Analytical Chemistry**, Prentice Hall Inc., New Jersey, 1998, pp. 339-357.
- [18] M. Cybulska, A. Jedraszek-Bomba, S. Kuberski, H. Wrzosek, *Methods of chemical and physicochemical analysis in the identification of Archeological and historical textiles*, **Fibers and Textiles in Eastern Europe**, **16**(5), 2008, pp. 67-73.
- [19] O.M. Abdel-Kareem, *The long-term effect of selected conservation materials used in the treatment of museum artifacts on some properties of textiles*, **Polymer Degradation and Stability**, **87**, 2005, pp. 121-130.
- [20] C.N. Nelson, R.F. Johnson, *Attribute characterization schemes for ancient textiles*, **ARS Textrina**, **9**, 1988, pp. 11-41.

- [21] M. Cybulska, T. Florczak, J. Maik, *Archeological textiles- Analysis, identification and reconstruction*, **5th World Textile Conference AUTEX**, 2005, pp. 878-883.
 - [22] R. Zemaityte, V. Jonaitiene, R. Milasius, S. Stanys, R. Ulozaite, *Analysis and identification of fiber constitution of archeological textiles*, **Materials Science (Medziagotyra)**, **12**(3), 2006, pp. 258-261.
 - [23] A.H. Madsen, *The wool material in thenercheological textile finds*, **NESAT II Textile Symposium**, Copenhagen, 1988, pp. 20-25.
 - [24] D.A. Peggie, A.N. Hulme, H. McNab, A. Quye, *Towards the identification of characteristic minor components from textiles dyed with weld (*Reseda luteola L.*) and those dyed with Mexican cochineal (*Dactylopius coccus Costa*)*, **Microchimica Acta**, **162**, 2008, pp. 371-380.
 - [25] J. Wouters, *Dye analysis in a broad perspective: a study of 3rd to 10th century Coptic textiles from Belgian private collections*, **Dyes in History and Archaeology**, **13**, 1995, pp. 38-45.
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