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USING THE MICROSCOPIC AND SPECTROSCOPIC TECHNIQUES TO IDENTIFY AND CHARACTERIZE ARCHEOLOGICAL ARTIFACTS

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Abstract

The aim of this work is the identification of the fiber type, degree of deterioration and of the natural dyes employed in the manufacture of some pieces of Egyptian Coptic textiles in order to help in its documentation and conservation. So, fibers from that Coptic fabric were analyzed using optical microscope and scanning electron microscope with energy –dispersive spectroscopy (EDS) to investigate the surface morphology of the object. X-ray diffraction (XRD) technique was used to define the damage degree of the artifact fibers. The results shows that the deterioration of the fibers is due to ageing. The investigation was completed by the vibrational characterization of indigo natural dye, which was detected by Fourier Transform Infra Red (FTIR) spectroscopy. The archeological fibers were identified as wool dyed with a natural dye (indigo) as the field literature suggests, with samples of this period. The archeological dye has the same characteristic bands as the modern indigo dye. Many metallic substances were detected which suggest the mordanting or the soil effect, accumulated on the ancient artifact. Characterizing the components of the ancient dyes and of the used fabric, could lead to an understanding of the reactions that deteriorate the ancient artifacts, which in turns helps the scientists, art historians and conservators to establish a more detailed data record, to evaluate the present condition of the object so they can restore the artifact properly.

Keywords: *Coptic fabric; XRD; FTIR; SEM; OM; Archeological fibers; Conservation; Indigo dye.*

Introduction

Archeological textile studies are now recognized as a robust source of information for anthropological inquiry. Over the past two decades several important developments have been made, enabling a more integrated approach to their study than in the past. Addressed topics ranged from the development of methods for analyzing degraded fibers, to the comparative study of specific histories of textiles and clothing traditions [1]. The application of analytical techniques, initially developed in the field of material science, for objects of art and archeology gives the art historians and archeologists the possibility to gain information about the material composition of such objects and prepares answers to the questions of where, when or who is the author of the artifact. Additionally, such investigations can help to understand the way of manufacturing artifacts and hence the way of life of the cultures studied. Scientific

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investigations are also valuable and in some cases indispensable for conservation projects in order to differentiate the original parts of an object from later additions, former restoration works, falsifications and even fakes. Moreover, this analysis can provide key information for the application of an appropriate treatment in current interventions of conservation and restoration [2, 3].

New standards of documentation with the use of modern non-destructive analytical methods are necessary if we want to preserve them for the next generations. Proper documentation of these objects requires cooperation between researchers representing different areas of science, like archeology, chemistry, textile technology and the art history [4]. There are many published papers with analyses of ancient textiles that could be considered as guides in the examination of such artifacts.

The first objective of archeological textile reconstruction is the identification of fiber constitution. The ability to accurately identifying fibers is of great importance to conservators, allowing the most appropriate methods of treatment to be employed [5]. In some instances the techniques range from low level magnification to scanning electron microscopy.

The modern technology (Fourier Transform Infrared FTIR) allows to determine a fiber constitution of textiles and sometimes even the former red or blue colors (chemical analysis) [6-11]. FTIR can identify organic as well as inorganic substances. The unknown substances can be identified by comparison to a library of known materials [12].

According to A. Domenech-Carbo et al [13], FTIR is the technique chosen in the study of artifacts because it only requires a small quantity of sample. Some infrared methods, such as ATR-FTIR can even measure whole samples without pretreatment. In one particular application performed by Ortega-Aviles et al [14] only about 1.0mg of sample was used to investigate the authentication of the Virgin of Sorrow painting by means of FTIR.

Dye is another characterized archeological attribute of the textile. The identification of natural dyes present in historical textiles can contribute to evidence earlier restorations and provides key information for the application of an appropriate treatment in current intervention of conservation and restoration [15-16]. The literature about the characterization of natural dyes in Coptic textiles is relatively extensive. For example, J. Wouters in 1995 [17] presented different studies using HPLC-DAD of extracts from Coptic objects. Later on, between 2003 and 2004, results about the natural dyes of Coptic textiles from National Museum in Warsaw were presented employing HPLC-DAD [18], LC-DAD-MS [19] and LC-DAD/fluorescence detection/MS [20]. Other interesting research article was presented by A. Verhecken [21], where the objective was to establish a correlation between the age of textiles from Egypt, Syria and Israel and the dyes used for them. Further work was carried out by R. Hofmann-de Keijzer et al. [22], where the authors give an overview of dyes and dyeing techniques used in the Late Antiquity in Egypt presenting their results about an investigation of natural dyes in two Coptic textile fragments from the Museum Für Angewandte Kunst (Vienna).

Energy dispersive X-ray spectroscopy (EDS, EDX or EDXRF) is an analytical technique used for the elemental analysis or chemical characterization of a sample. It is one of the variants of XRF. As a type of spectroscopy, it relies on the investigation of a sample through interactions between electromagnetic radiation and matter, analyzing x-rays is emitted by the matter in response to being hit with charged particles. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing x-rays that are characteristic of an element's atomic structure to be identified uniquely from each other [23]. The output of EDX analysis is an EDX spectrum. The higher the obtained peak is in a spectrum, the more concentrated the element is in the sample.

For the identification of individual pigments additional investigations are necessary, where scanning electron microscopy (SEM) in combination with energy dispersive X-ray microanalysis (EDX) has been used widely. A Scanning Electron Microscope (SEM) can be utilized for high magnification imaging of almost all materials. With SEM in combination with

EDX it is also possible to find out what elements are in different parts of a sample. The instrument is very suitable for different kinds of investigations. It is possible to investigate the fiber structure in wood and paper, metal fracture surfaces, production defects in rubber and plastic.

X-ray fluorescence (XRF) as well as x-ray diffraction analysis (XRD) has gained a lot of interest in the last decades [24] as both methods have been proved to be sufficiently non-destructive to identify the degree of deterioration and degradation of the artifact, if used with care and in respect to possible damages due to extensive radiation doses [25].

The main problem hindering the proper analysis and documentation of archeological textiles is a lack of co-operation between scientists dealing with different aspects of these objects- archeological, artistic, historical and technological. The paper includes the examining of manufacturing methods and raw material used, which is fundamental for proper identification of the object, its origin and dating.

Proper and detailed analysis can be very helpful in the conservation of fabrics. The data obtained can be used in the process of the reconstruction of archeological textiles. In the case of historical textiles, it allows proper identification of the workshop and thus, can be very helpful in determining a fabric's age and origin.

Egypt is one of the first countries where natural dyes were used, and its climatic and cultural conditions are favorable to conservation of archaeological textiles [26, 27]. This study examines the characteristics of the archeological fibers obtained form two different Egyptian Coptic samples by using optical microscope, scanning electron microscopy (SEM), Fourier transform infrared spectra (FTIR) and x-ray diffraction (XRD), and establishes a more detailed data record for the ancient wool fibers. Also, in order to have a better understanding of the archeological dyed wool fibers, sample of modern indigo blue dye was used as a comparison.

Experimental

Samples

The examined undocumented Coptic textile fabric is obtained from the Applied Arts Museum in Egypt, the sample dates back to 5-7 century. Fibers from the sample were taken to be analyzed for confirmation and to document the origin of this artifact. To confirm the type of the fibers and of the dye applied on it, modern wool fabric sample dyed with modern indigo was taken as reference (blank) sample for comparison. Figure 1 represents the undocumented archeological sample under investigation.

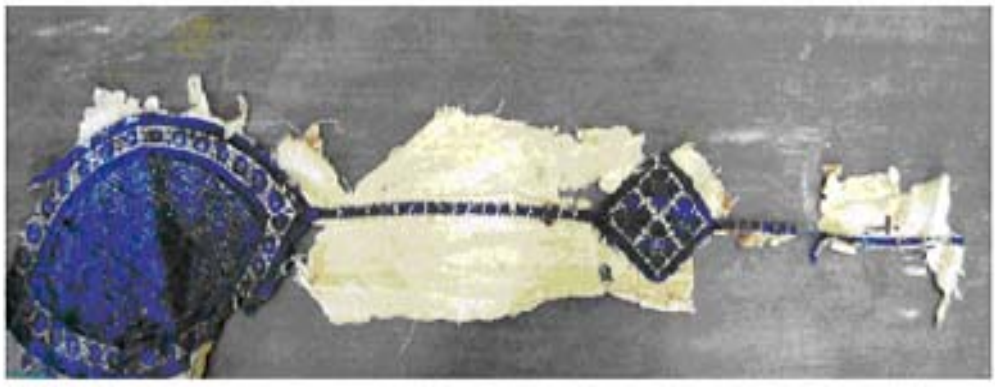

Fig. 1. The archeological artifact under investigation

Different techniques were used to investigate the undocumented samples, as shown below.

Optical Microscopy

The surface morphology of the both tested samples (modern and artifact) was investigated by optical microscope (OM) through a video microscope system of SDL International-UK, at 1000X magnification.

Fourier Transform Infra Red Spectroscopy with Attenuation Total Reflection

FTIR-ATR spectra of the above mentioned artifact as well as the blank sample were recorded with Nicolet 380 Spectrometer using a zinc selenid crystal with the wavelength range 650 - 4000cm^{-1}. To ensure reproducible contact between the crystal surface and the fabric, a pressure of about 18Kpa was applied to the crystal holder [28]. The FTIR absorbance frequencies for the investigated samples were recorded with an average of 128 scans using a resolution of 4cm^{-1}.

X-ray Diffraction

X-ray diffraction analysis of the archeological samples under test was carried out using Philips X-ray diffraction, type PW 1840. The sample was analyzed using Ni filter and CuKa radiation (θ = 1.540˚A) at generator intensity of 40kV and a generator current of 25mA. The samples were scanned at 2θ in the range of 5˚-50˚ at scan rate 2˚min^{-1}.

Scanning Electron Microscope with Energy Dispersive X-Ray

The scanning electron microscope (SEM) investigation was carried out using SEM of model (Philips XL30) attached with Energy Dispersive X-ray (EDX) unit, with accelerated voltage 30 V, magnification of 10X up to 4000X and resolution of 3.5nm. The surface morphology of the tested samples was measured on very small samples coated with gold. For EDX analysis, the tested fibers were carbon coated.

Results and Discussion

Morphological investigation of fibers taken from the artifact was carried out using OM. The examination of the cross-section of these fibers by optical microscopy provides sufficient information to confirm the type of these fibers. The taken images from the ancient artifact were compared with images taken from the new wool fiber sample. Figure 2 (a and b) revealed that both types of fibers have typical wool morphological features in common, i.e., the investigated fibers look like long cylinder with scaly corkscrews all aligned in a single direction [29] which confirms that the tested archeological artifact fibers are wool in nature.

FTIR-ATR was employed to investigate the function groups of fibers as well as the dye applied on them. The data obtained by absorption, from the spectrum of the blue color of the examined artifact, as well as the new wool fabric sample dyed with new indigo dye, are shown in table 1. It is clear that there is a considerably matching between the two analyzed dyed samples. Where, common function groups are obtained at nearly the same wave numbers in both tested samples. This result ensures that the tested artifact is wool, while the blue color found on it, is obtained from indigo dye [30].

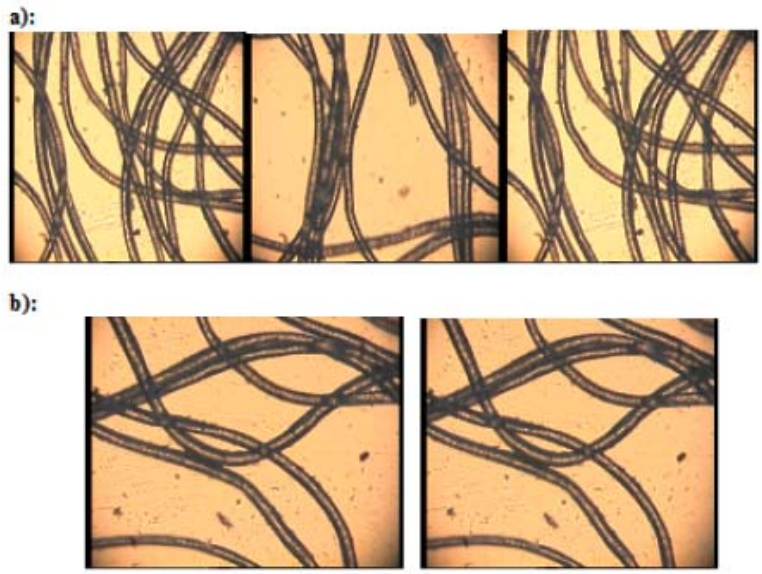

Fig. 2. Wool fibers under the optical microscope:
a) ancient fiber sample, and b) new fiber sample

Table 1. The characteristic function groups assigned to wool dyed with indigo dye.

Wave number of the obtained peaks (cm^{-1})		Function group related to indigo dye	Function group related to wool fabric
Modern dyed sample	Archeological dyed sample		
3290	3250	N-H Primary and Secondary Amines	N-H stretch. Of Serine amino acid
2927	2900	O-H stretch	C-H stretch
1647	1600	C=C Stretch Aromatic	N-H bending in Lysine amino acid
1448	1455	C-H bending	C-C stretch. in tyrosine amino acid (finger print region of wool below $1500cm^{-1}$)
1040	1060	C-O stretch (finger print region less than $1100cm^{-1}$	Cystine oxides

A slight shift can be seen between the obtained band regions of all the characteristic groups of both modern and archeological indigo and wool fabric. With the archeological artifact, changes can be seen in the shape of the obtained bands appeared in the broadening of the bands indicating the increase in the disorder structure of the sample molecules [30].

The absorption band at 3290-3250cm^{-1} assigned to N-H stretching is common between wool fabric and indigo dye [31, 32]. This band appeared as a slightly sharp band indicating that it represents serine amino acid in wool or primary and secondary amines of indigo dye [31-35].

The band at ≈ 2900-2927cm^{-1} is assigned to O-H stretching in case of indigo and to C-H stretching in case of wool fabric.

The signal at ≈1600-1647cm^{-1} is assigned to C=C stretching for indigo, while for wool it is assigned to N-H bending in lysine amino acid.

The band at ≈ 1448-1455cm^{-1} assigned to C-H bending in case of indigo and as C-C stretching of tyrosine amino acid for wool [34].

The signal appeared at ≈ 1040-1060cm^{-1} represents C-O stretching of indigo and cystine oxides (mono and dioxides) for wool fabric. It is known that the region after 1100 cm^{-1} is considered the fingerprint region of indigo dye [36, 37].

The measurement of the degree of crystallinity provides useful data while characterizing fibers using X-ray diffractometry. X-ray reflections data of tested fibers taken from ancient as well as modern wool samples are shown in table 2. The XRD intensity profiles of the fibers show a well resolved spectrum of wool, with four characteristic reflections at around ; 2 θ ≈ 10°, 18°, 23° and nearly 30° [38]. The first two peaks are at medium intensity, yet the third is very sharp, strong intensity. The smallest peak at 2 θ ≈ 30° is another feature shared by all the tested wool fibers. This indicates that the basic morphology of the archeological fibers have not altered after burial for centuries.

The Crystallinity index of both ancient and modern samples was calculated using the obtained results represented in table 2 according to the following formula [39].

$$\text{Crystallinity index} = \frac{If - Is}{If} x100$$

Where: I_f is the relative intensity of the fundamental peak (2θ ≈ 23˚), and I_s is the relative intensity of the secondary peak (2θ ≈ 30 ˚) in case of ancient sample. While the I_f at 2θ ≈ 10˚ and I_s at 2θ ≈ 18˚ in case of modern samples. It was found that, the cryatallinity index of the ancient object is higher than that of the modern one. This may a consequence of ageing, together with the soiling effects due to burial.

Table 2. The characteristic reflections and the peak intensities of the examined ancient and modern wool samples respectively

	2 θ	d-value	Peak intensity	Crystallinity index %
Ancient wool sample	8-10°	13.82	180	43.07
	17-18°	5.19	280	
	20-23°	3.81	650	
	27-30°	3.27	370	
Modern wool sample	9-10°	9.77	260	40.00
	16-18°	4.64	156	
	20-22°	4.17	140	
	28-34°	3.13	111	

SEM- EDX analysis gives more information about the presence of metals on the morphology of the samples. The data generated by EDX analysis consist of a spectrum with peaks corresponding to chemical elements that are found in the composition of the analyzed sample [40]. Figure 3 represents the EDX spectrum of the investigated ancient specimens. It is clear that, several elements were detected: sulfur, silicon, calcium, iron, copper and zinc, depicted in different areas of the examined ancient fibers. Sulfur, silicon and calcium were detected in large quantities; the sulfur and silicon were the most abundant as the first element is the main component of wool protein, while silicon is the main part of soil.

Figure 4 (a-d) shows the SEM images of archeological and modern wool fiber samples. The SEM in figure 4 (a) showed that the scales of modern wool are clear and arranged compactly around the fibers. While the SEM analysis of surface morphology of the ancient fiber samples revealed optimal damage that occurred to the scale structure as a result of the ageing process of the sample, also the images showed that the ancient samples were heavily soiled, traces of dirt being accumulated on the fibers surface. The area of the sample where the scales completely disappeared, also shows tearing of the fibers. The degradation and deterioration of the fibers could have taken place at different times, when the textiles were buried or after they were excavated. An attempt was made to relate the damage of the fibers to the degradation caused in the buried time.

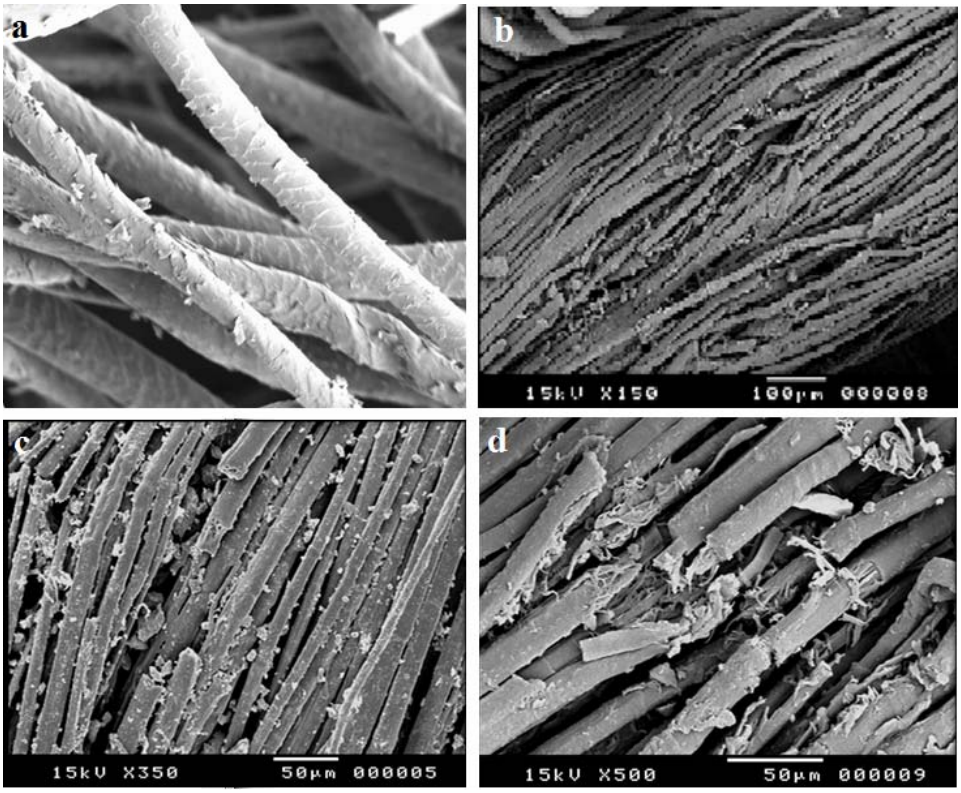

Fig. 4. SEM images of the tested wool fibers: a) modern wool fibers and b), c) and d) ancient wool fibers with different magnifications (100X, 350X and 500X).

Conclusion

An original Coptic artifact thought to be from the Coptic age, was investigated using nondestructive techniques to help in its documentation and to a properly conservation. Optical microscopic analysis and the FTIR spectra showed that this artifact was made of wool fibers and the dye applied on it was indigo dye. SEM images clarified the degree of deterioration and degradation, while the EDX showed that there are traces of sulfur and silicone, elements that correspond to wool components and to the burial effects. The XRD analysis revealed that the burial conditions did not affect the basic morphology of the archeological fibers.

The dye and fibers identified in the sample under study are in agreement with the commonly reported materials for Coptic textiles belonging to the same age.

With the help of modern analysis techniques we can obtain preliminary information on the types of fibers, dyes and the degree of deterioration and degradation of the archeological textiles in museums, which could lead to an understanding of the reactions that lead to the deterioration of the ancient artifact which in turns helps the scientists, art historians and conservators to establish more detailed data, to record and evaluate the present condition of the object, the degradation phenomena so they can restore the artifact properly. This means that, there are more opportunities for collaboration between scientists from different disciplines to enhance the analysis and documentation of archeological textiles.

References

[1] I. Good, *Archeological textiles: A review of current research*, **Annual Review of Anthropology, 30**, 2001, pp. 209-226.

[2] E.S. Rodríguez, A.A. Rodríguez, M.A. García Rodríguez, M. del Egido, C. Cámara , *Identification of natural dyes in historical coptic textiles from the National Archaeological Museum of Spain*, **E-Conservation Magazine**, **15**, 2010, pp. 32-4

[3] M. Schreiner, B. Fruhmann, D. Jembrih-Simburger, R. Linke , *X-rays in art and archeology - An overview*, **International Center for Diffraction Data**, **47**, 2004, pp. 1-17.

[4] 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.

[5] C.N. Nelson, R.F. Johnson, *Attribute characterization schemes for ancient textiles*, **ARS Textrina**, **9** , 1988, pp. 11-41.

[6] 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.

[7] M. Cybulska, T. Florczak, J. Maik, " *Archeological textiles- Analysis, identification and reconstruction*", **5th World Textile Conference AUTEX**, 2005, pp. 878-883

[8] M. Quack, B. Bunsenges, "*Molecular epectroscopy and molecular dynamics: Theory and experiment*", **Physikal Chemisctry**, **99**, 1995, pp. 231-245

[9] S.P. Davis, M.C. Abrams, J.W. Brault, **Fourier Transform Spectrometry**, Academic Press, San Diego, 2001, p. 284.

[10] P. Garside, , P. Wyeth, "Identification of cellulose fibers by FTIR spectroscopy: Thread and single fiber analysis by Attenuated Total Reflectance", **Studies of Conservation**, 48 (4), 2003, pp.3-8

[11] A.H. Madsen, *The wool material in thenercheological textile finds*, **NESAT II Textile Symposium**, Copenhagen, 1988, pp. 20-25.

[12] B.C. Smith, **Fundamentals of Fourier Transform Infrared Spectroscopy**, CRC Press, USA, LLC, 1996, pp. 15-53.

[13] M.T. Domenech, F. Bosch, J.V. Gimeno, V. Martinez, *Fourier Transfrom Infrared Spectroscopy and the Analytical Study of Works of Art for Purposes of Diagnosis and Conservation,* **Analytica Chimica Acta**, **330,** 1996**,** pp 207-215.

[14] M. Ortega-Avi, P. Vandenabeele, D. Tenorio, M. Jimenez-Reyes, N. Gutierrez, *Spectroscopic investigation of a "Virgin of Sorrows" Canavas Painting: A Multi-method Approach,* **Analytica Chimica Acta**, **550**(1-2), 2005, pp. 164-172 .

[15] 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.

[16] N.N. Cherilyn Nelson, F.J. Robert, *Attribute characterization schemes for ancient textiles*, **ARS Textrina**, **9**, 1988, pp. 11-41.

[17] 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.

[18] J. Orska-Gawrys, I. Surowiec, J. Kehl, H. Rejniak, K. Urbaniak-Walczak, M. Trojanowicz, *Identification of natural dyes in archeological Coptic textiles by HPLC-PDA*, **Journal of Chromatography A. 989**, 2003, pp. 239-248.

[19] B. Szostek, J. Orska-Gawrys, I. Surowiec, M. Trojanowicz, *Investigation of natural dyes occurring in historical Coptic textiles by high performance liquid chromatography whith UV-Vis and mass spectrometric detection,* **Journal of Chromatography A. 1012**, 2003, pp. 179-192.

[20] M. Trojanowicz, J. Orska-Gawrys, I. Surowiec, B. Szostek, K. Urbaniak-Walczak, J. Kehl, M. Wróbel, *Chromatographic investigation of dyes extracted from coptic textiles from the National Museum in Warsaw*, **Studies in Conservation, 49**, 2004, pp. 115-130.

[21] A. Verhecken, *Relation between age and dyes of 1st millennium AD textiles found in Egypt*, **Proceedings of the 4th Meeting of the Study Group. Textiles from the Niley Valley**, 16-17 April 2005, Antwerp, pp. 208-209.

[22] R. Hofmann de Keijzer, M.R. van Bommel, M. de Keijzer, *Coptic textiles: dyes, dyeing techniques and dyestuff analysis of two textile fragments of the MAK Viena,* **Proceedings of the 4th Meeting of the Study Group. Textiles from the Niley Valley**, 16-17 April 2005, Antwerp, pp. 214-228.

[23] * * *, **Energy Dispersive Ray Introduction: Documentation,** http://cime.epfl.ch/files/content/sites/cime2/files/shared/Files/Teaching/MSE_603_2011_Autumn/Chapter%2012%20-%20EDX.pdf, (accessed at 20.02.2014).

[24] M. Schreiner, B. Fruhmann, D. Jembrih-Simburgger, R. Linke, *X-ray in art and archeology-an overview*, **JCPDS (**International Center for Diffraction Data), **47**, 2004, pp. 1-17.

[25] H. Barbara, **Infrared Spectroscopy: Fundamentals and Application**, John Wiley & Sons, Ltd., Southern Gate, Chichester, England, 2005.

[26] H.E., Ahmed, *Protease enzyme used for artificial ageing on modern cotton fabric for historic textile preservation and restoration*, **International Journal of Conservation Science, 4**(2), 2013, pp. 177-188.

[27] H.E. Ahmed, *A new approach to the conservation of metallic embroidery threads in historic textile objects from private collections*, **International Journal of Conservation Science, 5**(1), 2013, pp. 21-34.

[28] R.M. Christie, R.R. Mather, R.H. Wardman, **The Chemistry of Color Application**, Blackwell Science Ltd., Oxford, USA, 2000.

[29] M. Leona, F. Casadio, M. Bacci and M. Picoll, *Identification of The Pre-Columbian Pigment Maya Blue on Works of Art by Noninvasive UV-VIS and Raman Spectroscopic Techniques*, **Journal of The American Institute for Conservation**, **43**(1), 2004, pp. 39 – 54.

[30] M. Odlyha, C. Theodorakopoulos, R. Campana, *Studies on woolen threads from historical tapesteies*, **AUTEX Research Journal**, **7**(1), 2007, pp. 9-18.

[31] J. Yao, Y. Liu, S. Yang and J. Liu, "*Characterization of Secondary Structure Transformation of Stretched and Slenderized Wool fibers with FTIR Spectra*", **Journal of Engineered Fibers and Fabrics**, **3**(2), 2008, pp. 1-10, http://www.jeffjournal.org

[32] E. M. Osman, M.N. Michael, H. Gohar, *The Effect of UV/Ozone on Natural Fabrics*, **International Journal of Chemistry**, **2**(2), 2010, pp. 28-39.

[33] N. Chanayath , S. Lhieochaiphant, S. Phutrakul, *Pigment Extraction Techniques from the Leaves of Indigofera tinctoriaLinn. and Baphicacanthus cusia Brem. and Chemical Structure Analysis of Their Major Components*, **CMU Journal**, **1**(2) , 2002, pp. 149-160.

[34] V. Bianda, J. Constenla, R. Haubrichs, .P. Zaffalon, " *Colorful experiments for Students: Synthesis of Indigo and Derivatives*", **Chemistry: Bulgarian Journal of Science Education**, **22**(1), 2013, pp. 52-65

[35] Q. Cao, S. Zhu, N. Pan, Y. Zhu, H. Tu, *Characterization of archeological cotton (G. herbaceum)fibers from Yingpan*, T**echnical Fibers in Historical Archeology**, **4**, 2009, pp 18-28.

[36] B. Lipp-Symonowicz, S. Sztajnowski , A. Kutak, *IR spectroscopy as a possible method of analysis fiber structures and their changes under various impacts*, **Intech Open Science**, 2012, pp. 1-15.

[37] H. Manfred, H. Meier, B. Zeeh, **Spectroscopic Methods in Organic Chemistry**, George Thieme, New York, 1997.

[38] S. F. Ibrahim, *Investigation into the Effect of UV/Ozone Treatments on the Physical Properties and Dyeing Kinetics of some Polymeric Fabrics*, **Ph.D. Thesis,** Faculty of Science, Cairo University, 2003.

[39] J. Cao, C.A. Billows, *Crystallinity determination of native and stretched wool by X-ray diffraction*, **Polymer International**, **48**, 1999, pp 1027-1033

[40] ***, Energy dispersive X-Ray (EDX) composition analysis. http://www.intertek.com/analysis/microscopy/edx/, (accessed at 20.02.2014)

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