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Archaeological Painted and Dyed Textiles: Studying Variations and Morphological Characteristics Using Microscopy Techniques

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THE MAIN objective of this study is to identify the differences between two important techniques of colouring and decorating textiles in ancient Egypt, namely the dyeing and painting and their influences on the morphology of archaeological textiles. In this study experimental samples were prepared from artificially aged dyed and painted linen textiles with some of the common pigments and natural dyes used in ancient Egypt, to obtain samples similar as possible to the archaeological ones. The samples were studied and investigated using a Scanning Electron Microscopy coupled with EDX unit (SEM-EDX) and Ultraviolet-visible (UV-VIS) microscopy. The results showed that the inorganic pigment particles are deposited on and in-between the fibres' surfaces, and serve as a protective coating for the fibres against the morphological deterioration caused by artificial ageing. In contrast, the dye molecules are small enough to penetrate and colour the fibres uniformly. They also decreased the degradation of the surface morphology of the fibres after ageing.

Keywords: Archaeological Textiles, Pigments, Natural Dyes, Accelerate ageing, SEM-EDX, UV-VIS microscopy.

Introduction

Textiles are naturally perishable materials, which therefore rarely survive in the archaeological archives. A notable exception is Egypt, where the sandy soil and the special burial rituals have helped the preservation of a large number of textiles, which formed a unique evidence of the spinning, weaving, dyeing and textile iconography over thousands of years [1]. The study of textiles can also shed new light on religious, cultural and economic life [2]. Textiles can be coloured by using organic or inorganic colourants, whether pigments or dyes, each of them require considerably different application methods because of its physical and chemical properties, it also leaves specific chemical signatures on the fibres [3].

Dyes are one of the main components of artworks and archaeological discoveries. The importance of natural dyes through prehistory and as well in historical periods is well- known and the high-quality dye was considered as invaluable as the noble metals [4][5]. The use of the textiles as painting substrate dates back to the ancient Egyptian cultures [6]. Among the painted textiles are mid-Eighteenth-Dynasty shrouds, as the Resti shroud with texts and vignettes from the Book of the Dead. It has been painted with hematite for red, Egyptian blue for blue, orpiment for yellow [3][7]. Ageing is the process of change over time, the concept of ageing applied to museum objects usually combines decay with degradation. The definition of the ageing term can be: irreversible changes that appear slowly in the long term [8]. The main aim of accelerating thermal degradation is the production of an artificially aged model of linen textile which is analogous as possible to the archaeological fabric, with the consideration that thermal deterioration increases with increasing temperature and ageing period [9].

UV-VIS microscopy uses fluorescence and phosphorescence to study the characteristics of organic or inorganic material. Moreover, it can provide distinct elaborated images of the coloured layers or the fibre morphology [10]. UV-VIS microscopy as well helps in the identification of weave structure, the torsion direction of the yarns and the used colouring techniques in archaeological fabrics either by painting or by dyeing [11] .The SEM allows greater detail, accuracy than the optical microscope [12]. Generally, SEM has been used for many years in

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the field of cultural heritage. It has been applied to a vast number of archaeological objects or similar experimental models and is now used with additional, powerful and innovative technologies to enrich morphological information with chemical details; physically and structurally [13]. As well as, it can be used for simultaneous imaging and analysis of biological degradation in ancient material, also for the investigation of the efficiency of conservation material [14 , 15].

This article sheds more light on the variations between dyeing and painting techniques on ancient textiles and identifying the materials and methods used in manufacturing them. Moreover, this paper aims to assess the influence of different colourants on the archaeological textiles morphology.The study was done on experimental 2 sets of artificially aged linen textile samples similar as possible to the ancient ones. The first set of mockups was dyed with indigo, cochineal and madder natural dyes, and the other set was painted with malachite, calcite and black charcoal. All mockups were investigated using Scanning Electron microscopy coupled with EDX unit (SEM-EDX) and Ultraviolet-visible microscopy (UV-VIS), in addition to the visual investigation as a starting point. The information presented in this work will be helpful to other researchers and conservators investigating textiles and for the assessment of textiles and their appropriateness for restoration. The results of this article will help Egyptologists, academic researchers and conservators to get information about the differences between the materials and techniques of textiles colouration used in ancient Egypt. In addition, the information presented in this paper will help conservators in producing new mockups, coloured fabrics that simulate the older ones. These mockups can be used as test samples in the research of conservation and training young conservators. In addition, this information will help in choosing the right materials and methods in the treatment of coloured archaeological textiles.

Major Differences between Dyeing and Painting Techniques

Two fundamental methods for colouring textile materials were well-known in ancient Egypt, dyeing and painting, each one requires considerably different application methods because of its physical and chemical properties; It also leaves specific chemical signatures [3]. A dye can be defined as a high-coloured substance that is used to dye a variety of materials such as textiles, leather, paper, medicine, cosmetics and toothpaste, etc. In terms of dye chemistry, a dye molecule has two main chemical groups, namely Chromophores and Auxochromes groups. By dyeing, the fabrics are dyed through the same colour, while by painting; the fabric receives a coloured pattern by using a mixture of pigment and binder; which might be applied directly to the textiles or onto a painting ground. During the dyeing process, each fibre should be left with the dye evenly distributed in its substance instead of being mostly limited to its outer surface [16]**.** The dye is commonly used as an aqueous solution and may need a mordant to enhance the stability of the dye on the fibre. All ancient Egyptian dyes are organic components that could bind with the natural and synthetic fibres and make them coloured [17]. Otherwise, pigments are usually inorganic compounds made up of particles that are insoluble in the binding media of application. Thus, the pigment is simply removed unless fixed with a binding media [18]. Mineral pigments may be slightly limited in the range of composition, while organic dyes may be more difficult to identify, not only because a single plant or animal source may give multiple components, but also because these components break down into many different components. In addition, a single colour component could be obtained from different sources. Dyes are often combined and additional dyeing aids, such as mordants, chelating agents or pH-altering agents are added to help fibres adhere with dyes [19]. Painted textiles are complex, heterogeneous, and usually consists of multi-layered. The characterization of materials and techniques used for the production of painted fabrics is essential to improve our understanding of these objects. The restorer must understand the textile and colour elements, their complex interactions and their often conflicting needs, in order to be able to make informed decisions on cleaning, consolidation and preventive conservation processes [20]. The oldest known example of painted linen found at Gebelein and goes back to the Predynastic period, now at the Turin Museum. The use of textiles as painting substrate is due to ancient Egyptian culture [21]. The painted textiles have many forms ,including processional banners [22], flags, costumes and accessories, wall hangings, painted and embroidered pictures, religious objects, such as thangkas and ancient Egyptian mummy cloths. These different objects are used in many different contexts and have a wide range of ornamental, social, political, and religious functions. The paint may be applied to just one area of the surface of a painted textile, which affects its appearance, function, decay and as well conservation, as the painted and unpainted parts have two clearly different surfaces [10].

Fig. 1. (A) Show dyed linen textile, Tutankhamon tunic, No. JE 62625, New kingdom, Cairo Egyptian museum. (B) Show funerary shroud, Painted linen textile – No.TR 9.12.95.1, Roman period, Cairo Egyptian museum.

Gettens and Stout [1966] named some common inorganic pigments formerly used in ancient periods like oxides, phosphates, carbonates, sulfates, sulfides, and heavy metals silicates. There are only a few organometallic compounds that form pigments such as Emerald green and pigments constitute of pure elements such as carbon and gold [23]. The most common approach of painting, which has been used since ancient times, is that a liquid paint is usually applied to a surface of the support as coating, often with a brush, and sometimes with fingers, a stick, or a swab as well. The pigment cakes were probably made by mixing finely ground pigment with gum Arabic and water and left to dry and used in the same way as the modern watercolour painting [24]. Arabic Gum was widely utilized in ancient Egypt as a binding media for watercolours techniques [25]. According to Herodotus, it is also a prevailing constituent of ink [6].

The knowledge of tools and dyeing techniques of the ancients has survived till nowadays. These ancient techniques were kept as a top secret, as they have been passed down from father to son through the centuries. Plenty of natural dyes that were used thousands of years ago have proven very successful and still present a trusty and delightful colour. Recipes are often lost, but ancient manuscripts and books provide us with a good opportunity to discover these recipes [20]. Dyes are classified in accordance with their chemical structures or methods of application. A group of atoms in dye molecules named chromophores as anthraquinone, azo, carbonyl, methine, nitro and others, are responsible for the dye colour. As well as, substituents named auxochromes, as carboxyl, amine, hydroxyl and sulphonate, withdraw and/or donate electrons, in consequence, generate or intensify the colour of the chromophores [26]. Natural dyes are produced by both plants and insects. They are largely limited to red, yellow, blue and brown dyes. The other colours are obtained by mixtures of these or by the use of various mordants [20]**.** In ancient Egypt, the uses of natural dyes, both individually and in combination, evidenced that a minimum of three different dyeing techniques were known (1) direct dyeing which directly use the dye whithout mordants or metal salts to fix the colour. (2) Substantive dyeing. As safflower, madder and henna need mordants to fix the dye. The dyeing bath is usually prepared before the immersion of the yarn or fabric. (3) Double or over-dyeing. This method consists of more than one dyeing step. A well-known example is the production of colours such as purple [27]. According to (Bogensberger and Russell-Motondorf 2020), purple is particularly appealing, as most of the old textiles studied; the use of plant dyes is usually a mixture of red and blue dyes. These dyeing techniques were used not only in the double-dyeing of fibres, but also in the spinning of blue and red fibres to obtain purple dyes[28].

Materials and Methods

Materials

Scoured and raw linen textiles were provided by Egylan Comany from Alexandria, Egypt. The linen fabrics with tabby weave 1/1. Respectively, the count of the warp and the weft threads are 26 and 23/ cm. Commercial natural dyes and pigments were supplied by “Kremer Pigment” including malachite, calcite and carbon charcoal.

Natural dyes as indigo, cochineal and madder. Gum Arabic was used as a medium for the pigments by dissolving it in hot water and leaving it for a day till it was fully dissolved [29].

Preparing Experimental Samples

A group of linen specimens were painted directly without preparing layer by using a brush, as this technique is most often used in textile painting in ancient Egypt [30]. The other group of samples was dyed with the mentioned above selected natural dyes. The dyeing processes was done as stated by the methods in the published literature [31], by using Alum (potassium aluminium sulphate $[KA_1(SO_4)_2.12H_2O)]$) as a mordant with madder and cochineal dyes. For indigo, a vat dyeing method was used. All experimental samples (Fig.2) were then aged thermally in accordance with Kerr et al (1989) and Feller (1994) in an electronic oven at a steady temperature of 140°C for 72 h [32][33].

Methods

Visual Observation

Visual observations and low-level magnifications are very valuable in the first stage of the assessment of the condition of the textile objects. These observations could result in much variant data related to the type of the materials used, the construction, spinning and weaving structure, and colouring techniques. An in-depth examination of the interface of the painted textile may indicate, for example, the possible existence of a ground layer. The condition of textiles as the presence of dirt, wrinkles, splits, peeling and erosion of the paint layers can also be documented [10].

Ultraviolet and Visible (UV-VIS) Microscopy

The experimental linen fabric specimens were investigated by NIKON ECLIPSE E600 with a high-pressure mercury lamp as a UV source (UV-2A- EX330-380- DM400- BA 420) and fibres Optic Visible source (Shott KL 1500). The images were captured by the camera of NIKON DS- FI1C, NIKON DS-U3 Digital Sight, and the software was used is Nikon ACT-V2.6.

Scanning Electron Microscopy with Energy-Dispersive X-Ray Spectroscopy (EDX)

SEM was carried out to investigate the tested fibres' surface morphology. Each sample was placed on an adhesive carbon disc mounted onto an aluminium SEM stub. The small piece of fibre samples was coated with a thin film of gold for 90 seconds, using POLARON SC 7620 SPUTTER COATER to increase their conductivity and avoid charging effect [34].

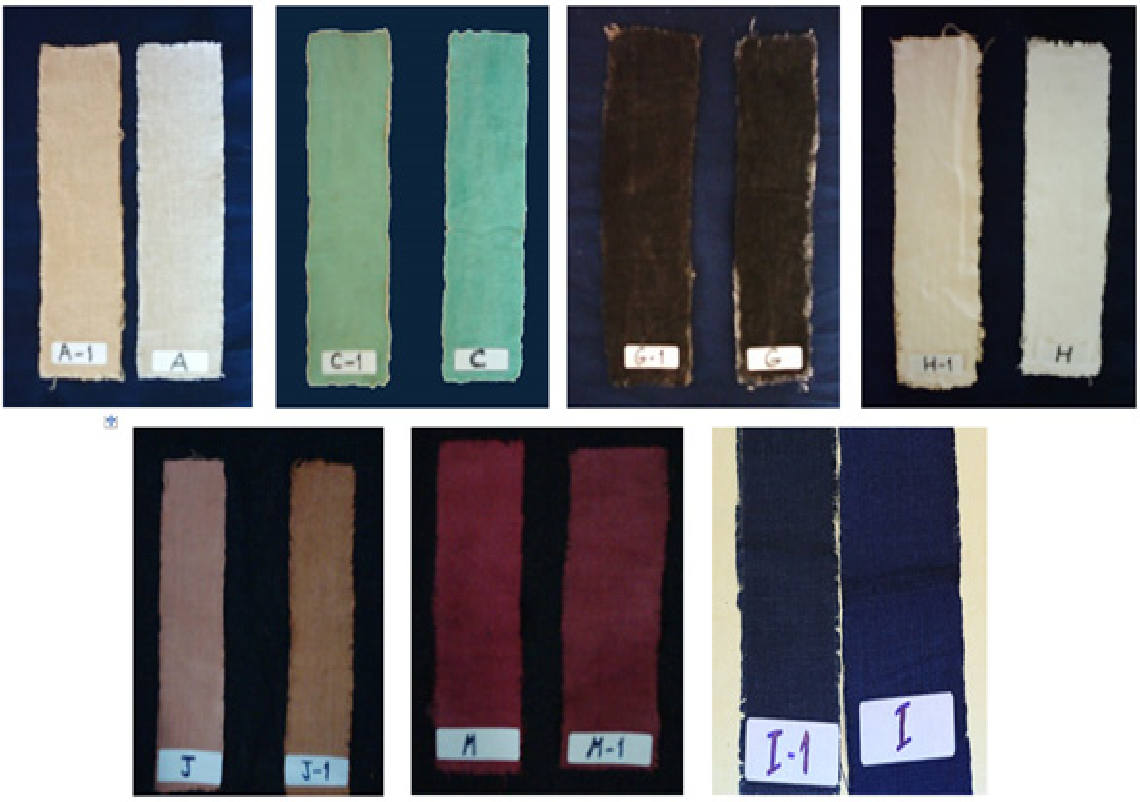

Fig. 2. Experimental samples before and after ageing, (A, A-1) standard linen mockups, (C, C-1) malachite-painted mockups, (G, G-1) charcoal-painted mockups, (H, H-1) Calcite-painted mockups, (J, J-1) madder-dyed mockups, (M, M-1) cochineal-dyed mockups, (I, I-1) indigo-dyed mockups.

Examination of the mockup samples was undertaken on the Jeol JSM 5600 system configured with an EDX detector, operating mostly at a 20-kV accelerating voltage and the working distance was 20-mm.

Results and Discussion

Visual observation and UV-VIS microscopy

Visual observation and UV-VIS microscopy of the empirical painted linen textiles with calcite, charcoal and malachite illustrated that the morphology of the fibres is completely covered by the pigment layers and their particles are too large to penetrate into the fabric, so they were deposited on and in-between the linen fibre surface. On the contrary, the visual observation and the UV-VIS microscope of experimentally dyed fibre with indigo, cochineal and madder dyes [Fig.3] demonstrated that the fibres are dyed in the same colour as the applied dye, the dye is evenly distributed over the textile fibres, as the dye molecules are small sufficient to saturate the fibres with the colour of the dye. In addition, it is observed visually some changes in the colours of both pigments and natural dyes where they have become darker due to ageing.

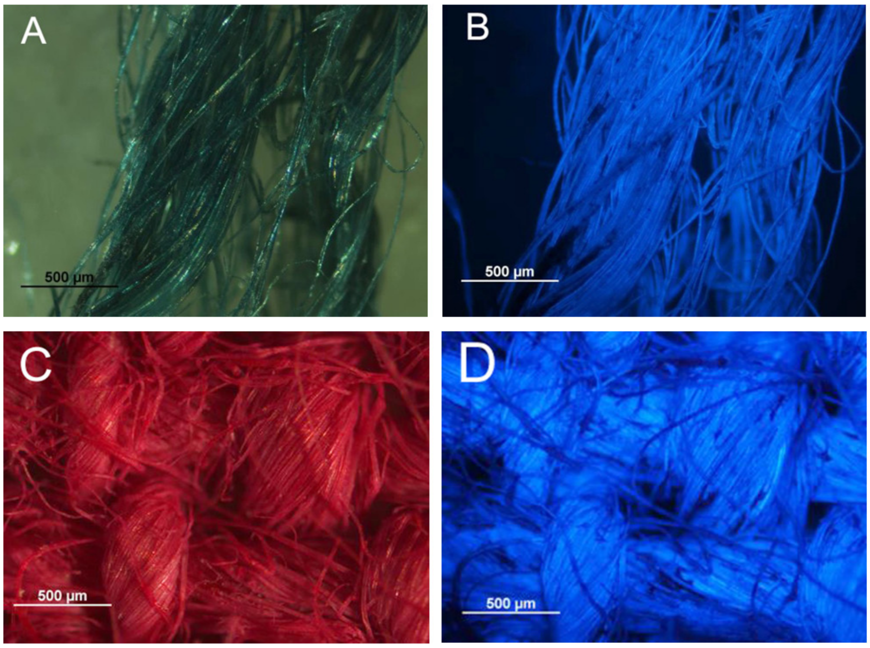

Fig. 3. UV-VIS miceroscope images of (a, b) fiber samples dyed with indigo dye, (c, d) Cochineal-dyed sample.

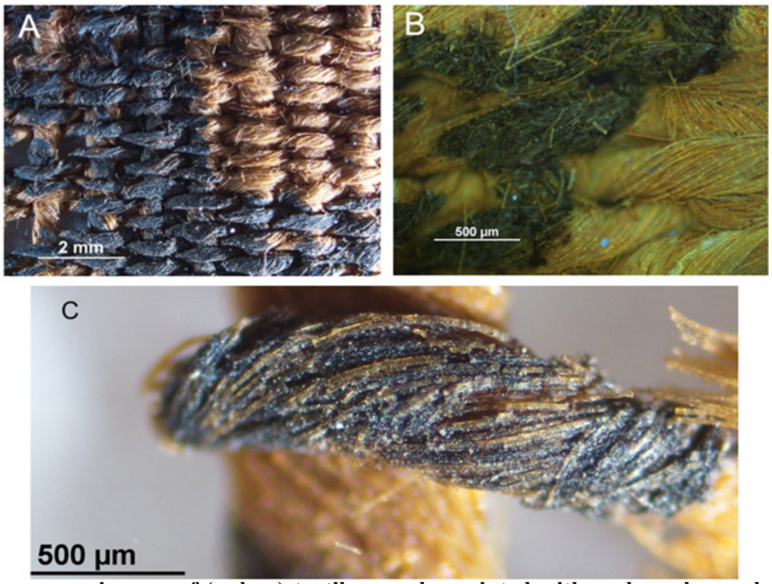

Fig. 4. UV-VIS miceroscope images of (a, b, c) textile samples painted with carbon charcoal. The images show cleary the mask and deposition of the particles on and in-between the fibre surfaces.

SEM-EDX Microscopy

The SEM micrographs of the experimental samples painted with calcite, charcoal and malachite [Fig.5] showed the deposition of pigments particles on and in- between the fibers, without the linen fibres being damaged. The results generally elucidate that the pigments act as a protective role as they protect the morphology of the linen fibres from the degradation caused by heat ageing, where the morphology of the fibres is completely covered by the pigment layers. The SEM photos of the experimental samples dyed with indigo, madder, and cochineal [Fig. 6] showed that the dye molecules are small enough to penetrate into the fibres without considerable changes in either the morphology or the weaving structure of samples. The characteristic scales of flax fibres are obvious, and only a few fibres are slightly damaged in a form of small scratches, transverse crack, and longitudinal split on the linen fibre surfaces. The conditions of the dyeing procedure may as well have a differential impact on the morphological features of dyed linen fibres. It is observed from EDX patterns in [Fig. 7, a, b, c] that the spectral profiles of the mockups colored with malachite, calcite and charcoal pigments are compatible with the existence of chemical elements that compose these pigments. EDX results of experimental dyed fibre samples are compliant with the presence of (Al, S, K) elements of alum mordant [Fig.7, d].

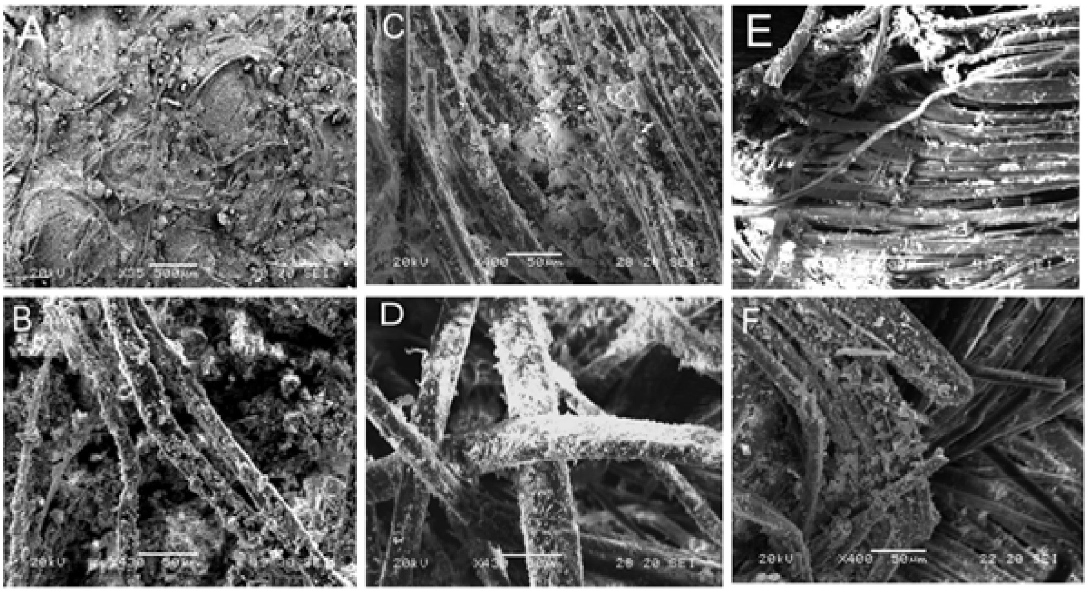

Fig. 5. SEM microphotographs of the painted textile samples with (a, b) malachite, (c, d) calcite, (e, f) black charcoal.The images illustrate the deposition of pigments particles on and in-between the fibers.

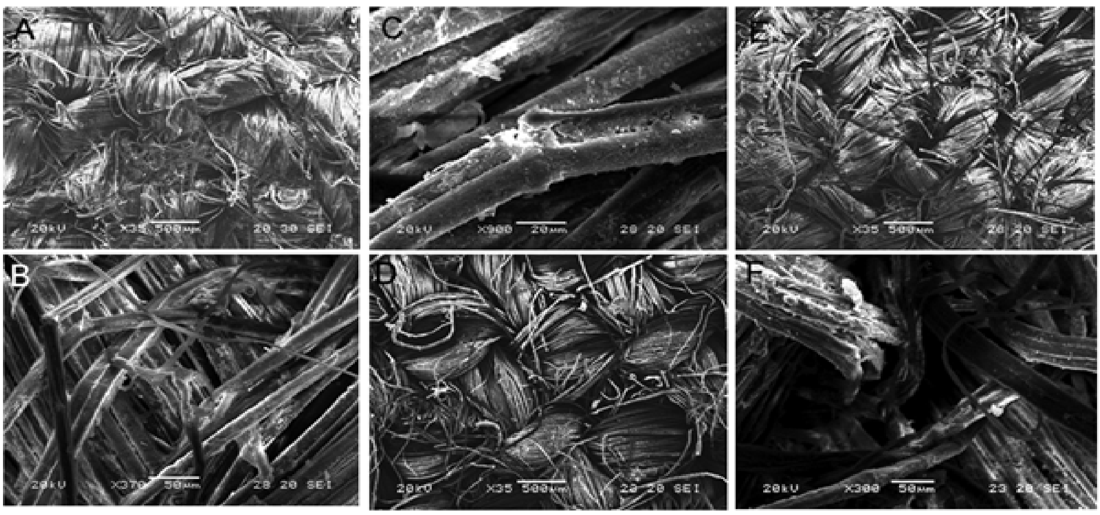

Fig. 6. SEM microphotographs of the dyed textile samples with (a, b) indigo, (c, d) madder, (e, f) cochineal, show that the dye molecules are small enough to penterate into the fibres without changing the fibre morphology. Also show minor degredation of the linen fiber surface after thermal ageing as small scratches and longitudinal splitting.

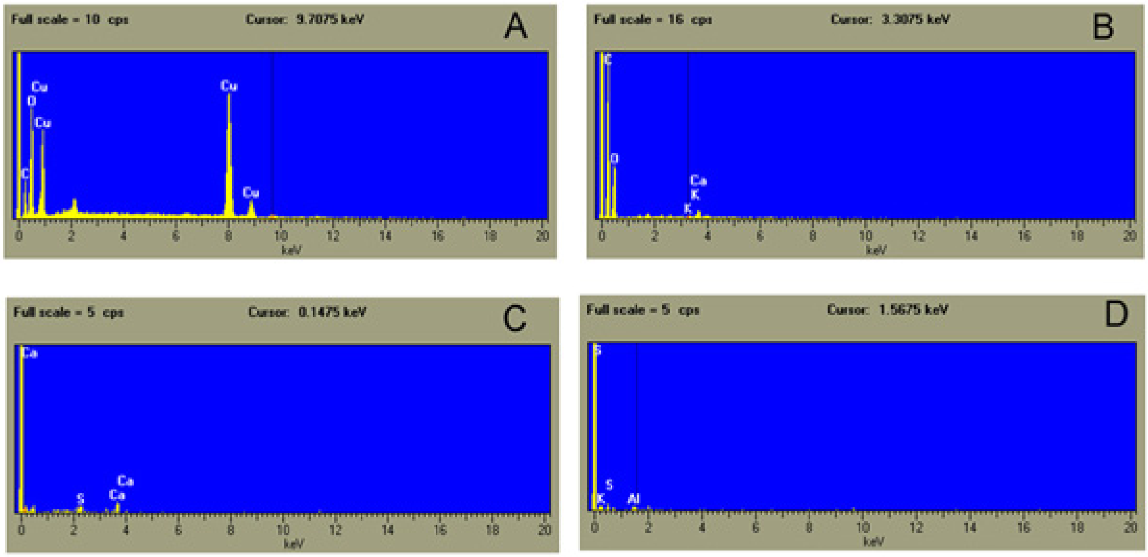

Fig. 7. EDX results of the experimental coloured textile fibres with (a) malachite, (b) black charcoal, (c) calcite and (d) madder.

Conclusion

Two fundamental methods for colouring textile materials are known in ancient Egypt, dyeing and painting, each of them require considerably different application methods because of its physical and chemical properties; it also leaves specific chemical signatures. The categorization of dyed and painted textiles made here makes it possible to draw analogies of different production techniques. This knowledge is essential for an understanding of the composition and morphological characteristics of the painted and dyed textiles and for a better understanding of their different production and deterioration; These are all essential for further investigations and conservation plans. The first important step of the study is the visual and microscopic examination. The results of these methods illustrate that the pigments act as a protective role as they protect the morphology of the linen fibres from the degradation caused by heat ageing. Nevertheless, natural dyes also protect and decreased the surface morphology deterioration of linen fibres.

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Conflicts of interest

The author declares no conflict of interest.

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المنسوجات الأثرية الملونة والمصبوغة : دراسة الاختلافات والخصائص المورفولوجية باستخدام تقنيات الفحص المجهري

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الهدف الرئيسي من هذه الدراسة هو التعرف على الفروق بين تقنيتين مهمتين لتلوين وتزيين المنسوجات في مصر القديمة ، وهما الصباغة والرسم وتأثيرهما على شكل المنسوجات الأثرية. في هذه الدراسة تم تحضير عينات تجريبية من منسوجات كتانية مصبوغة ومطلية العمر صناعياً مع بعض الأصباغ الشائعة والأصباغ الطبيعية المستخدمة في مصر القديمة ، للحصول على عينات مماثلة قدر الإمكان للعينات الأثرية. تمت دراسة العينات وفحصها باستخدام الفحص المجهري الإلكتروني المقترن بوحدة EDX (SEM-EDX) والفحص المجهري المرئي فوق البنفسجي (UV-VIS) أظهرت النتائج أن جزيئات الصبغة غير العضوية تترسب على أسطح الألياف وفيما بينها ، وتعمل كطبقة واقية للألياف ضد التدهور المورفولوجي الناتج عن التقادم . في المقابل ، جزيئات الصبغة صغيرة بما يكفي لاختراق وتلوين الألياف بشكل موحد. كما أنها قللت من تدهور الشكل السطحي للألياف بعد التقادم.