



Chemical Analysis For Assessing The Authenticity Of Archaeological Textiles: A Comprehensive Comparative Study Between Experimental And Antique Samples



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Abstract

This research presents a comprehensive comparative study aimed at authenticating archaeological textiles through the analysis of experimental and antique samples. The study employs meticulous examination techniques and advanced analytical methods to investigate the similarities and distinctions between an experimental textile piece and an authentic antique textile fragment, providing a robust framework for verification. A multidisciplinary approach is adopted, incorporating visual examination, microscopic analysis, chemical characterization, and non-invasive imaging techniques. An experimental textile sample, designed to replicate historical textile properties, serves as a controlled reference for comparison. Both the experimental and antique textile samples undergo various analytical tests, including fiber identification, dye analysis, weave pattern analysis, and structural assessment. The study aims to identify unique characteristics that reliably differentiate genuine archaeological textiles from modern imitations or reproductions. The findings carry significant implications for the authentication of archaeological textiles, contributing to the development of standardized criteria and methodologies. Through the comparative analysis, intricate material details and distinctive features emerge, aiding in the discernment of original artifacts from counterfeit counterparts. The research highlights the importance of scientific analysis in the field of archaeological chemistry and its role in preserving cultural heritage.

Keywords: Archaeological textiles; Counterfeiting; Linen; Thermal aging; Weave structure

1. Introduction

Textile artifacts are abundant in museums, storage facilities, and excavation sites, representing an invaluable national treasure. The materials of these artifacts vary in terms of fibers, dyes, coloured substances, and even inks used for writing. With significant advancements in scientific methods of examination and analysis to identify these components, methods of imitation and forgery have also evolved for all types of materials previously mentioned. These methods include employing various aging techniques and utilizing techniques and materials that can rival the original ones. As a result,

the scope of imitation and forgery has expanded, whether in terms of fibers, dyes, coloured materials, or even inks, to give the imitated pieces an archaeological character. This highlights the importance of the study, shedding light on the authenticity of these textiles. This study can unveil the methods of forgery and imitation, as well as reveal means of detection through adhering to principles, rules, and various analysis and examination methods.

Textile forgery, the act of creating counterfeit textiles to deceive collectors, researchers, and the market, has become a growing concern in the realm

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of cultural heritage and antiquities. Forgers employ various techniques to mimic historical textiles, challenging the ability to discern genuine artifacts from skillful imitations. Many methods were used by forgers for textile forgery like Aging Techniques that Forgers often use accelerated aging methods to artificially age textiles, making them appear older than they actually are. These techniques involve exposure to chemicals, light, and environmental conditions to induce discoloration, wear, and degradation characteristic of antiquity. In addition, dye replication, replicating historical dyes is a common method. Forgers attempt to match the colors and fading patterns of authentic textiles, sometimes using natural dyes or applying post-dyeing treatments to mimic age. Weave Reproduction: Skilled forgers aim to reproduce the intricate weave patterns found in historical textiles. This involves mimicking the weaving techniques, thread counts, and designs characteristic of a specific era. Distressing and Damage: Forgers may intentionally distress textiles by fraying edges, adding faux stains, and simulating insect damage to create the appearance of age and use.

Therefore, the authenticity assessment of archaeological textiles has become a critical issue in the field of antiquities. Previously, scholars categorized Egyptian antiquities as Pharaonic or Egyptian, Greek, and Islamic artifacts, enabling experts to distinguish between them [1]. However, the emergence of counterfeit artifacts, including textiles, has raised concerns regarding the ability to differentiate between genuine pieces and their replicas [2]. Initially, reproductions were distinguishable from authentic antiquities, as restorers and professional artists legally produced them without the intention to deceive [3]. Nevertheless, the rise of counterfeit manufacturing techniques, such as accelerated industrial aging and advanced examination and analysis tools, has presented challenges in identifying forged artifacts with identical materials and appearances [4]. There are many approaches utilized to verify the authenticity of archaeological textiles. Multidisciplinary Analysis: Authenticity assessment often involves a combination of techniques such as visual examination, microscopy, spectroscopy, and chemical analysis. These methods can reveal material composition, dye types, and manufacturing techniques. Microscopic Examination: High-resolution microscopy, including optical and electron microscopy, allows for the detailed analysis of fiber structures, surface features, and aging effects. Dye Analysis: Chemical analysis of dyes using techniques

like high-performance liquid chromatography (HPLC) can determine the presence of authentic historical dyes. Radiocarbon Dating: In cases where organic material is present, radiocarbon dating can provide a direct estimation of the textile's age, aiding in authentication. Comparative Analysis: Comparing the textile in question to authenticated samples from the same era can help identify discrepancies in material, weave, and dye characteristics.

The prevalence of counterfeit antiquities has led to heightened international efforts to detect and combat forgery, safeguarding the integrity of historical artifacts [5]. Skilled forgers can meticulously replicate intricate details, including signs of natural aging and material composition, making it increasingly difficult to detect their counterfeit creations [6]. It is important to distinguish between forgeries, which involves creating replica models using modern materials, and counterfeiting, which involves modifying and disguising falsehoods as genuine artifacts [7]. In the case of archaeological textiles, forgers may add decorations or apply old dyes to modern fabrics, attempting to forge proof of a specific era [8]. Conversely, legitimate copying processes, carried out by professional artists and restorers, aim to reproduce antiquities for educational or legal sale purposes [8]. To address the challenges posed by counterfeiting and forgery, this study focuses on comparing an antique textile piece with artificially aged fabric. Electron microscopy, optical microscopy, and other analysis methods will be utilized to conduct the comparison and assess the distinguishing features between the authentic and counterfeit textile samples. The outcomes of this research are crucial for establishing effective methods of authenticity verification in archaeological textiles. By identifying unique characteristics and markers of genuine artifacts, this study contributes to the development of standardized criteria and methodologies in the field of authenticity assessment. It also aids in preserving the historical significance of archaeological textiles and ensuring their accurate representation within cultural and educational contexts.

2. Materials and Methods

2.1. Technical Description of the Experimental Piece

The experimental piece utilized in this study consisted of natural raw linen fabric. The fabric's specifications are detailed as following:

- Fabric Type: 100% natural raw linen
- Weaving Structure: Plain 1/1
- Fabric Colour: Ranging from white to yellow

- Number of Warp Threads: 30/cm
- Number of Weft Threads: 23/cm

To replicate the dyeing techniques commonly observed in Egyptian museums, natural dyes were chosen. Specifically, a red color was extracted from the Madder plant. Ferrous sulphate was employed as the anchoring material in the experimental piece.

2.2. Dyeing Process for the Experimental Piece

To prepare the experimental pieces, a total of eight linen cloths were selected, each measuring 1 meter by 1 meter and weighing 337 grams. Prior to dyeing, the linen cloth underwent a thorough cleaning process to eliminate impurities, dust, and any water-soluble materials. The cloth was washed with running water and immersed in a washing bath containing soap and water. It was then heated on a stove, ensuring the temperature remained below 75°C, and stirred consistently for half an hour [9].

For the dyeing bath, water was added at a rate of 333 cm³, and a dye solution of 666 cm³ was prepared and heated on a stove. The linen cloth was placed in the dyeing vessel and left for 1.5 hours, with continuous stirring to ensure proper distribution of the dye solution over the fabric. Throughout the process, it was crucial to maintain a temperature below 75°C and monitor the evaporation to maintain the water proportion in the bath [10].

To extract the dye from the Madder plant (*Rubiatinctorum*), approximately 100 grams of Madder roots were weighed and thoroughly washed with water to remove any impurities. The plant material was then soaked in approximately 1000 cm³ of water for 24 hours, allowing the dye compounds to dissolve in the water. This soaking process helps in extracting the dye molecules from the plant material. After the soaking period, the mixture of soaked Madder and water was subjected to boiling for two hours while maintaining a careful control of the temperature below 65°C. This temperature control is important to prevent the degradation of the dye compounds. During the boiling process, small amounts of water were added as needed to compensate for evaporation caused by the elevated temperature, ensuring the solution maintains its desired concentration. Following the boiling, the solution was allowed to cool naturally at room temperature for about an hour. This cooling period helps in the further interaction of the dye molecules with the liquid, enhancing the dye extraction process. Subsequently, the solution was filtered to separate and remove any solid particles or impurities, resulting in a pure dye solution ready for use in dyeing applications (Figure 1) [11].



Figure 1: Experimental piece after dyeing

2.3. Thermal Aging of the Experimental Piece

In a study by Feller, thermal aging was conducted at a temperature of 100 °C for 72 hours to simulate the rate of damage or deterioration that would typically occur over approximately 25 years of natural aging under normal conditions [12]. Accelerated aging refers to the process of intentionally subjecting materials to conditions and factors that significantly increase damage, leading to a breakdown in their properties. Thermal aging is commonly employed to assess the stability of restoration and strengthening materials, as well as the changes they undergo over time.

In the case of the linen samples being studied, an accelerated aging process was carried out at a temperature of 100 °C for a period of 9 days. This duration was chosen to ensure the samples experienced the maximum degree of damage within the controlled environment of the restoration laboratory at Cairo University [13].

2.4. Visual Examination

Visual examination, conducted with the naked eye, is a fundamental method used to identify decorative styles, detect visible signs of damage, and determine whether the observed damage is a natural result of aging. This examination is typically performed prior to further detailed analysis and serves to distinguish the artifact from modern experimental pieces. It involves assessing the histological structure of the textile, including the number of warp and weft threads per centimeter, as well as examining the direction of twisting of these threads. Additionally, the visual examination includes a tactile component, where the fabric is handled to evaluate its fragility and susceptibility to fiber splitting.

2.5. Digital Microscopy Examination

The digital microscopy examination was employed to determine the fiber type of the textile piece and to identify the weaving technique employed, establishing its relationship to the corresponding historical era. This examination

involved comparing the textile under study with authenticated artifacts from museums or excavation sites using the same microscope. By scrutinizing the fibers, it was possible to discern the specific method of fiber twisting and to investigate subtle damage manifestations that may not be visible to the naked eye, utilizing different levels of magnification.

2.6. Scanning Electron Microscope Examination

The scanning electron microscope (SEM) was employed to analyze the fabric fibers and investigate key indications of fiber damage. This examination aimed to differentiate between naturally occurring aging effects and artificially induced damage. It also sought to determine the nature of cuts observed on the fabric, discerning whether they resulted from inherent fragility due to age or deliberate use of sharp tools to mimic original characteristics.

2.7. Infrared Spectroscopy

Infrared spectroscopy, specifically Fourier Transform Infrared Spectroscopy with Attenuated Total Reflectance (FTIR-ATR), was employed to analyze the textile piece. This technique allowed for the identification of the dyes present in the fabric and comparison with modern samples. Additionally, it facilitated the examination of the fundamental components of the textile, such as hemicellulose, and provided insights into the chemical changes occurring in the textile fibers.

2.8. Ultraviolet Imaging

Ultraviolet imaging was employed to photograph both the experimental textile piece and the artifact. This technique utilized ultraviolet rays to detect potential textile counterfeiting. By focusing ultraviolet light on the textile, any emitted light or fluorescence could be observed, aiding in the identification of counterfeit materials or alterations.

2.9. Color Change Measurements

Color change measurements were performed on the dyed linen samples following thermal aging, as well as on the artifact under investigation. The color parameters were measured and represented by the following values:

L: Represents the lightness component, ranging from 0 to 100, where higher values indicate lighter colors and lower values indicate darker colors. In this study, L represents the difference between whiteness (100) in the positive direction and darkening (-100) in the negative direction.

A: Represents the red-green color component. Positive values indicate more redness, while negative values indicate more greenness. A does not represent the difference between red and green colors, but

rather the position along the red-green axis.

B: Represents the yellow-blue color component. Positive values indicate more yellowness, while negative values indicate more blueness. B does not represent the difference between yellow and blue colors, but rather the position along the yellow-blue axis. [28]

These color parameters were utilized to quantify and assess the extent of color changes in the textile samples and the artifact. By analyzing these values, valuable insights were obtained regarding the effects of thermal aging and potential alterations in the original color characteristics.

2.10. XRD Analysis

X-ray diffraction analysis was used to determine the degree of crystallinity of cellulose in both the archaeological and experimental samples. This was conducted to study the impact of natural aging and industrial aging on the crystalline structure of cellulose. Natural aging leads to a reduction in the crystallinity of cellulose. The degree of crystallinity of cellulose was determined using X-ray diffraction by comparing the distinct reflection intensities of the crystalline structure of cellulose with the intensity of the non-crystalline region. One of the most common methods for measuring the crystallinity of cellulose is the Segal method. This method involves analyzing a sample of cellulose (linen) using X-ray diffraction at angles ranging from 10° to 40°. The measurements are taken at an angle of 18° for peak height and an angle of 22.6° for reflection intensity. The comparison between these measurements is performed using the Segal equation for crystallinity:

Cr

Where (Cr) represents the degree of cellulose crystallinity, (I002) is the maximum peak height at angles between 22° and 24°, commonly around 22.6° ± 0.2, and (Iam) stands for the height of the diffraction region (the low region between the reflections at angles 22°-24° and 14°-16°) from the baseline at 18°, which is distinctive for non-crystalline materials. [27]

3. Results

3.1. Visual Inspection

Visual inspection of the artifact revealed distinctive characteristics of natural antiquity. One noticeable aspect was the fading of the colors used in dyeing the artifact, which appeared distinct and significantly different from the experimental fabric pieces. Despite attempts to simulate industrial

obsolescence, the colors of the experimental pieces remained clearer compared to the artifact's dyes. This distinction is clearly illustrated in Figure 2. Furthermore, the artifact exhibited color defects, with certain areas showing pigment disappearance and others displaying a significant degree of fading [14].

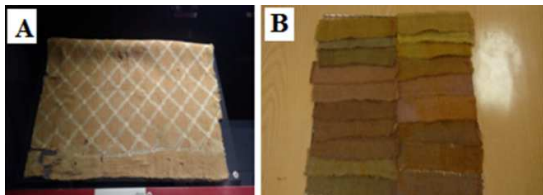


Figure 2: Comparison between an Artifact (A) and an Experimental Piece (B)

3.2. Examination with a Digital Microscope

The digital microscope examination revealed that the tissue structure of the piece is 1/1 plain, as depicted in Figure 3. Furthermore, the examination confirmed that the fibers composing the piece are made of linen [15].



Figure 3: Shows the weaving structure of both the artifact (A) and the experimental piece (B) at a magnification of 10X

3.3. Scanning Electron Microscope Examination

The scanning electron microscope (SEM) examination conducted on the fabric samples revealed various types of damage present on the surface and between the fibers, as illustrated in Figure 4. This examination provided valuable insights into the extent of dirt penetration between the fabric fibers and offered a detailed visualization of the fabric's surface. These observations were crucial for distinguishing between the original artifact and the textile piece subjected to thermal aging [16].

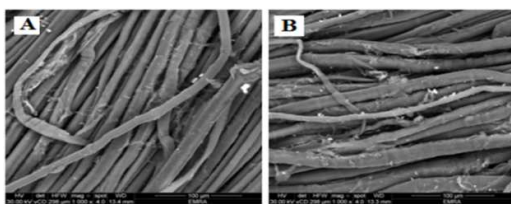


Figure 4: Shows the results of the scanning electron microscope examination conducted on an archaeological sample (A) and an experimental piece (B)

3.4. Fourier Transformation Infrared Spectroscopy (FT-IR)

Figure 5 presents the FT-IR spectra of the antique piece, revealing distinctive features. Within the 1500-1800 cm^{-1} range, a prominent peak is observed, indicating the presence of carbonyl groups resulting from the degradation of cellulose fibers. Furthermore, a characteristic peak within the 3600-3900 cm^{-1} range corresponds to the hydroxyl group (OH) present in linen fibers. In contrast, Figure 6 displays the FT-IR spectra of the experimental piece. The spectra demonstrate the persistence of hemicelluloses, as these compounds require more time for degradation to occur and eventually disappear [17].

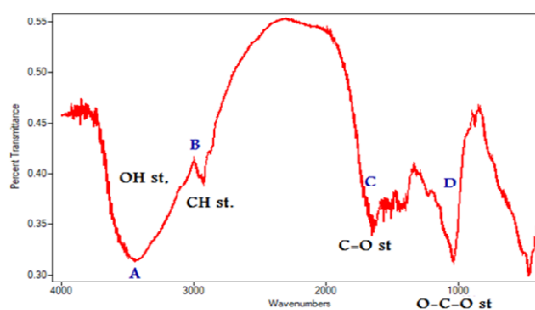


Figure 5: shows the results of spectroscopic analysis, specifically changes in the FT-IR spectrum of the archaeological piece.

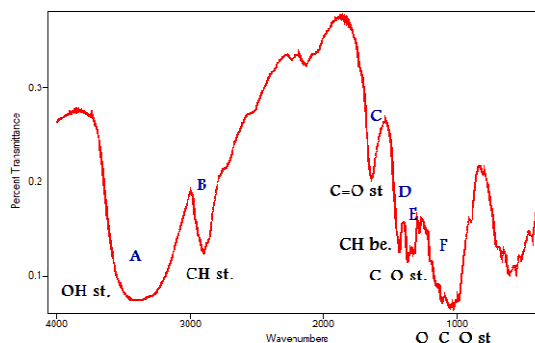


Figure 6: Shows the results of spectroscopic analysis, specifically changes in the FT-IR spectrum of the experimental piece.

3.5. Ultraviolet imaging

Ultraviolet imaging revealed that the artifact appears in a light color, as demonstrated in Figure 7. This result contrasts with the purple color observed when photographing antique artifacts using this imaging technique.

The image of the artifact captured under ultraviolet light reveals a distinct light color, as depicted in the figure. In contrast, when ultraviolet imaging is performed on antique artifacts, a purple color tends to be observed. This disparity in

coloration between the artifact and other pieces becomes evident through the use of ultraviolet imaging.



Figure 7: illustrates the contrasting results obtained from ultraviolet imaging of the artifact and the experimental piece.

3.6. Color change measurements

The color change measurements, as shown in Table 1, demonstrate noticeable differences between the artifact and the experimental pieces. The artifact exhibited a color change measurement of 65.41, indicating a significant degree of fading. This value significantly deviates from the color change measurements observed in the experimental pieces, which experienced varying degrees of darkening. The data presented in Table 1 clearly highlight the distinct color changes undergone by the artifact compared to the other experimental samples.

Table 1
Color parameter changes for the samples

Sample	Color parameters		
	A	B	L
Linen + madder Without ageing	6.62	7.7	59.47
Linen + madder With thermal ageing	7.75	13.43	56.11
Antique piece	5.2	16.21	65.41

These measurements provide valuable insights into the color parameter changes experienced by both the artifact and the experimental samples, highlighting the significant fading observed in the artifact compared to the darkening observed in the experimental pieces.

The results presented in Table 1 demonstrate notable differences in color parameter changes among the samples, including the artifact and the experimental pieces.

For the Linen + Madder samples without thermal aging, the A (red-green) and B (yellow-blue) values indicate slight changes, suggesting minimal shifts in these color components. However, the L (lightness)

value shows a decrease from 59.47 to 56.11, indicating a slight darkening of the samples.

In contrast, the antique piece exhibits more significant color changes. The A value decreases to 5.2, indicating a shift towards green, while the B value increases to 16.21, indicating a shift towards yellow. These changes suggest a noticeable alteration in the red-green and yellow-blue color components. Additionally, the L value decreases to 65.41, indicating a significant darkening of the antique piece.

Overall, the results suggest that both the experimental samples and the artifact have undergone some color changes. However, the artifact shows a distinct pattern of fading, as indicated by the significant decrease in the L value. This suggests that the artifact has experienced more pronounced color alterations compared to the experimental pieces, which primarily exhibited slight darkening. These findings provide valuable insights into the effects of thermal aging and the potential color shifts in the original characteristics of the artifact.

3.7. Color change measurements

The analysis results of the modern experimental sample revealed that the distinct reflection of cellulose appeared at an angle of 23.1 with an intensity of 3670. On the other hand, in the archaeological sample, the distinctive cellulose reflection appeared at an angle of 22.6 with an intensity of 1485. This indicates a decrease in the degree of cellulose crystallinity due to deterioration caused by natural aging of the artifact.

To utilize this type of analysis for detecting textile forgery, it is essential to have a registered database of cellulose crystallinity results from numerous textile pieces within our museums and storage facilities. This database would serve as a guide and reference for forgery detection experts, aiding them in identifying potential forgeries.

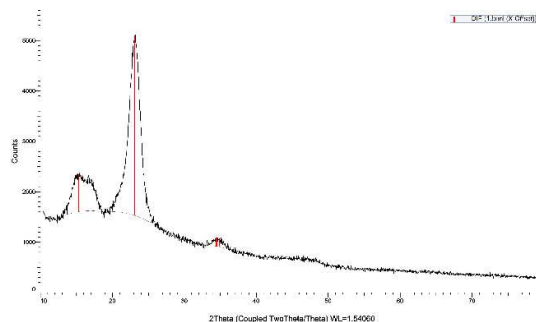


Figure 8: Illustrates the X-ray diffraction pattern of the experimental sample.

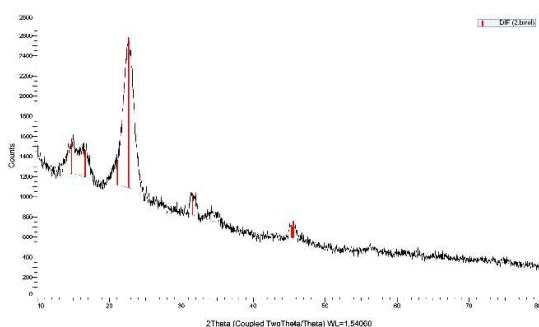


Figure 9: Illustrates the X-ray diffraction pattern of the archaeological sample.

4. Discussion

The visual examination of the fabric piece revealed that it was executed in the style of batik decoration, which commonly involves the use of different colors and the application of wax to cover unwanted areas during the dyeing process. The archaeological fabric appeared more fragile and exhibited fiber splitting compared to the experimental pieces, indicating signs of natural aging [18]. Furthermore, the artifact exhibited noticeable fading of the dye colors, which differed significantly from the experimental fabric pieces. The artifact also displayed areas where dyes had disappeared or faded considerably. These observations, along with the manifestations of natural age-related damage, provided valuable insights into the authenticity of the artifact [19].

Through digital microscope examination, the spinning and weaving processes used in the textile industry were studied. The microscopic analysis of imitation pieces allowed for the determination of the era to which the fabric piece could be attributed, as weaving techniques varied across different time periods. This method proved to be a significant factor in authenticating the artifact, as weaving techniques exhibited distinct characteristics, such as the use of different thread types, twisted threads for a wrinkled surface, or variations in the combination of warp and weft threads [20].

Stains on the textile piece, their shape, extent of spread, and the behavior of dyes provided additional evidence. These stains indicated the unintended migration of dyes during the dyeing process, specifically in the batik style. The dyeing method observed in the artifact differed from the experimental pieces, where the dye had fully penetrated the fabric. Counterfeiters often use modern materials to replicate the dyeing process, resulting in a different appearance compared to the original antique weave [21].

Examination using a scanning electron microscope confirmed that the textile piece was made of linen fibers. The detection of modern synthetic fibers or other natural fibers that were not used in the past is an important aspect of identifying the quality of textile fibers [22]. The damage observed on the artifact, as opposed to the experimental piece, indicated that it was more likely caused by thermal aging rather than natural wear and tear over time.

FT-IR analysis revealed the use of madder dye in both the experimental and archaeological pieces. This analysis helped identify the types of pigments and binding agents used in each case and determine their compatibility with the era to which the artifact is attributed. The use of madder dye in the research aided in accurately comparing the results and validating the findings [23].

The analysis also focused on hemicellulose, a component of the fabric's composition. It was observed that hemicellulose had disappeared from the archaeological tissue due to its weaker composition compared to cellulose, which remains present. This finding serves as an important indicator of the authenticity of the archaeological fabric. Hemicellulose takes longer to be affected by natural damage, making its analysis crucial in revealing the authenticity of the artifact [24].

Ultraviolet photography played a significant role in distinguishing the artifact from counterfeits. The artifact appeared in a light color when photographed under ultraviolet light, whereas counterfeit artifacts typically exhibited a purple color under the same conditions [25]. This technique provided valuable visual evidence to differentiate between genuine and counterfeit pieces. [29]

The measurements of color change further supported the distinction between experimental and authentic pieces. These measurements indicated that textiles, being a naturally weak material compared to other archaeological materials, are highly susceptible to industrial aging processes. The artifact displayed more pronounced and permanent deterioration compared to the experimental pieces, emphasizing the challenge of artificially aging textiles [26].

5. Conclusions

This study aims to provide a methodology for effectively distinguishing between original artifacts and modern textile counterfeits that are used to deceive buyers and falsely claim their antiquity. To achieve this objective, several tests and analyses were conducted, including the utilization of the digital microscope, scanning electron microscope (SEM), and FTIR spectroscopy for pigment analysis. Additionally, the coloring of the artifact was examined to gain insights into the pigments used

during the ancient Egyptian era, their manufacturing techniques, and the study of damage manifestations.

By employing these diverse analytical techniques, this study aimed to establish a robust approach to authenticate artifacts. By comparing the obtained results with established historical knowledge and genuine artifacts, researchers can make informed assessments regarding the authenticity of the textile pieces. This comprehensive methodology serves as a valuable tool in preventing counterfeiters from deceiving potential buyers, providing a scientific basis to verify the genuineness of the artifacts. The findings from this study contribute to the ongoing efforts in preserving cultural heritage and safeguarding the integrity of the antiquities market.

6. Conflicts of interest

There are no conflicts to declare.

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