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The usage of AgNPs/Klucel G Nanocomposites And Some Conventional Materials in The Conservation of Archaeological Linen Textile Remains

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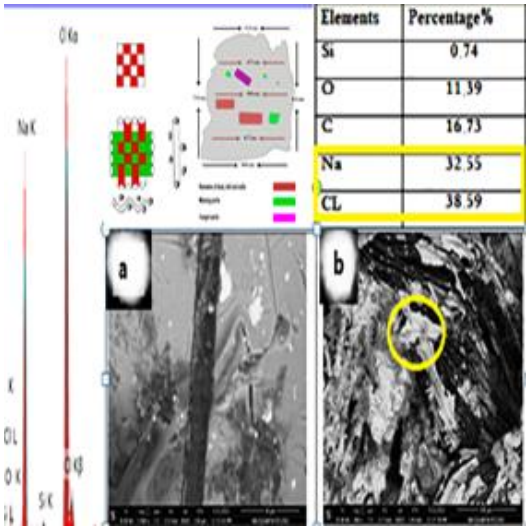
HIGHLIGHTS

- Documenting and diagnosing the degradation aspects of the artifact using various examination and analysis methods.
- Evaluating the effectiveness of the surfactant and nanocomposites in the treatment and conservation of the archaeological piece.
- Clarifying the condition of the archaeological linen piece before and after the treatment process.

GRAPHICAL ABSTRACT



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ABSTRACT

Treatment and conservation operations are considered the most important processes for preserving cultural heritage collections, in particular archaeological textiles. This research outlines the methods used for treating and preserving an ancient piece of linen textile recovered from the Qusayr excavations in the Red Sea. The piece was cleaned using the traditional detergent "Orvus WA Paste surfactant", and then, it was consolidated to prevent damage and combat the microbiological

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attack using silver nanoparticles loaded on hydroxypropyl cellulose polymer (AgNPs/Klucel G). Various methods were employed to diagnose the decay of the artifact. Scanning electron microscope coupled with an energy-dispersive X-ray unit (SEM-EDX) was used to scrutinize the surface morphology and determine the elemental composition of the archaeological piece. X-ray diffraction (XRD) was employed to ascertain the rate of cellulose crystallization of the piece. According to the results; it was deduced that, the artifact was found in an area near the sea, which was confirmed by the presence of sodium chloride salt residues on its surface. The microbiological study demonstrated that AgNPs/Klucel G was effective in inhibiting fungal strains that were isolated from the artifact.

1. Introduction

The conservation process of archaeological textiles is considered one of the most important procedures, providing social, commercial, and artistic evidences about different civilizations. The main basis for conservation of the archaeological textiles is to preserve them for the longest possible period of time against deterioration factors that may affect them during display or storage.[1, 2] Conservation processes comprise cleaning, disinfection, consolidation, and storage. [3-5] In the past, the process of conservation of archaeological textiles was carried out without following any scientific rules. It was done by sewing or by methods similar to household washing.[6] This research discusses the stages of treatment and conservation of the remains of an ancient piece of linen fabric that was discovered in the Qusayr excavations in the Red Sea Governorate. The study recorded the archaeological item and identified the factors causing its deterioration by examining and analyzing it. The artifact was also preserved by cleaning it with a surfactant to remove dust and dirt, and by treating it with silver nanoparticles loaded on hydroxypropyl cellulose polymer (AgNPs/Klucel G) to prevent microbiological damage. Ionic surfactant detergents have been used to wash many archaeological textiles. Orvus WA surfactant, produced by FLUKA Company, was used to wash some pieces of dyed Bedouin textiles preserved in the Museum of Civilization in Jordan. It was used at a concentration of 1g/L, and SCMC sodium carboxymethyl cellulose was used with it at a concentration of 0.1g/L, dissolved in deionized water as a dirt-carrying agent.[7] Orvus WA surface cleaner was used at a concentration of 0.1% to clean a piece of carpet after treating it with dimethyl formamide (DMF), and removing the glue present on the surface of the carpet. The carpet was immersed in the cleaner after treating it with dimethyl formamide. The washing bath contained SCMC.[8] Nano materials have recently attracted great attention because of their unique properties, as they have high surface area relative to their volume, and thus they have unique chemical, electronic, and physical properties.[9] Silver particles are considered a nanometer engineering material that in recent years has attracted the attention of many researchers to study its effects and risks on the environment and humans. Silver (Ag) and silver nanoparticles (AgNPs) have been used in many consumer products that humans use, such as the textile industry, in order to give them the functions of resisting microbes and controlling the unpleasant odors. For resisting the growth of microbes on textiles, this can be carried out by adding antimicrobials or by incorporating biocides during the processing processes of the fibers used in the manufacture of these textiles.[10] Silver Nano particles loaded on hydroxypropyl cellulose polymer (AgNPs/Klucel G) were used in an experimental study to combat microbiological damage to linen textiles for some fungal strains isolated from the exhibition and storage halls of Sohag National Museum. The results of the study showed the high effectiveness of AgNPs/Klucel G in inhibiting fungal strains as well as in improving mechanical, physical, and morphological properties of linen textiles.[11]

2. Materials and methods

2.1. Materials

- Silver Nano particles AgNPs 3% prepared by chemical reduction of silver nitrate

$AgNO_3$ in the presence of polyvinylpyrrolidone polymer (PVP) as a stabilizing agent and loaded on hydroxypropyl cellulose polymer (Klucel G) added to hydrogen peroxide H_2O_2 as a bleaching agent at a concentration of 3%.

- Ionic surfactant Sodium Lauryl Sulphate (Orvus WA Past), produced by Talas USA.
- Sodium carboxymethyl cellulose SCMC, produced by Al-Gomhouria Chemicals Company- Egypt.
- Potato dextrose agar (PDA) used as a nutritional medium for fungi, produced by the HIMEDIA REF Company- Indian.
- Nutrient agar as a nutritional medium for bacteria, produced by Techno Pharmachem, Bahadurgarh, Haryana-India.

2.2. Methods

2.2.1. Documentation and scientific study of the archaeological piece

Many tools have been used to document the dimensions of the piece and its condition before treatment such as magnifying glasses, digital cameras, in addition to using a USB digital microscope.

2.2.2. Examination and analysis procedures

2.2.2.1. Identification of fibers by microscopic examination (optical microscope)

A Celestron - LCD digital microscope II equipped with a 5MP imaging unit, was used in the laboratory of organic materials, Faculty of Archeology, Sohag University, Egypt, in the process of examining the fallen remains of the archaeological samples in order to identify the type of fibers in which that piece was woven from. The examination process was carried out by placing the fallen samples from each piece in a small glass beaker containing distilled water, then placing the beaker containing the samples on a heating disc until the boiling point was reached for 20 minutes. Then the remaining parts of the pieces were transferred after the boiling process into a test tube with a capacity 20 ml of distilled water, shaking this tube well until the samples disintegrate and become in the form of small cones, then approximately 0.5 ml of those cones are taken with distilled water and placed on a glass slide and spread evenly on the slide, making sure that the water on the surface of the slide evaporates.[12-15] Then cover the slide with a designated examination cover so that the sample is ready for examination under the optical microscope.

2.2.2.2. USB digital microscope

A USB digital microscope of the type (Digital Microscope Rohs1600X 2 MP Zoom Microscope 8Led) was used to examine the weave structure and diagnose dirt and stains on the archaeological piece [16-18].

2.2.2.3. Scanning electron microscope (SEM) equipped with an EDX unit

Scanning Electron Microscope (Quanta FEG250 NRC) coupled with Energy-dispersive X-ray spectroscopy (EDX) was used to investigate the archaeological linen piece's surface morphology and identify the most important elements found on the fibers [19, 20].

2.2.2.4. Attenuated total reflection – Fourier transform infrared spectroscopy (ATR/FTIR)

ATR-FTIR was used to identify the distinct functional groups of the archaeological fibers. The analysis process was carried out using a Bruker Alpha device in the Microanalysis Unit at the Faculty of Science, Sohag University, Egypt. Instrument was configured with ATR sample cell including a diamond crystal with a scanning depth up to 2 micrometers. The spectrum was recorded using the absorption method over a spectral range of 400-4000 cm^{-1} with an accuracy of 4 cm^{-1} [5, 11, 12, 21].

2.2.2.5. X-ray diffraction

Bruker D8 Advance XRD devise was used to determine the rate of crystallization of cellulose polymer, the main component of archaeological fiber's piece. The measurement process was carried out by comparing the results of measuring the crystallization rates of samples dropped from the artifacts that were used in the analysis with a standard lin-

en sample with the same dimensions as the archaeological samples. The measurement were carried out at the (XRD) unit, Faculty of Science, Sohag University, at room temperature, and the scanning range of the 2θ angle was 10–30.

2.2.2.6. Microbiological study

Many microbial swabs were taken from the affected surfaces of the archaeological piece by scraping those using sterile medical swabs.[22] Petri dishes containing nutrient media were inoculated with Nutrient Agar bacteria and PDA fungi, and this stage was carried out in a sterilizing UV-type CHEM.TECH sterilization cabin. After the process of inoculating the plates with sterile medical wipes, they were incubated at 28-30 degrees for a period of time of 48-72 hours for Nutrient Agar media for bacteria and 1-7 days for PDA media for fungi.[23] The purified bacteria and fungi isolated from the artifact were identified according to their morphological characteristics using Olympus BX51 compound microscope equipped with Toup Tek XCAM1080PHA (Toup Tek, Zhejiang, China) digital imaging system at Microbiology Laboratory, Department of Botany, Faculty of Science, Sohag University.

2.2.3.The treatment and conservation of the archaeological piece

- **Wet cleaning of the archaeological piece**

The ionic surfactant detergent belongs to the primary alkyl sulfate family Orvus WA Paste and it produced by Talas USA used at a concentration of 1%. This class of detergents has good cleaning and high foaming properties.[24] The chemical composition is Sodium Laury Sulphate [25].

- **The microbial efficiency of AgNPs 3% / Klucel G 1%/H_2O_2**

Before applying the AgNPs/Klucel G/ H_2O_2, the effectiveness of the compound was measured against microbial strains isolated from the archaeological piece. The inhibition zone diameter of AgNPs/Klucel G/ H_2O_2 for microbes isolated were measured using a well-cut diffusion technique [26, 27].

3. Results and Discussion

3.1. Documentation and scientific registration of the archaeological piece

Through photography of the archaeological piece, it was found that it is the remnants of a piece of textile set aside for study as a result of the excavations of The English Mission in the city of Al-Qusayr in the Red Sea, and preserved in the museum store in the city of Qeft . Visual examinations and examinations using magnifying glasses, and the use of a measuring tape showed the dimensions and specifications of the archaeological piece as shown in Table 1. and Fig 1.

3.2. The results of examination and analysis of the fallen parts of the archaeological piece

3.2.1. Examination to identify the type of fibers

The light microscope was used to identify the type of woven fibers, including both the warp and weft threads of the archaeological textile piece. Through examinations, it was found that the warp and weft threads of the archaeological piece were woven from linen fibers, as shown in Fig. 2.

3.2.2. Examination to determine the weave structure and diagnose some of degradation aspects

A USB digital microscope was used to examine the weave structure and diagnose dirt and stains on the archaeological piece. Through the examination process, it was found that the weave structure of the archaeological piece was a plain weave 1/1. Also, through the examination, it was revealed that there was a group of loose dirt on the surface of the piece, as well as the presence of fungal

Table 1. Technical specifications of the archaeological piece extracted from the excavations of Al-Qusayr in the Red Sea.

Description and dimensions of the piece	The remains of an irregular piece of linen fabric. The dimensions of the piece are 8.5cm long on one side, 7.9 cm on the other side, and 5.5cm wide on one side, and 6.8cm on one side.
Type of fiber	Linen fibers as shown by microscopic examination
The number of warp threads in 1/2 inch	27 warp threads
The number of weft threads in 1/2 inch	32 weft threads in 1/2 in
Weave structure	plain weave 1/1
Weight /g	1.2899g
Thickness/mμ	21 μ m

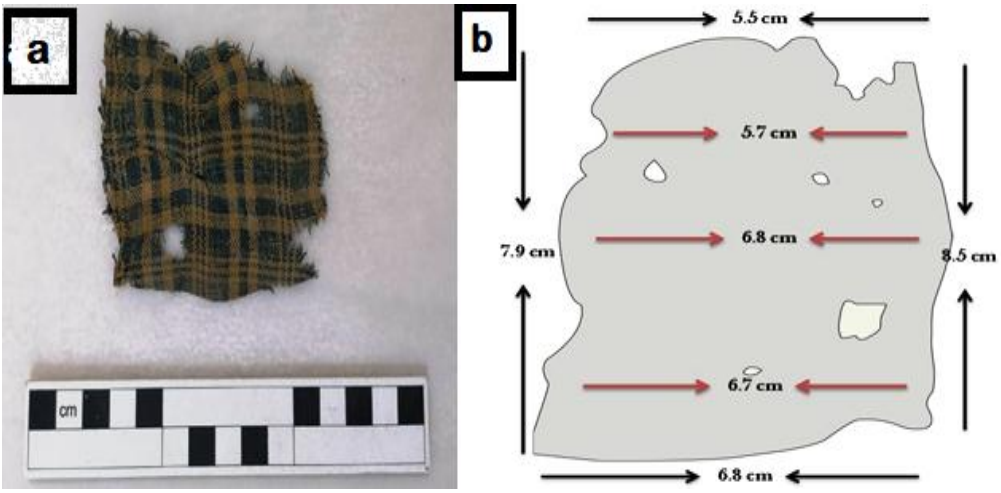


Fig. 1. The artifact where (a) the piece before the treatment process, (b) the piece shows the dimensions on each side.

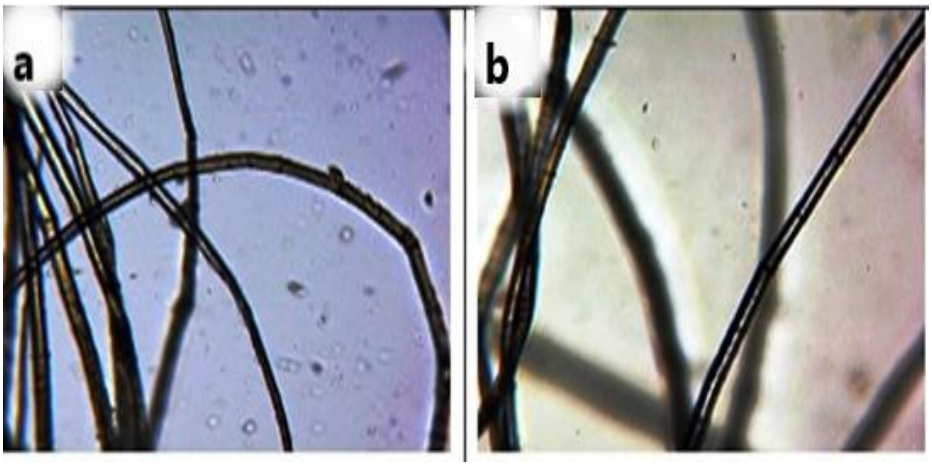


Fig. 2. The woven linen fibers, including the warp and weft threads of the archaeological piece, with light microscope at 30X magnification.

spots, in addition to many missing, irregularly shaped parts of the archaeological piece, as shown in Fig. 3.

3.2.3. Examination and analysis of the surface morphology of the archaeological piece using an SEM equipped with an EDX unit

Through the results of examination and analysis using SEM-EDX shown in Fig. 4. the results confirmed that the fibers from which the piece was woven are linen fibers. The results also showed high levels of chlorine (Cl) (38.59%) and sodium (Na) (32.55%). This is due to the proximity of the area from the Red Sea where the piece was discovered, which is rich in sodium chloride (NaCl). The appearance of silicon element (Si) is due to the nature of the sandy coastal environment in which the piece was discovered.

3.2.4. Results of spectroscopic analysis with ATR-FTIR spectrometer

The FTIR infrared spectrum in Fig. 5. showed the appearance of the spectral bands 432.70 cm^{-1} and 523.20 cm^{-1}, which express changes in the structure of the large molecules of the cellulose polymer. The bands 1095 cm^{-1}, 1335 cm^{-1}, as well as the 1430 cm^{-1} band, appeared these are bands that express decreased rates of cellulose crystallization, increased rate of dissolution of the fiber structure, and increased content of hydroxyl bonds (OH). The range 1380cm^{-1} also appeared which is a range that expresses the occurrence of a transformation in the cellulose polymer molecules, where the large cellulose units are transformed into single monomer units. This process is called depolymerization [28]. The spectral band 1642.94 cm^{-1} appeared which expresses the processes of hydrolysis of the cellulose polymer, the main component of the artifact's fibers.[29] The spectral bands appeared 1367.57 cm^{-1}, 1316.57 cm^{-1}, 2900.39 cm^{-1}, and all of these bands express the expansion and vibration of the C-H bond in polysaccharide materials such as the cellulose polymer, the main component of linen fibers.[30] The characteristic bands of the C-O carbonyl groups also appeared which are the bands 1104.49 cm^{-1} and 1025.71 cm^{-1}. The spectral band 3324.39 cm^{-1} also appeared which is a distinctive band for the hydroxyl groups in the cellulose polymer.

3.2.5. Measure of the crystallinity index of the archaeological piece

The crystallinity index of the archaeological linen piece is calculated according to the Segal peak height method a maximum intensity value I002 is found between the scattering angles of 2θ=23.2337° .The minimum value Iam is taken using a minimum in the data, typically between 2θ=18.9941°. Then the crystallinity index of cellulose polymer, the main component of linen fibers, was calculated for the archaeological piece according to the following equation:-

$$CI = \frac{IC - Iam}{IC} \times 100$$

Where CI expresses the Crystalline Index, IC represents the highest crystalline point and Iam represents the amorphous region in the archaeological piece [31, 32]. Through (XRD) analysis and the application of the previous equation in Fig. 6. and Table 2, it was found that there was a decrease in the polymerization index of cellulose polymer, the main component of linen fibers of archaeological piece, when compared to the crystallization index of the standard linen sample, as the cellulose crystallization index of the archaeological piece reached 75.1%, while the crystallization index of the standard linen sample was 94.4. % . A decrease in the crystallization indices of the artifact is confirmed by the appearance of some spectral bands in the analysis by infrared spectrometer, such as the spectral band 432.70

cm^{-1}, 523.20 cm^{-1}, 1430 cm^{-1} and the spectral band 1335 cm^{-1}, which are bands

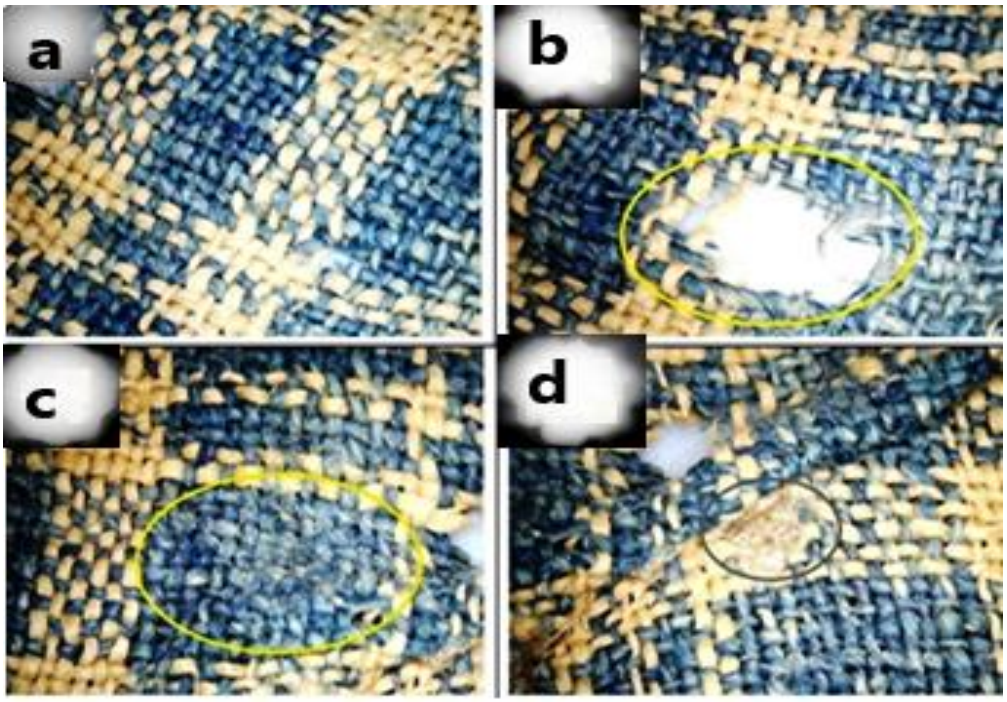
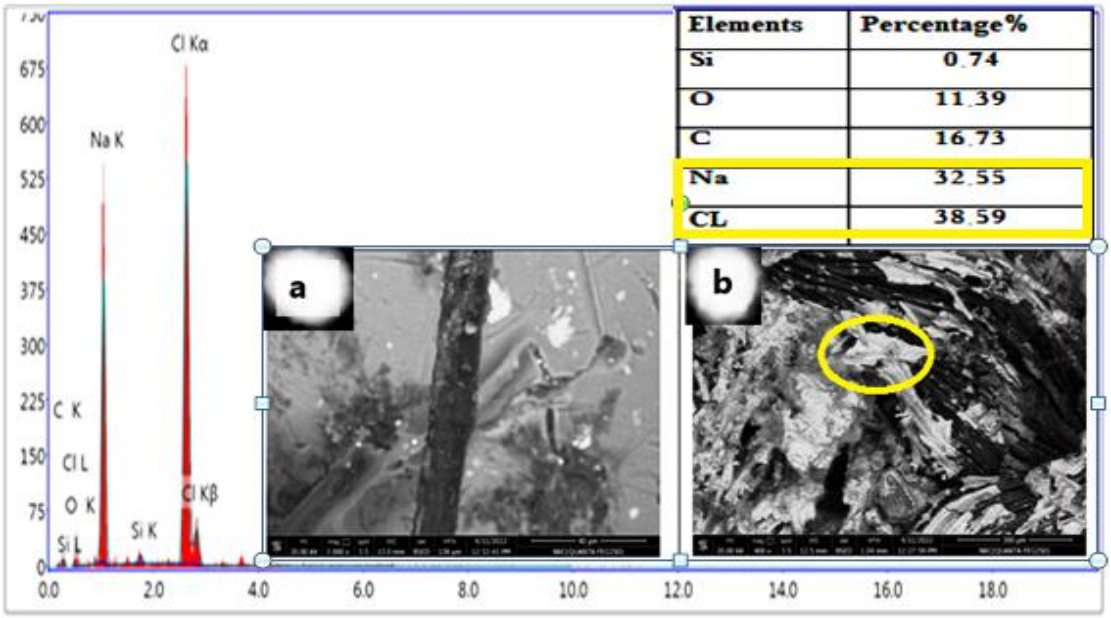


Fig. 3. At 30x magnification using a USB digital microscope of the archaeological linen piece, where (a) shows the plain 1/1 weave structure , (b) shows the missing parts, (c) loose salts and dirt, while (d) represents the fungal spots on the archaeological piece.



Elements	Percentage%
Si	0.74
O	11.39
C	16.73
Na	32.55
CL	38.59

Fig. 4. The results of the EDX analysis of the archaeological piece and the SEM examination, which shows the accumulation of sodium chloride salt on the morphological surface of the archaeological piece (a and b).

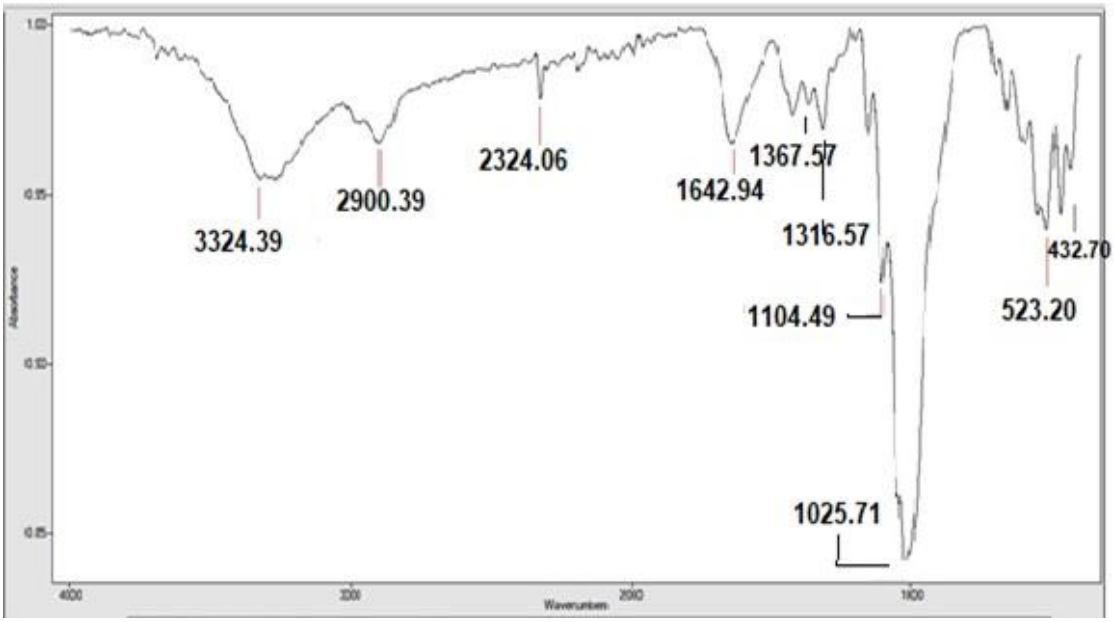


Fig. 5. ATR-FTIR spectrum of the archaeological sample extracted from the Qusayr excavations in the Red Sea.

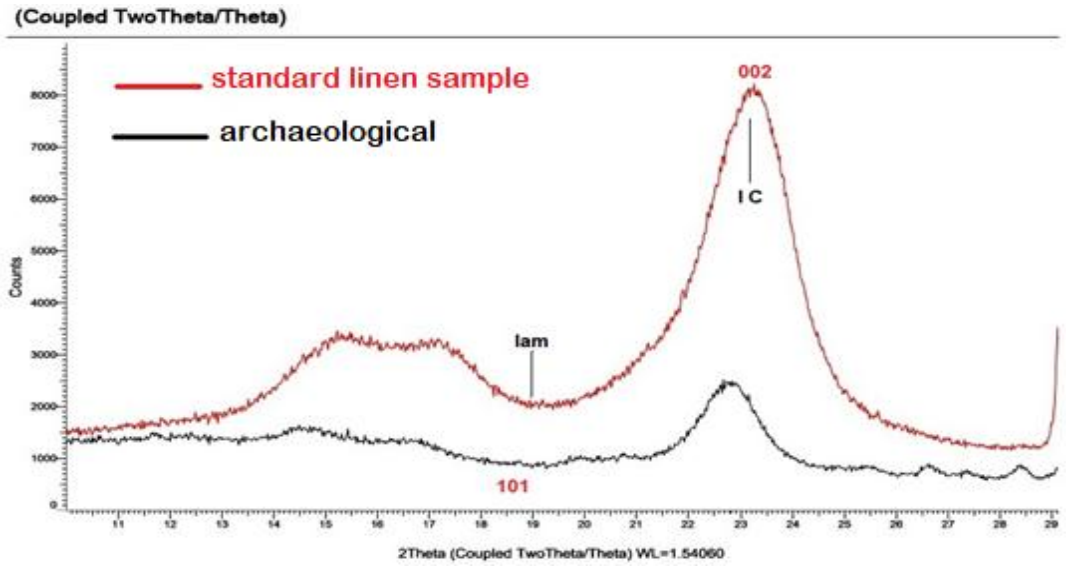


Fig. 6. X-ray diffraction pattern of the archaeological piece for calculating the crystallinity index in comparison to a standard linen sample.

Table 2. Crystallinity index of Archaeological piece compared with standard linen sample

N	Sample	Crystalline area IC	Amorphous area Iam	Crystalline index CI (%)
1	Archaeological piece	2θ 23.2337° Y 3682.84	2θ 18.9941° Y 1667.83	75.1%
2	Standard linen sample	2θ 23.2337° Y 8162.75	2θ 18.9941° Y 450.76	94.4. %

that reflect a decrease in cellulose crystallization rates, an increase in the dissolution rate of the fiber structure, and an increase in its content of hydroxyl bonds (OH) [28].

3.2.6. Identification of isolated microorganisms

Through identification according to morphological characteristics using microscopic examination of microorganisms, the fungal strains that dominated on the archaeological piece were identified, which belonged to two strains, Aspergillus niger and Penicillium oxalicum as shown in Fig. 7..These fungal strains have been spotted on many archaeological textiles in Egyptian and Arab museums [34 ,33]. As for bacteria, no growth occurred for any type of bacteria.

Through the examinations and analysis of the archaeological piece, it became clear that it has a plain weave structure 1/1, and it contains remnants of dust, dirt, and salts, and some missing parts, in addition to the presence of fungal spots, as shown in the damage map in Fig. 8.

3.3. Treatment and conservation of the archaeological piece

3.3.1. Wet cleaning by using surfactant Orvus WA paste at a concentration of 1%

In the treatment process, the surfactant Orvus WA paste, produced by the American company Tales was used at a concentration of 1%, along with the use of sodium carboxymethyl cellulose (SCMC) at a concentration of 0.1% as a sequestering agent that prevents the sedimentation of dirt again on the surface of the textiles. The wet cleaning was done using the immersion method. At the beginning of the treatment process, the piece was moistened using distilled water due to the severe state of dryness that the piece was suffering from, as well as to straighten the bent parts of the piece before the immersion process. The moistening process was carried out over three days, twice a day using a traditional hand sprayer. The treatment process was carried out using four basins. The first basin was a humidification basin using only distilled water, and its function was to prepare the piece to receive the surfactant, the piece was submerged in it for five minutes. The second was the washing basin containing the Orvus WA at a concentration of 1%. With (SCMC) at a concentration of 0.1%, the piece was immersed in it for some time about 15 minutes with mechanical movement with the palm or using brushes. The third and fourth baths are rinsing baths to remove the surfactant residue that may remain stuck on the surface of the textiles after the washing process, the pieces were immersed in each bath for only 5 minutes as shown in Fig. 9. Also Fig. 10. shows the washing solutions resulting from the wet cleaning process, which shows the significant change in the form of the solution as a result of the dust and dirt that was removed from the archaeological piece.

3.3.2. Applying the AgNPs/ Klucel G/ H_2O_2

After the artifacts were completely dry after washing with a surfactant, a composite of AgNPs 3% Klucel G 1%/ H_2O_2 3% was applied by spraying. However, the process of applying the nanometric compound to the archaeological piece was preceded by a microbiological study to study the efficiency of the compound used in resisting the fungal strains Aspergillus niger and Penicillium oxalicum that were isolated from the artifact to clarify the effect of the compound used as an antifungal. The efficiency of the compound was studied on fungal strains using the well-cut diffusion technique in agar.[11, 27] Fig.11. shows the diameter of the inhibition zone resulting from applying the compound to fungal strains isolated from the artifact extracted from the Qusayr excavations.

Once the compound's effectiveness was confirmed, it was applied to the artifact while a Teflon surface was present. This was done to prevent the artifact from sticking to the surface it was placed on during the treatment process with the nanometric compound, as illustrated in Fig. 12. Also Fig. 13. shows the archaeological piece before and after treatment and conservation processes.

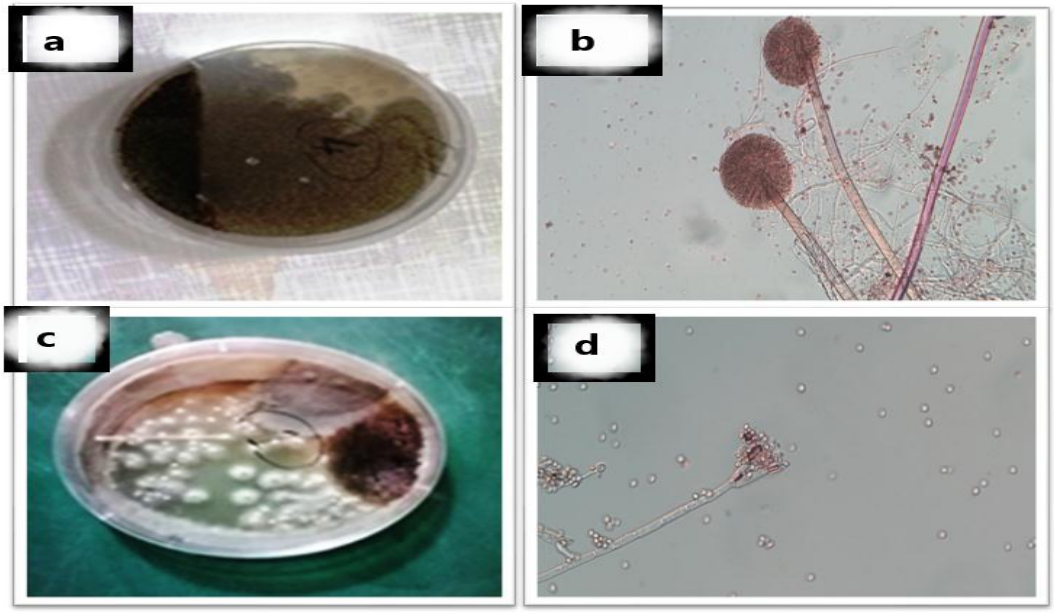


Fig. 7. Isolated fungal strains where (a, b) *Aspergillus niger*, while(c, d) *Penicillium oxalicum.*

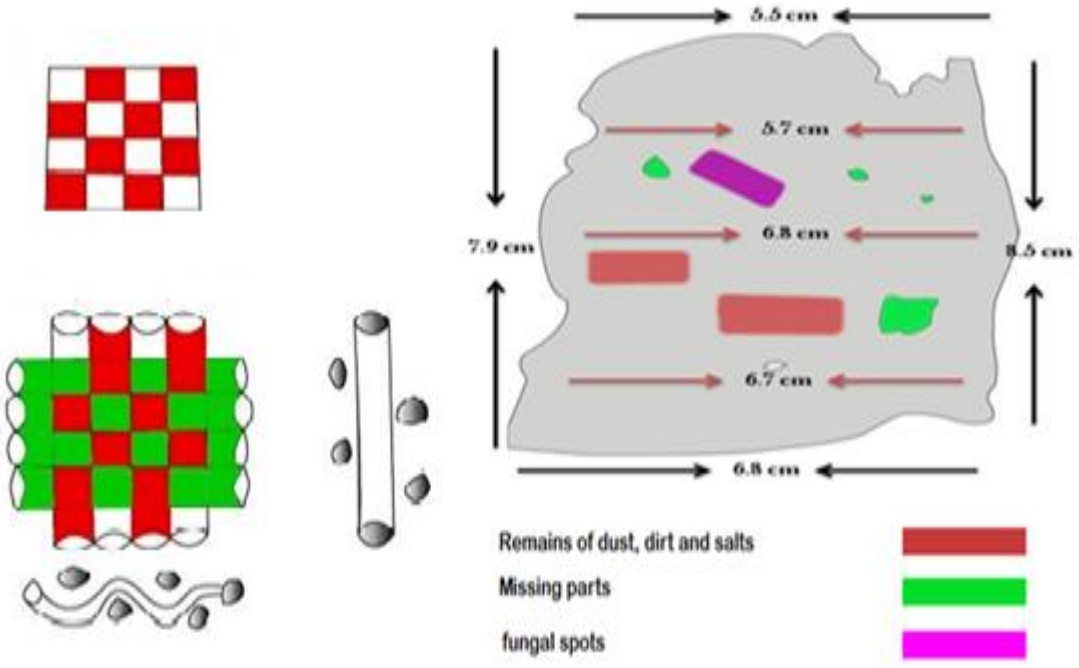


Fig. 8. Plain weave structure 1/1 and map of the most degradation aspects of the archaeological piece.

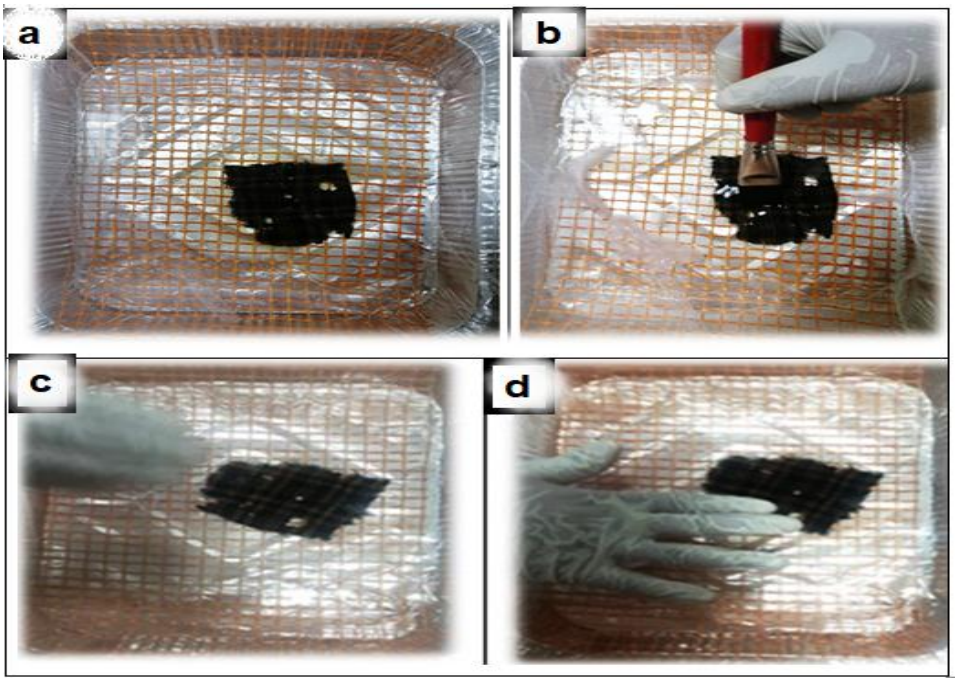


Fig. 9. The washing baths for the archaeological piece, where (a) is the distilled water basin, (b) is the surfactant basin, and (c, d) is the final rinsing basins.

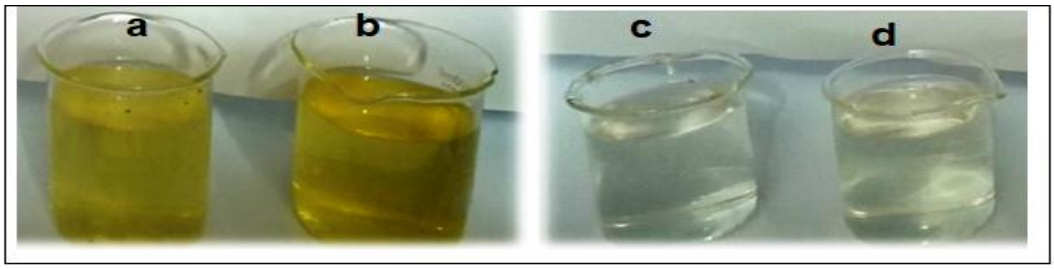


Fig. 10. The washing solutions resulting from the wet cleaning process, Where (a) is the wash solution for the first bath, (b) is the wash solution for the surfactant basin, (c, d) are the wash solution for the rinse tanks 3,4.

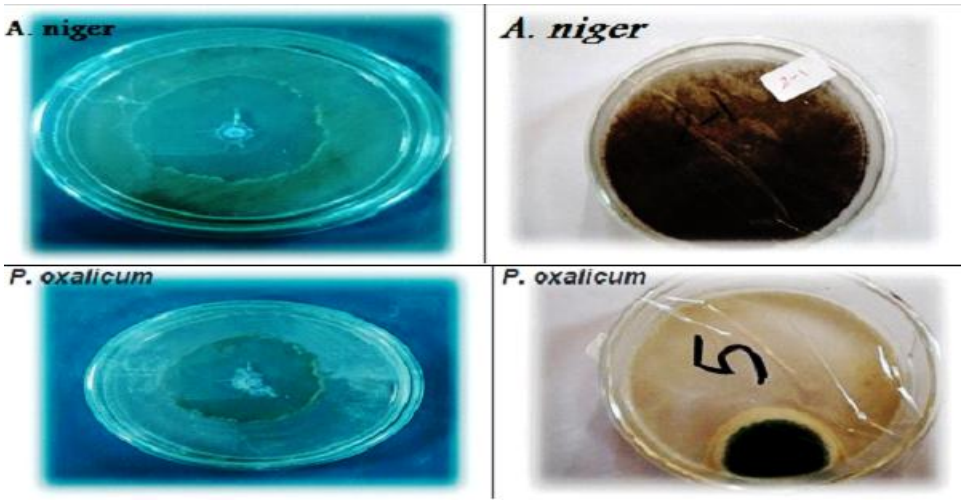


Fig. 11. The diameter of the inhibition zone of the nanocomposites against isolated fungal strains.

Fig. 12. Applying the nanocomposites by spraying on the artifact in the presence of a Teflon surface.

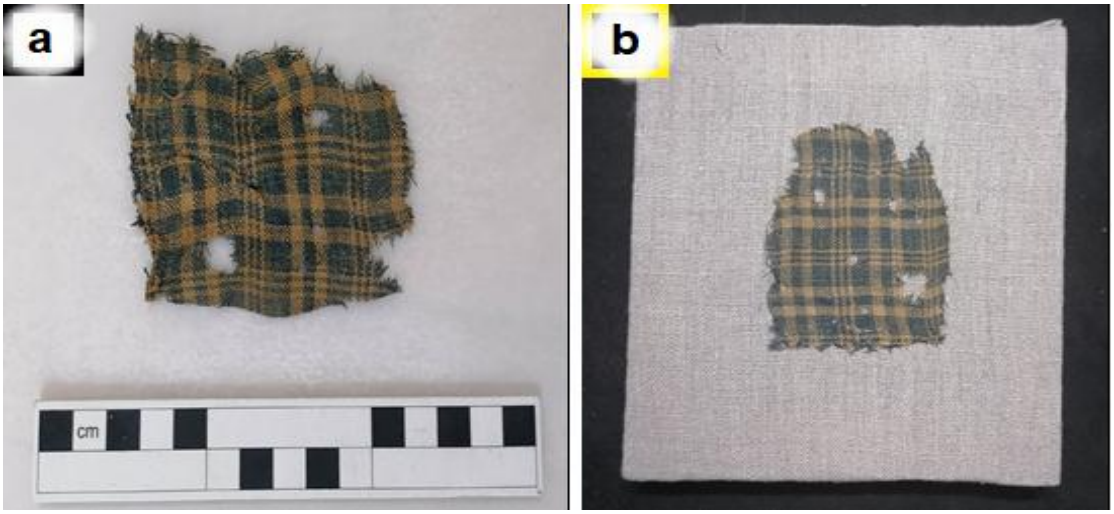


Fig. 13. The archaeological piece where (a) its condition before treatment and conservation processes, and (b) the piece after treatment.

4. Conclusion

Wet cleaning of archaeological textiles is considered one of the most dangerous stages of treatment because it is irreversible process. Through examination and analysis of the artifact, it became clear that it was a piece discovered in a place near to the sea as a result of the presence of residues of sodium chloride salts covering the morphological surface of the artifact. The microbiological study of the artifact also revealed that two fungal strains dominated the artifact, *Aspergillus niger* and *Penicillium oxalicum*. The study of the efficiency of the nanocomposites also demonstrated the effectiveness of AgNPs/Klucel G in inhibiting the fungal strains that were isolated from the artifact.

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