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Experimental and Applied Study to Evaluate the Effectiveness of Silver Nanoparticles Added to Some Plant Volatile Oils and Chemical Insecticides Against Anthrenus Verbasci Larvae Infected Museum Woolen Textiles

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Abstract: The present work is undertaken to harness nanotechnology as one of the most important frontier sciences for developing the effectiveness of volatile oils and chemical insecticides. Experimental studies were done to evaluate the effectiveness of two volatile oils (Cinnamon and Eucalyptus) oils and two chemical insecticides (carbon disulfide and paradichlorobenzene) before and after adding Silver Nanoparticles on the color change and the morphology of infected dyed woolen textile by Anthrenus verbasci. The results showed that cinnamon oil is the best material, either used alone or with silver nanoparticles, as it didn’t affect the woolen samples' color, functional groups, or morphology. A new approach was applied to preserve a decorated historical textile no. 9697, dating back to Muhammad Ali’s period (1805-1953 A.D), using cinnamon oil added to silver nanoparticles. Because it was stored in uncontrolled environments at the Museum of Islamic Art, Cairo, it was infected by insects, especially Anthrenus verbasci. This piece's documentation, examination, and analyses were performed to develop a scientific conservation treatment plan. Photoshop, Illustrator, Digital Microscope, FTIR- ATR, and SEM were performed for this purpose. This paper will review the conservation treatment step by step.

Keywords: *Anthrenus verbasci*; volatile oils; chemical insecticides; silver nanoparticles; wool; madder; color measurement; SEM.

1. Introduction

Wool is fine, soft, curly hair that grows on the body of sheep, some types of goats, rabbits, camellias, and camels, forming a protective covering known as a fleece [1,2]. Sheep were the most important source of wool in prehistory [3,4]. Archaeological textiles are predominantly woolen, which was the case in the analyzed samples. As a protein fiber, wool withstands the acidic Finnish soil more than plant fibers. Over time, the wool degrades, the fiber´s surface scales disappear, and the inner microfibrillar structure becomes visible [5].

Natural dyes are known for dying protein fibers like wool. Natural dyes produce very uncommon, soothing, and soft shades [6-8]. Historical textiles are fragile and more prone to damage [9]. The deterioration of textiles can take several forms, such as the biodegradation of fibers resulting in brittleness or even decay, discoloration, staining, etc. All textiles can be deteriorated by light, insects, and microorganisms, which singularly or together cause considerable loss of their tensile and elastic properties [10,11]. The most deterioration factor is insects such as Anthrenus verbasci (L). Commonly called varied carpet beetle, it is one of the most common species found in museums [12,13]. The varied carpet beetle is one of the most dangerous pests, and its larvae cause serious damage to antiquities inside museums, especially those made of animal materials such as wool, silk, fur, and feathers.

Pest control is still largely based on using chemical pesticides; the excessive usage of these pesticides to control insect pests can raise insect resistance [14]. Chemicals react adversely with museum materials, are toxic to the public, and are harmful to the environment [15]. The most common chemical insecticide is carbon disulfide (CS_2), a colorless and organic liquid at room temperature. It has a sweet, pleasing, and ethereal odor in its pure state, but in its unpure state, it has a bad odor. The effect of carbon disulfide on the insect is that it causes a toxic effect on the insect’s nervous system [16,17]. Another example of a chemical insecticide is paradichlorobenzene, a solid material that sublimates at high temperatures and affects dyes and colors. It is often used in textile storage cabins as a protective material to keep textiles from insect infestation [18-21]. Due to the long-term toxicity of chemical insecticides, we need to select a new, highly environmentally friendly pesticide that avoids this problem.

Natural products are an excellent alternative to synthetic pesticides as a means to reduce negative impacts on human health and the environment [22,23]. Volatile oils have attracted attention in recent years as pest control agents due to their insecticidal effect [24]. There are substances with insecticidal effects, like the one obtained from Cinnamon belonging to the genus Cinnamomum (Lauraceae), which includes more than 250 aromatic evergreen trees. It is a very important spice and useful substance in medicines, industry, and food. It is found in Southeast Asia, China, and Australia [25-28]. Eucalyptus (family Myrtaceae), an Australian native including 700 species, is a genus of tall, evergreen, and magnificent trees cultivated all over the world for its oil, gum, pulp, timber, and medicine [29-31]. The applications of nanotechnology have been used to enhance the efficacy and reduce the environmental contamination of a pesticide’s active ingredient [32]. Nano pesticides are Nano-sized materials that provide pesticide properties, slow degradation, and controlled release of active ingredients for a long time. These properties of Nano pesticides make them environmentally safe and less toxic compared to chemical pesticides [33-36]. A number of formulation types have been suggested, including emulsions, Nanocapsules, and products containing pristine engineered nanoparticles, such as metals and metal oxide [37-39]. Nanoparticles (NPs) generally have a much larger surface than bulk materials for the same nanoparticle material, higher reactivity, variable chemical properties, and wavelength similar to light wavelength [40]. The nano-scale silver plays a role in understanding and the ability to manipulate biological processes [41]. Silver nanoparticles also have the efficacy of acting against insects [42].

The present study aims to evaluate the effectiveness of volatile oils and chemical insecticides before and after adding silver nanoparticles and to apply the best material in the treatment of dyed woolen textiles stored at the Museum of Islamic Art, Cairo.

2. Materials and Methods

2.1. Materials.

2.1.1. Volatile oils.

Two commercially available volatile oils were applied to woolen textiles: Cinnamon oil (Lauraceae) and Eucalyptus oil (Myrtaceae). They were purchased as pure oils (Branded in Egypt) from a Pharaonic Company of Medicinal and Aromatic Oils.

2.1.2. Chemical insecticides.

Two commercial chemical insecticides, carbon disulfide (Dithiocarbamates) and paradichlorobenzene (Chlorinated aromatic hydrocarbon), were applied to dyed woolen textile. They were purchased from El-Gomhria Company, Egypt.

2.1.3. Woolen samples.

Woolen fabrics were supplied from El-Mahla Spinning & weaving, Tanta, Egypt. Natural madder dye was purchased from commercial markets (C.I. 9).

2.2. Methods.

2.2.1. Dying fabrics by natural madder dye.

The dying was performed by the exhaustion method using a liquor ratio (LR) of 1:20 (1g of fabric per 20 ml of the bath) for 30 minutes at a temperature [60°C]. In order to adhere to the fabric. The unfixed dyestuff was removed by rinsing three times with cold water (5 minutes at room temperature, 25°C, LR (1:20) [43,44].

2.2.2. Thermal aging of wool samples.

Wool samples were thermally aged in the Museum of Islamic Art laboratory in an oven at 140°C using water to create steam for 18 hours, which is 50 years [45,46].

2.2.3. Exposure of woolen fabric to varied carpet beetle (*Anthrenus verbasci*).

The dyed woolen fabric was exposed to larvae of varied carpet beetles for one month to attack the woolen fabric and make holes in it.

2.2.4. Preparing volatile oils and chemical insecticide solutions.

Volatile oils (Cinnamon oil, Eucalyptus oil) and chemical insecticides (carbon disulfide, paradichlorobenzene) were prepared at a concentration of (3% v/v) in acetone.

2.2.5. Silver nanoparticles synthesis.

Silver nanoparticles (Ag-NPs) were synthesized using the chemical reduction method and Citric acid as a reducing agent [47]. 0.5 mg silver nitrate was dissolved in 50 ml distilled water and then heated with vigorous stirring. Plurunic was added as an emulsifying agent to silver nitrate solution under vigorous heating and stirring, 0.5 mg citric acid solution was added to the previous solution drop by drop, and the color changed from colorless to pale yellow, then a fine grey precipitate formed. After vigorous stirring and heating for one hour, the precipitate was collected after the supernatant of the reaction solution was discarded, washed three times in double distilled water, and once in ethanol solution [48]. Then, the particles were dried in a hot air oven at 60°C for two hours [49]. The dried particles were used as a UV-visible spectrophotometer and Transmission Electronic Microscope TEM for further analysis.

2.2.6. Preparing Ag NPs with volatile oils or chemical insecticide solutions.

Volatile oils (Cinnamon oil, Eucalyptus oil) were prepared at a concentration of (1.5% v/v) in acetone, and 5% silver nanoparticles of the oil concentration were suspended in it by using rode ultrasonic apparatus for 10min (Volatile oil 1.5% v/v, Ag-NPs 0.075 g). In the same manner, Ag-NPs 0.1 g was suspended with (2% v/v) chemical insecticides (Carbon disulfide, Para dichlorobenzene). These concentrations achieved the highest mortality on Anthrenus verbasci, as shown in Table 1 [50-52].

Table 1. Mortality of Anthrenus Verbasci larvae by volatile oils and chemical insecticides.

Materials	Concentration %	Mortality %
Cinnamon oil	3	92
Cinnamon oil Ag-NPs	1.5	100
Eucalyptus oil	3	85
Eucalyptus oil Ag-NPs	1.5	92
Carbon disulfide	4	85
Carbon disulfide Ag-NPs	2	100
Para dichlorobenzene	4	70
Para dichlorobenzene Ag-NPs	2	98

2.2.7. Ag Nanoparticles assessments.

2.2.7.1 UV-Vis spectroscopy.

The spectrum can be used to analyze the materials' composition and structure. For a particular wavelength in the ultraviolet-visible ranges, the absorption degree is proportional to the components of the materials. Therefore, the characteristics of the materials are quantitatively reflected by the spectrum, which changes with the wavelength. A Shimadzu Ultraviolet-Visible spectrophotometer with a double beam was used to obtain the absorption UV-visible spectra of Ag-NPs. The well-suspended sample was scanned from 200 to 800 nm in a 4 cm quartz corvette. The analysis was performed at the National Research Centre, Dokki, Giza, Egypt.

2.2.7.2. Transmission electronic microscope TEM.

The morphology and particle size of the dried Ag particles were measured by Transmission Electron Microscope TEM, where TEM images were obtained by JEOL 1400 - electron microscopy operated at 60 kV at the Faculty of Agriculture laboratory, Cairo University, Giza. Before taking a TEM image, a few amounts of the sample were suspended in distilled water aided by an ultrasonic apparatus for 5 minutes. A drop of well-dispersed diluted sample was placed onto a Copper grid (200 mesh and covered with a carbon membrane) and dried at ambient temperature. A drop of phosphor tungstic acid (0.4%) as a stain was deposited over the dried sample. The particle size was taken as an average of ten particles.

2.2.8. Evaluation of the effectiveness of the materials used on dyed woolen samples.

2.2.8.1. Color measurement.

The dying properties of the dyed wool fabrics were examined to study two main parameters: dye ability expressed as color strength (K\S) and fastness to light, which was evaluated instrumentally by measuring the color difference parameter (ΔE). This gives a more accurate and precise value for the actual amount of faded dye. The CIE Lab values of the dyeing were measured using a double-beam Optimatch spectrophotometer (Data color international Spectra flash SF450-UK). The colors are given in CIE Lab coordinates: L* corresponding to the brightness (100 = white, 0 = black), a* to the red-green coordinate (positive sign = red, negative sign = green), and b* to the yellow-blue coordinate (positive sign = yellow, negative sign = blue). Also, Δl, Δa, and Δb were calculated according to [53]:

Δl*= L sample after treatment- L^o sample before treatment;

Δa*= a sample after treatment- a^o sample before treatment;

Δb*= b sample after treatment- b^o sample before treatment;

The total color difference ΔE* between two color stimuli $\Delta E = \{(\Delta L *)2 + (\Delta a *)2 + (\Delta b *)2\}1\backslash 2$ ΔE* = {(ΔL*)2 + (Δa*)2 + (Δb*)2} 1/2.

The analysis was performed at the National Research Centre, Fabrics and Threads Department, Dokki-Giza, Egypt.

2.2.8.2. Morphological study.

The morphology of the surface of the woolen samples was investigated using a Scanning Electron Microscope (SEM). Scanning electron micrographs were taken of selected samples to assess fiber fracture patterns and the damaged aspects of these fibers. The examination was performed using JEOL JSM 400 LV EDX Lin L ISIS-Oxford (high vacuum) at the Faculty of Sciences, Assiut University.

2.2.8.3 Fourier transform infrared spectral analysis with attenuation total reflection (FTIR-ATR).

The structural changes occurring in the fibers were determined by using Bruker 1 238 2310 (Model: Alpha, PN: 1003271/07, SN: 104633, Made in Germany, Museum of Islamic Art, Cairo, Egypt) from 4000 to 400 cm^{-1} at ambient temperature.

2.2.9. Study of the dyed woolen textile stored at the Museum of Islamic Art, Cairo.

2.2.9.1. Photoshop and Illustrator documentation

The purpose of using the Photoshop program is to clarify the places of damage in the artifact, and the main objective of using Illustrator is to unload the decorative shapes to help the conservator in the restoration process.

2.2.9.2. Examination by digital microscope.

Examination by Digital Microscope was used to clarify the details of the textile.

2.2.9.3. Measurement of pH.

The acidity was measured using the Adwa AD 1000 Three-point pH device at the Museum of Islamic Art. The pH values were measured in separate places due to the presence of many colors, which indicated the different dyes used in this textile.

2.2.9.4. Microbiological examination.

Several swabs were taken from different places in the artifact to determine whether the textile object had any microbial infection. The examination was conducted in the Research and Conservation Center, Projects Sector microbiological laboratory at the Ministry of Antiquities and Tourism.

Microbial swabs were cultured on different media (Cellulose agar and Czapek´s medium) [54]. The Petri dishes are incubated for 4-21 days, and when microbial growths appears, they are isolated and purified individually and defined according to references [55,56].

Then, the dishes were examined under the microscope, Carl Zeiss Microscope Axioplane 2, using a specific program for measuring (axio vision) at the Research and Conservation Center-Microbiology Laboratory to identify the type of microorganism.

3. Results and Discussion

3.1. Silver nanoparticles assessments.

3.1.1. UV-Vis spectrophotometer.

The band of absorption spectra for the produced Ag-NPs was measured by scanning the suspended sample in a wavelength range from 200 to 800 nm in a 4 cm quartz corvette. Using a UV-Vis spectrophotometer on the sample of Ag-NPs, The position of this peak between 410-450 nm suggested the presence of Ag-NPs, as shown in Figure 1 [57,58]. An abroad peak centered at 432 nm was observed (Figure 2).

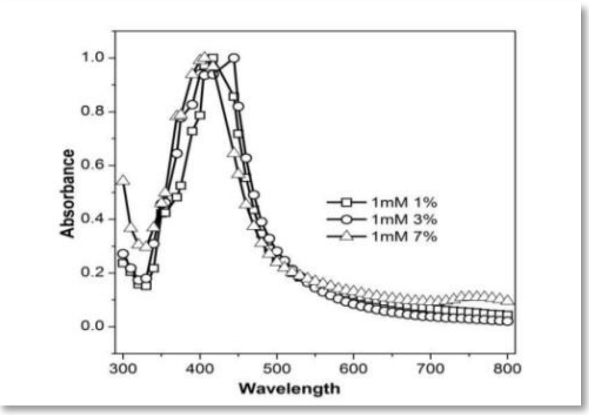


Figure 1. UV–Vis spectra of synthesized Ag-NPs at different concentrations [56].

The increase in the concentration of the Ag-NPs will increase the absorbance intensity of Ag-NPs. It is also observed that the peak with a concentration of 1% that occurs at 417 nm was shifted toward a higher wavelength of 444 nm at a concentration of 3%. Then the peak was shifted towards a lower wavelength of 406 at a concentration of 7%. This shift may be due to the particle size and shape [59].

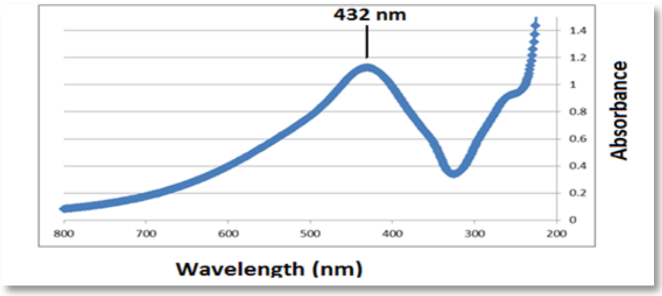


Figure 2. Ultraviolet, visible spectrum for the suspended Ag-NPs sample.

3.1.2 Transmission electron microscope TEM.

The morphology and size of Ag-NPs were measured by using a transmission electron microscope. Figure 3 (a) showed that the morphology of Ag-NPs was observed as spheres, and the particle size was taken as an average of ten particles, which was found to be approximately 20nm. Figure 3 (b) shows the size of the oil, which was bigger than the size of Ag-NPs owing to the large oil diffusion force in acetone.

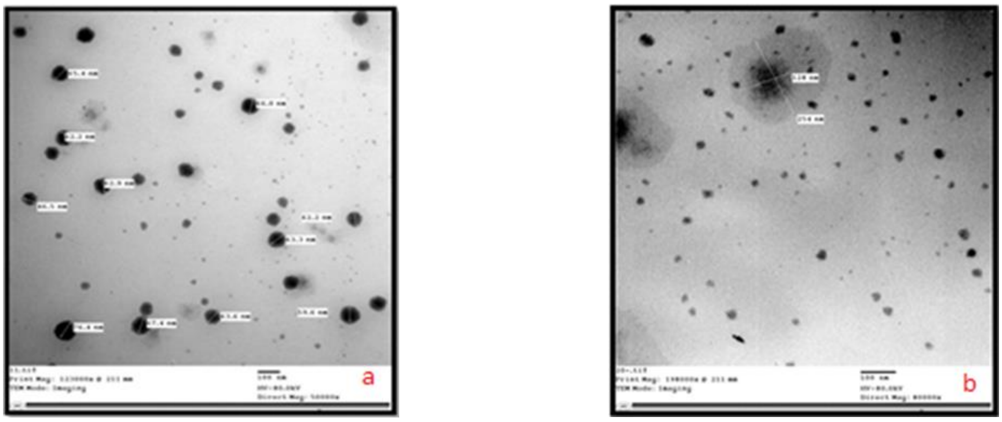


Figure 3. TEM image of **(a)** silver nanoparticles; **(b)** TEM image of volatile oil.

3.2. Effect of volatile oils and chemical insecticides before and after adding silver nanoparticles on dyed woolen samples.

3.2.1. Color changes.

The results in Table 2 showed that volatile oils and chemical insecticides before and after adding silver nanoparticles did not result in any noticeable color change on the surfaces of the experimental samples dyed with madder without any mordant and attacked by Anthrenus verbasci. ΔE values were in the acceptable range of less than 5 [60]. ΔE values also decreased after adding silver nanoparticles. The best material that did not affect the color change of the treated samples was cinnamon oil alone and with silver nanoparticles compared to the other materials; moreover, cinnamon oil with silver nanoparticles achieved the best results.

Table 2. The effect of volatile oils and chemical insecticides before and after adding silver nanoparticles on color change of dyed woolen samples with madder, without any mordant, and attacked by *Anthrenus verbasci*.

Sample	L	$\Delta L*$	a	$\Delta a*$	b	$\Delta b*$	ΔE
Standard sample (sample attacked by *Anthrenus Verbasci*)	46.05	-----	30.42	-----	29.07	-----	-----
The sample was treated with cinnamon oil 3%	45.44	-0.61	30.14	-0.28	27.77	-1.3	1.07
The sample was treated with Eucalyptus oil 3%	46.08	+0.03	31.52	+1.1	29.01	-0.06	1.28
The sample treated with carbon disulfide 3%	44.19	-1.86	30.17	-0.25	27.67	-1.4	2.02
The sample was treated with paradichlorobenzene 3%	44.63	-1.42	30.95	+0.53	28.40	-0.67	1.49

Sample	L	$\Delta L*$	a	$\Delta a*$	b	$\Delta b*$	ΔE
The sample was treated with 1.5% cinnamon oil with Ag NPs	46.51	+0.46	30.29	-0.13	29.04	-0.03	0.44
The sample treated with Eucalyptus oil 1.5% with Ag NPs	46.53	+0.48	28.85	-1.57	27.59	-1.48	0.88
The sample treated by carbon disulfide 1.5% with Ag NPs	46.64	-0.41	29.43	-0.99	28.15	-0.92	0.61
The sample treated with paradichlorobenzene 1.5% with Ag NPs	45.48	-0.57	30.67	+0.25	28.60	-0.47	0.68

3.2.2. Morphological study.

The morphology of the surfaces of the dyed woolen samples was investigated using a Scanning Electron Microscope (SEM). Scanning electron micrographs of selected samples were taken to assess fiber fracture patterns and the damaged aspects of these fibers (Figures 4-8). Wool fiber has a very complex hierarchical structure. It can be considered as a biological composite consisting of regions that are both chemically and physically different. The wool fibers consist of roughly cylinder shapes. Wool fiber is surrounded by cuticle cells, composed of scales overlapping each other in one direction [1]. Wool fibers are made up of a protein substance, keratin, and a fatty substance, lanolin [2]. The results in Figure 5 showed that the morphology of the sample treated with cinnamon oil after adding silver nanoparticles improved, and the surface was smooth with a homogeneous appearance. Figure 6 showed that the surface of the sample improved after adding silver nanoparticles compared to the sample treated with Eucalyptus oil only, where the residues of the oil on the surface decreased, and the surface became more homogeneous. The results in Figure 7 showed that the treatment with carbon disulfide alone had a moderate effect on the treated surface, while the surface morphology improved, and the residues were reduced after adding silver nanoparticles. (Figure 8) showed that the surface of the woolen sample treated with 3% paradichlorobenzene was better than that treated with 1.5% paradichlorobenzene added to silver nanoparticles, where the deposits on the surface decreased.

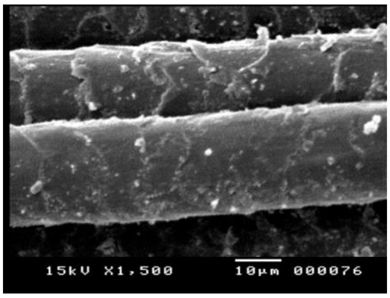


Figure 4. The morphology of the surface of standard dyed woolen samples with madder and attacked by *Anthrenus verbasci.*: 1500×.

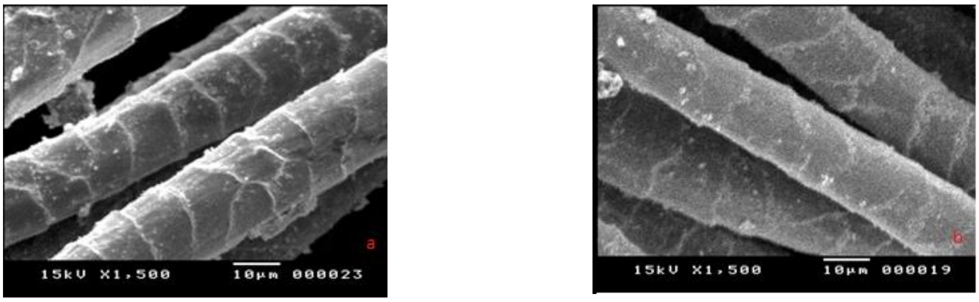


Figure 5. The effect of cinnamon oil on the morphology of the surface of dyed woolen samples with madder and attacked by *Anthrenus verbasci*: **(a)** Sample treated with cinnamon oil 3%; **(b)** Sample treated with 1.5% cinnamon oil Ag-NPs: 1500×.

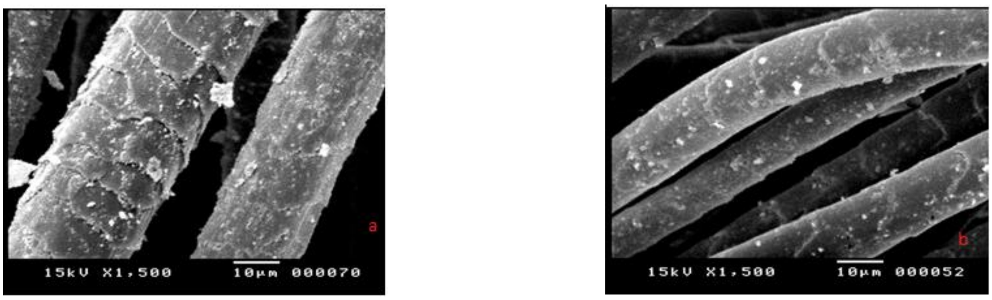


Figure 6. The effect of Eucalyptus oil on the morphology of the surface of dyed woolen samples with madder and attacked by *Anthrenus verbasci*: **(a)** Sample treated with Eucalyptus oil 3%; **(b)** Sample treated with 1.5% Eucalyptus oil + Ag-NPs: 1500.

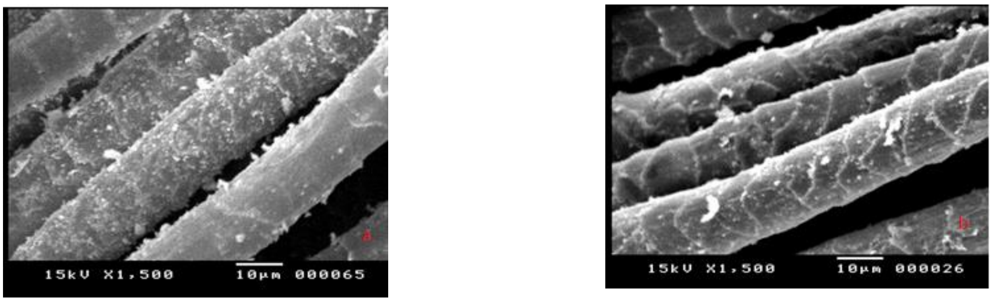


Figure 7. The effect of carbon disulfide on the morphology of the surface of dyed woolen samples with madder and attacked by *Anthrenus verbasci*: **(a)** Sample treated with carbon disulfide 3%; **(b)** Sample treated with 1.5% carbon disulfide Ag-NPs: 1500×.

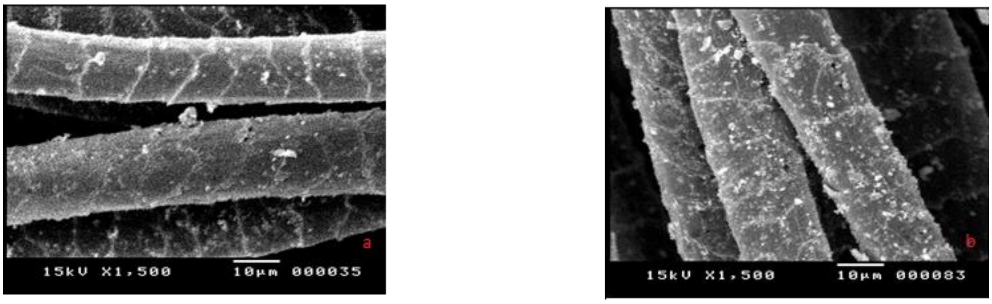


Figure 8. The effect of paradichlorobenzene on the morphology of the surface of dyed woolen samples with madder and attacked by *Anthrenus verbasci*: **(a)** Sample treated with paradichlorobenzene 3%; **(b)** Sample treated with 1.5% paradichlorobenzene Ag-NPs: 1500×.

3.2.3. Fourier transform infrared spectral analysis with attenuation total reflection (FTIR-ATR).

The wool sample has three main functional groups: –COOH, $–NH_2$, and –OH. The wide signal at 3277.77 cm^{-1} represents the vibrations of the N–H bond of primary amines, which are not charged, and –OH groups. Signals at 1600-1660 cm^{-1} (amide I) and 1520-1540 cm^{-1} (amide II) represent $–CONH_2$ (asparagine and glutamine) and –CONH– (peptide chain), respectively. At wave number range 1450 to 1400 cm^{-1}, characteristic signals for –COOH (aspartate and glutamate), and from 1100 to 1000 cm^{-1}, the signal for $–CH_3$ [61,62].

The results in (Figure 9) showed the FTIR-ATR spectrum of the untreated wool sample, compared to that of treated samples with cinnamon oil and cinnamon oil added to Ag NPs. It was found that amide I of the untreated wool sample appeared at 1626 cm^{-1} while the wave number of the sample treated with cinnamon oil was 1624 cm^{-1}. The results also showed no change in the frequencies of amide I bands of treated wool samples with cinnamon oil Ag-NPs which the wave number is 1624 cm^{-1}, and there is no change in the frequencies of amide II and amide III bands of all samples compared to untreated sample. All treated woolen samples show a slight decrease in band intensities compared to the untreated sample.

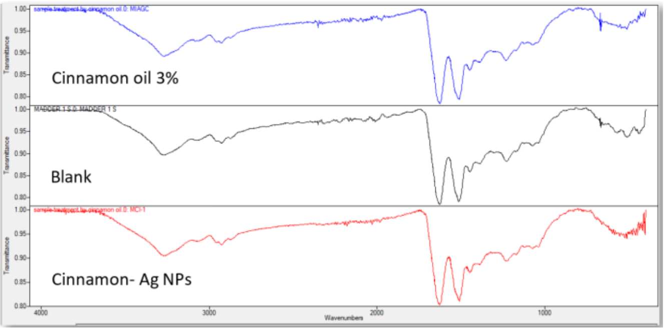


Figure 9. The effect of cinnamon oil before and after adding silver nanoparticles on the functional groups.

3.3. Applied the study on archaeological textile objects.

3.3.1. Description of object.

It was made from cashmere wool manufactured in East and West Asia, especially Kashmir in India. It dates back to the 13th- 19th centuries. Most of these large-sized artifacts were used as curtains inside palaces and buildings in the Islamic period. Some of these textiles were transferred to Muhammad Ali's palace as a kind of trade exchange over long periods of Islamic history. It is a square piece with many plant drawings. It is covered in different colors and has recurring plant patterns. This decoration is known in Kashmir as the Buta or Luza plant leaf. A swollen leaf of the calyx ends at the top with a pointed tip that bends inward, and two strips on both sides surround the piece. It is two narrow strips from the outside, and the inside, in the middle of them, is a more spacious strip in which there are intertwined plant motifs; while the artist deliberately made a decorative ornament in the form of a semi-square, the corners of which end inward in the form of a leaf [63].

The textile object is in very bad condition as a result of insect infection, *Anthrenus verbasci* Larvae, during storage. It contains many colors, such as red, which is predominant on the piece, green, black, blue, yellow, beige, and reddish. It was registered in the museum's archive on April 18, 1931 AD, under number 9697. It was a gift from Mrs. Mary Wiggin.

3.3.2. Documentation.

3.3.2.1. Photographic documentation.

Photographic documentation aims to document the condition of the textile piece before, during, and after the restoration, to clarify the stages of the restoration, and to show the effort done to achieve the whole work (Figures 10, 11), which shows the poor condition for this textile and also shows the deterioration due to larvae of *Anthrenus verbasci*.

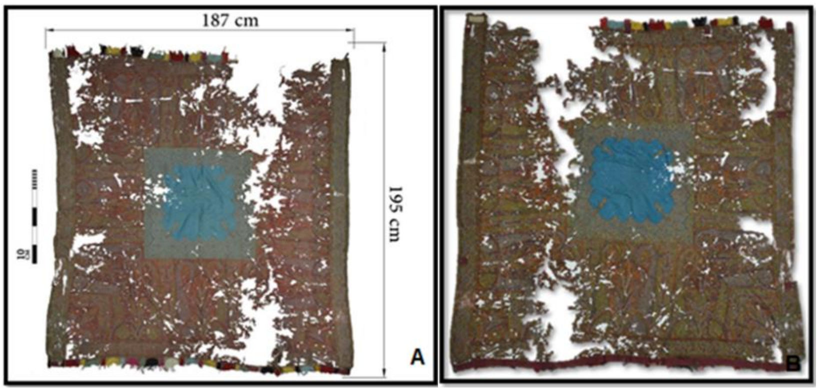


Figure 10. The textile piece before restoration **(A)** from front; **(B)** from back.

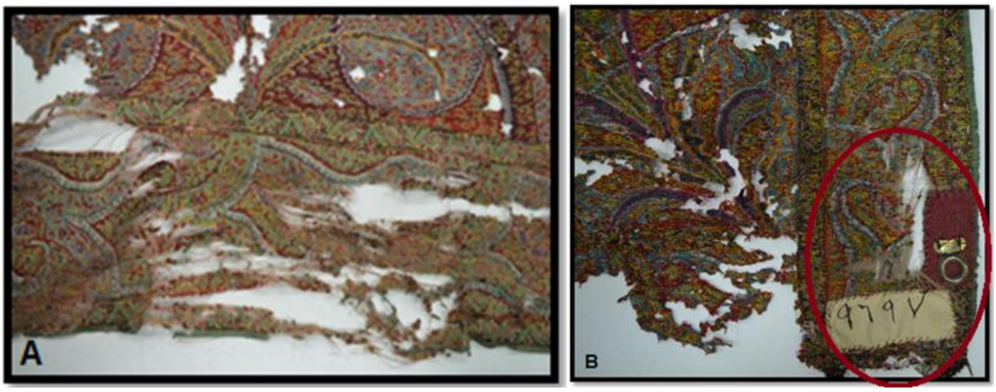
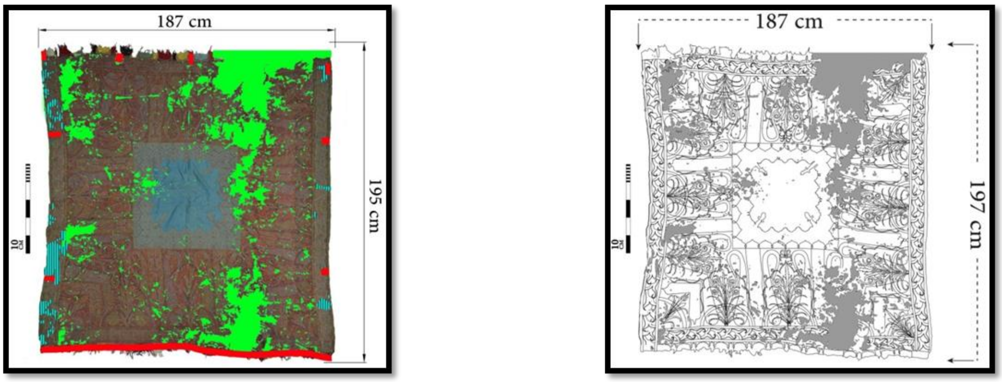


Figure 11. **(A)** ravel places before restoration; **(B)** previous restoration in the back.

3.3.2.2. Photoshop and Illustrator documentation.

As in (Figure 12 A), the green color represents the places of loss due to *Anthrenus verbasci*, the red color the previous restorations, and the blue color the places of ravel in the textile object (Figure 12 B) shows the places of the decorations loss and the shapes of the plant units.



(a) **(b)**

Figure 12. Shows **(a)** Photoshop; **(b)** illustrator documentation.

3.3.2.3. Examination and analysis.

By using Digital Microscope, as shown in (Figure 13), the fabric structure, which is 1 warp/ 1 weft (A), also the ravel parts due to the inappropriate handle in the storage area (B), and there previous restoration by using black thread (C).

(a) **(b)** **(c)**

Figure S13. Shows the textile piece under Digital Microscope before restoration **(a)** fabric structure; **(b)** Ravel places; **(c)** previous restoration.

By measuring the acidity of the artifact, it is clear that the average pH value is 5 at 25°C, and this pH is an indication of the normal condition or the natural pH of the dyed wool, as the wool is of acidic origin and the pH of wool is approximately pH 4.9 [64]. Moreover, most of the organic dyes used in dyeing the artifact are acidic. It is known that the different color shades obtained were obtained through several methods, such as changing the temperature of the dyeing bath, the pH and dyeing time, and different mordents [65].

From microbiological examination after 21 days (Figure 14), the dishes were examined, and we found that this microbial growth was caused by *Aspergillus niger* and *Aspergillus rhizopus* sp, as in (Table 3).

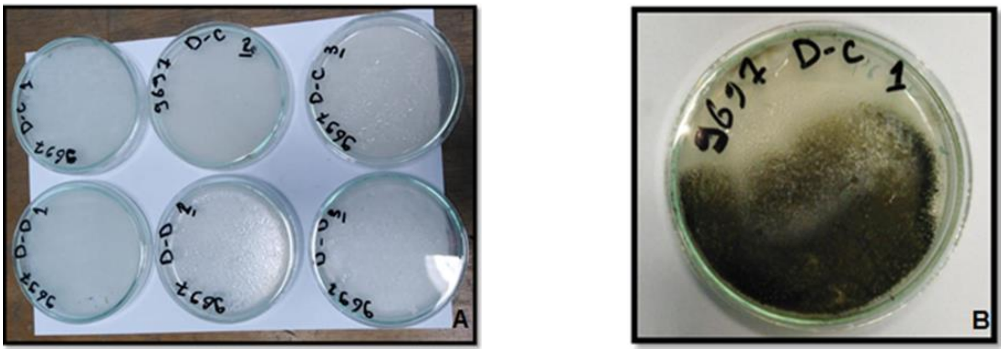


Figure 14. Shows the dishes **(A)** before; **(B)** after the growth of the microorganism.

Table 3. Shows the results of the microbiological examination of the swabs.

Swab(9697)	Czapek´s medium	Cellulose Agar medium
No.1	*Aspergillus niger* *Rhizopus sp.*	***Aspergillus niger***
No.2	*Aspergillus niger* *Rhizopus sp.*	***Aspergillus niger***
No.3	*Aspergillus niger*	***Aspergillus niger***

SEM Photos of the examined textile illustrated in Figure 15 show the wool fibers that were identified from different parts of the object. The fibers are in good condition, but dust can be seen. Also, from the shape of the distinctive scales of the wool fabric, the appearance of the scales on the wool fibers matches the shape of the distinctive scales of cashmere [66-69].

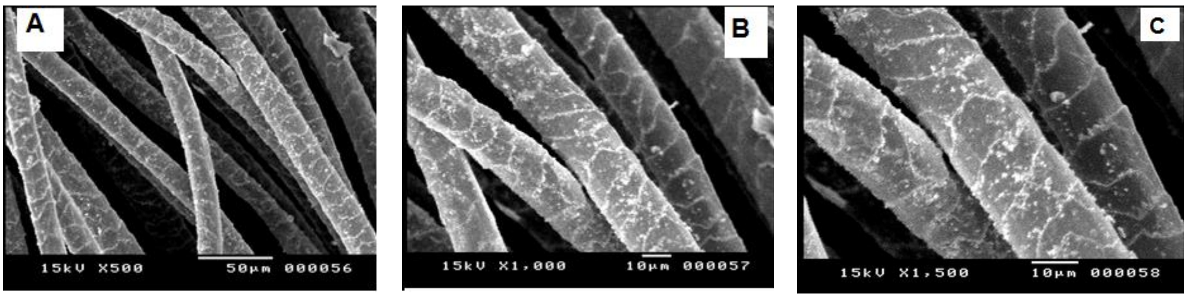


Figure 15. Shows SEM images of examined textile **(A)** Magnification power of 500×; **(B)** magnification of 1000×; **(C)** magnification of 1500×.

From FTIR-ATR, the small samples of different colors were investigated. Then, all the dyes that give this color were prepared and investigated. After that, the original samples were compared with new dyes to find the following: (Red color is a madder dye) as shown in Figure 16.

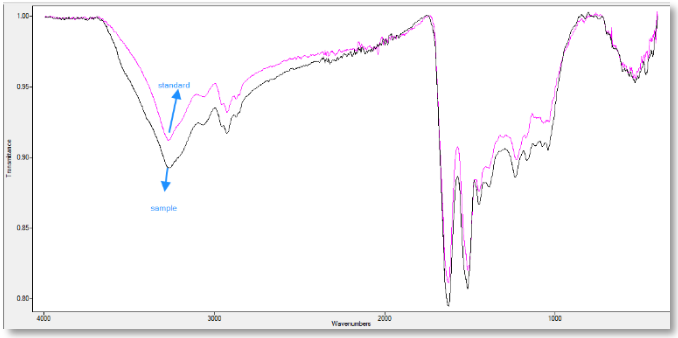


Figure 16. Spectra of wool dyed with madder dye (Standard sample ▬ , sample ▬).

3.4. Restoration processes.

3.4.1. Removal of the old restoration.

In the beginning, the old red tape and the number in the front of the textile object were removed, which was fixed by needle and thread, as shown in Figure 17.

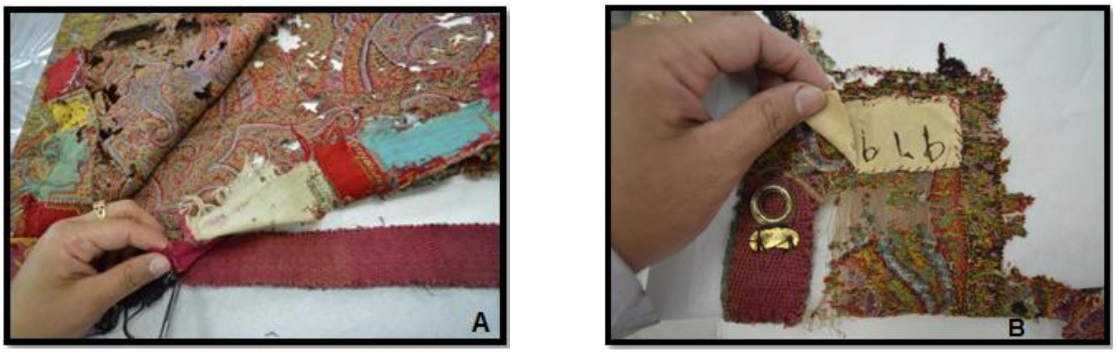


Figure 17. Removal of **(A)** the old red tape; **(B)** the number in the front of the textile object.

3.4.2. Preparing the linen support.

A new linen support was prepared and washed to remove chemical residues and prevent shrinkage at a later time due to humidity changes. Then, the new linen support was ironed to remove creases. Then, the researchers created a new metallic frame (aluminum) that was prepared to fix the textile object on it.

3.4.3. Treatment of the linen support with cinnamon oil added to silver nanoparticles.

The new linen support was treated with cinnamon oil added to silver nanoparticles by spray method to make future protection for the artifact from insect damage, as shown in Figure 18.

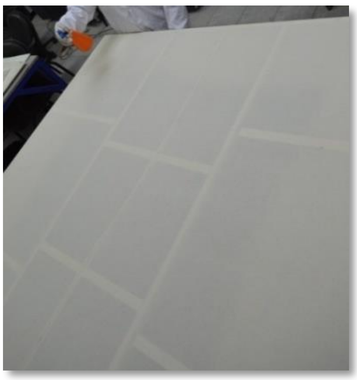
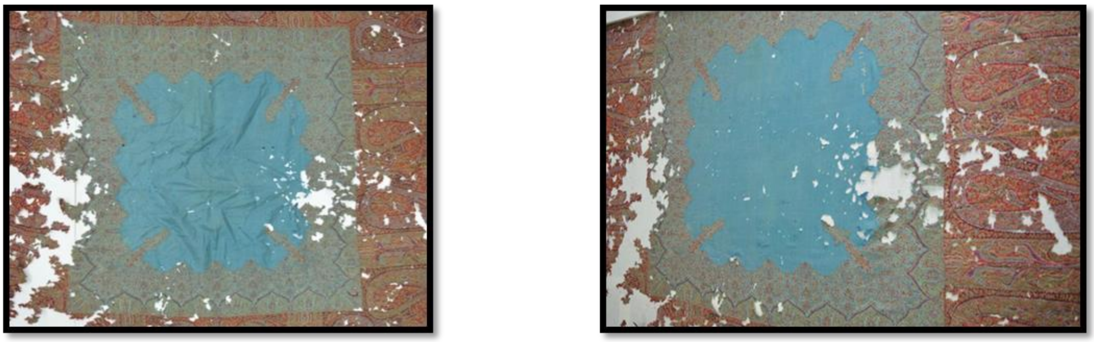


Figure 18. Treatment of the linen support with cinnamon oil-Ag-NPs.

3.4.4. Remove creases from textile objects.

The removal of creases was done using an ultrasonic device. The device was shed on the creases part until it was saturated with moisture. As this method did not give acceptable results, a metal holder was placed under the creased part of a textile object, tissue paper was placed on a textile object from the front, and water was sprayed on it. This method would move moisture from the wet paper to the textile object, and a magnet was placed on the edges of the object to make it as flat as possible.

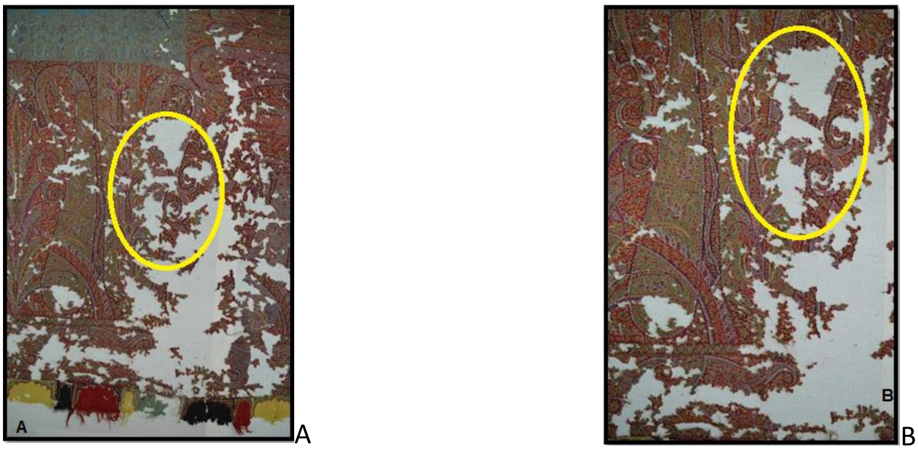


(a) **(b)**

Figure 19. (a) before removing creases from textile objects; **(b)** after removing creases.

3.4.5. The final support.

After completion of the object treatment, tacking stitches were used with a very fine needle and fine silk thread to fix it. At the beginning of the final stage, the edges of the object all around were attached by sewing with a small stitch technique, and afterward, the edges of the missing and vulnerable parts were attached by small stitches, as in Figure 20.



(a) **(b)**

Figure 20. (a) Before fixing; **(b)** after fixing.

3.4.6. Mechanical cleaning.

The cleaning was done after the completion of the restoration due to the poor condition of the textile object and its exposure to severe damage by insects. The textile was cleaned by using a vacuum cleaner, taking into account the placement of a plastic net over the textile object during cleaning (Figure 21).

Figure 21. Cleaning textile objects by vacuum.

3.5. Storage of textile objects.

In the case of the antique 9697, it cannot be stored on the metal frame on which the artifact was fixed. Due to the large size of the piece, after the completion of the restoration, which consumes a large storage space and causes the piece to be quickly damaged, a box suitable for the size of the artifact made of acid-free cardboard was used.

The artifact was folded on four layers with spacers of acid-free inert Ethafoam (Figure 22). Ethafoam has no harmful additives, prevents insects from moving to the objects stored inside, and is ozone-friendly. Ethafoam is resistant to moisture and chemicals and inert to light [70-73].

Figure 22. Fold textile objects and put foam between layers.

The artifact was wrapped with Tyvek, which is made of high-density polyethylene, non-woven fabric. It is a very strong material that is difficult to tear. It prevents the passage of water in one direction but allows air to pass in both directions. It is water-resistant and has been recommended for use in the preservation of textiles. It is stable for long-term use [70].

Acid-free matt board is prepared to support textile objects during storage and transportation. The box was prepared with the dimensions of the artifact after bending, which is 95×70×10 cm^3.

After preparing the box, it was wrapped from the outside using wrapping paper in order to preserve the box and increase its support due to the size of the textile object. After completing the preparation of the box, protective doses of cinnamon oil added to silver nanoparticles dissolved in acetone, cotton saturated with cinnamon oil- Ag NPs was placed inside a perforated bag next to the artifact, and another was installed in the box cover using paraloid B72 to protect the textile from any insect infestation, and these doses should be changed constantly. Conservation practices and packing are carried out to keep textile antiquities safe from deterioration and preserve them for the long-term effects of climate change [74-76].

4. Conclusions

This study concluded that Cinnamon oil without and with adding silver nanoparticles was the best-treated material that did not affect the dyed woolen samples and achieved the lowest value in color change compared to the rest of the studied materials. Moreover, the morphology of the sample surface improved, becoming smooth and homogeneous, especially after adding silver nanoparticles. The result also shows no change in the amide I, amide II, and amide III bands of cinnamon oil samples before and after adding silver nanoparticles compared to untreated samples. The surface of the dyed woolen samples treated with Eucalyptus oil was also improved after adding silver nanoparticles in color change and morphology compared to both studied chemical insecticides (carbon disulfide and paradichlorobenzene).

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Conflicts of Interest

The authors declare no conflict of interest.

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