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Evaluation of the effectiveness of some volatile oils and chemical insecticides were added to Silver Nanoparticles used for the treatment of infected dyed woolen textile with museum insect pests *Anthrenus verbasci* 



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#### Abstract

Experimental studies were done to evaluate the effectiveness of two volatile oils (Cinnamon, Eucalyptus oil) and two chemical insecticides (Carbon disulfide, Paradichlorobenzene) were added to Silver Nanoparticles to control a museum insect pest *Anthrenus Verbasci*. The results showed that 1.5% (v/v) concentration of cinnamon oil added to 5% silver nanoparticles was the most effective on controlling *Anthrenus verbasci*. The effects of these materials were also studied on the chemical and physical properties of dyed woolen textile by using FTIR-ATR, XRD, in addition to mechanical properties measurements.

Keywords: Anthrenus verbasci, Volatile oils, chemical insecticides, wool, Museum.

#### 1. Introduction

Anthrenus verbasci (L.) Commonly called varied carpet beetle, which is one of the most common species of carpet beetles which were found in museums [1, 2, 3]. The varied carpet beetle is one of the dangerous pests whose larvae cause serious damage to antiquities inside museums especially which made of organic materials, such as, wool, silk, fur and feathers [3]. Pest control is still largely based on using chemical pesticides, the excessive usage of these pesticides is to control insect pests can raise insects resistant [4]. Those chemicals may react adversely with museum materials and can be toxic to the public and harmful to the environment [5]. The most common chemical insecticide is Carbon disulfide (CS2) which is a colorless and liquid organic solvent at room temperature. In its pure state, it has a sweet, pleasing and ethereal odor. Exposure to CS2 concentrations of several hundred ppm for several days may cause headache, dizziness, vomiting

and local irritation. While for its effect on the insect, it causes a toxic effect on the insect's nervous system [6, 7]. Another example of chemical insecticides is Paradiclorobenzene which is a solid material that is difficult to sublimate at high temperature can affect dyes and colors. It is often used in textile storage cabin as a protective material to keep textiles from insect infestation. Exposure to Paradiclorobenzine may cause headache, vomiting, eye irritation and rhinitis [8, 9, 10, 11] & Bosworth, et al., 2003, Sirois, et al, 2007 & Wójcik, et al., 2014).

Due to the long term toxicity of chemical insecticides, we need to select a new highly environmental friendly pesticide that reduces this problem. Natural products are an excellent alternative to synthetic pesticides as a mean to reduce negative impacts to human health and the environment [12, 13]. Volatile oils have attracted attention in recent years as a pest control agents due to their insecticidal effect [14]. There are substances with insecticide effect, as the one obtained from Cinnamon belonging

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to the genus Cinnamomum (Lauraceae) and includes more than 250 aromatic evergreen trees. It is a very important spice and very useful substances in medicines, industry and food, it is found in Southeast Asia, China, and Australia [15, 16, 17, 18]. Eucalyptus (family Myrtaceae), an Australian native including 700 species, is a genus of tall, evergreen and magnificent trees cultivated world over for its oil, gum, pulp, timber and medicine [19, 20, 21].

Recently, the applications of nanotechnology have been used to enhance the efficacy and reduce the environmental contamination of a pesticide active ingredient [22Nano pesticides are Nano sized materials which provide pesticide properties, slow degradation and controlled release of active ingredient for long time. These properties of Nano pesticide make them environmentally safe and less toxic compared to chemical pesticide [23]. A number of formulation types have been suggested including emulsions (e.g., Nano emulsions), Nano capsules (e.g., with polymers), and products containing pristine engineered nanoparticles, such as metals and metal oxide [24, 25, 26]. ]. Nanoparticles (NPs) have a much larger surface compared to bulk materials generally, for the same nanoparticles material higher reactivity and variable chemical properties and wavelength is similar to light wavelength [27]. The Nano-scale Silver play roles in understanding and ability to manipulate biological process [28], also Silver Nanoparticles have the efficacy to act against the insects [29].

The aim of the present study is to evaluate the effectiveness of volatile oils and chemical insecticides were added to Silver Nanoparticles on museum insect pest Anthrenus verbasci and on dyed woolen textile.

# 2. Materials and Methods

# 2.1. Materials

# 2.1.1. Volatile oils

Two commercially available volatile oils were tested in this study, Cinnamon oil (Lauraceae) and Eucalyptus oil (Myrtaceae). They were purchased as pure oil (Branded in Egypt) from Pharaonic Company of medicinal and aromatic oils. The oils were extracted from the dried plants by steam distillation.

#### 2.1.2. Chemical Insecticides

Two commercially chemical insecticides, Carbon disulfide (Dithiocarbamets) and Paradichlorobenzen

(Chlorinated aromatic hydrocarbon) were tested in this study. 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 Turmeric dye was purchased from commercial markets (C.I. 75300).

- Mordent Potash Alum was purchased from El-Gomhria Company, Egypt.

#### 2.1.4. Insect culture

The culture of varied carpet beetle (*Anthrenus Verbasci*) was obtained from infested carpets at Manial palace in Egypt. The insects were reared on feathers at insect control laboratory, center of research and conservations of antiquities, Egyptian Ministry of Tourism and Antiquities under laboratory conditions (50-55% RH and temperature 27-30C°) as in (figure 1) [30, 31].

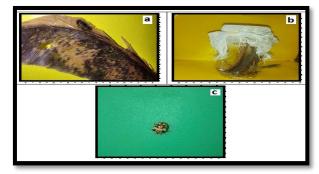


Figure1. Varied carpet beetle at laboratory: (a) larva (b) insect culture (c) adult

## 2.2. Method

#### 2.2.1. Dying fabrics by Natural Turmeric dye

The dyeing was performed by the exhaustion method using a liquor ratio (LR) of 1:20 (1g of fabric per 20 ml of bath) for 30 minutes at temperature [60° C]. In order to help the dye to adhere to the fabric. A mordent Potash Alum was added as concentrated solution (30 g/l). The unfixed dyestuff was removed by rinsing three times with cold water (5 minutes at room temperature [25 °C], LR 1:20) [32, 33].

#### 2.2.2. Thermal Aging of wool Samples

Wool samples were thermally aged in the laboratory of Islamic art museum in an oven at 140°C

using water to create steam for 18 hour which equals to a 50 year [34, 35].

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

Dyed woolen fabric was exposed to larvae of varied carpet beetle for one month to attack the woolen fabric, and make holes on it (see figure 2).



Figure2. The holes of larvae on woolen fabric (blue circles)2.2.4. Ag Nanoparticles synthesis

Silver Nanoparticles (Ag-NPs) were synthesized by using chemical reduction method and using Citric acid as reducing agent [36]. 0.5 gm Silver Nitrate was dissolved in 50 ml distilled water then heated with vigorous stirring. Plurunic was added as emulsifying agent to Silver Nitrate solution under vigorous heating and stirring, 0.5 gm Citric acid solution was added to the previous solution drop by drop and the color will change from colorless to pale yellow then fine grey precipitate will be 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 [29]. Then, the particles were dried in a hot air oven at 60 °C for two hour. The dried particles were used for further analysis as UV-Visible spectrophotometer and Transmission Electronic Microscope TEM.

# 2.2.5. Method of mixing of Ag NPs in the volatile oils or chemical insecticides solution

The dried fine grey Ag-NPs were suspended in Acetone solution of volatile oils or the chemical

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insecticides of concentration of 5% by using rode ultrasonic apparatus for 10min. where a predetermined Ag-NPs as (0.5, 1, 1.5%) (v/v) of the active material (volatile oils or chemical insecticides).

# 2.2.6. Ag Nanoparticles assessments 2.2.6.1. UV-Vis spectroscopy

UV-VIS spectroscopy is a very reliable technique for characterization of Synthesized Nanoparticles. Furthermore, UV-VIS spectroscopy is fast, easy, simple, sensitive, selective for different types of NPs, needs only a short period time for measurement. A Shimadzu Ultraviolet–Visible spectrophotometer with 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 4 cm quartz corvette. The analysis was performed at National Research Centre, Dokki, Giza, Egypt.

## 2.2.6.2. Transmission Electronic Microscope TEM

The morphology and particle size of the dried Ag particles were measured by Transmission Electronic Microscope TEM where TEM images were obtained by JEOL 1400 -electron microscopy operated at 60 kV at Faculty of Agriculture laboratory, Cairo University, Giza. Before taking a TEM image, a few amount of sample was suspended in distilled water aiding by ultrasonic apparatus for 5min. 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 [37]. The particle size was taken as an average of ten-particles.

# 2.2.7. Examination and analysis for Dyed woolen samples

# 2.2.7.1. Fourier Transform Infra-Red Spectroscopy with Attenuation Total Reflection (FTIR-ART)

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.

#### 2.2.7.2 X-ray Diffraction Analysis (XRD)

Wide angle X-ray scattering measurements of untreated and treated samples were carried out with EMPYREAN X-ray Diffractometer system which given 45 KV CU, radiation of 30mA, the analysis was performed at National Research Centre, Dokki, Giza, Egypt

#### 2.2.7.3. Mechanical Measurements

Mechanical parameters, such as tensile strength and elongation, were measured by Instron Model 3345, According to the ASTM: D 5035-21 Standard test method for breaking force and Elongation of textile fabrics (Grab Test) in the warp directions, the conditions of mechanical properties measurement was temperature  $20^{\circ}C \pm 5$  and humidity  $65\% \pm 5$ . Woolen fabrics were cut into 15cm strip length and 3cm widths. Three samples per treatment set were

tested, and the breaking load averaged for each sample [38], National Research Centre, Department of fabrics and threads, Bohooth st.- Dokki-Giza, Egypt.

#### 2.2.8. Fumigation test

Concentrations of volatile oils and chemical insecticide which were added to Silver Nanoparticles were prepared. Filter papers (whatman No.1) with size 11cm were treated with 1 ml of each concentration of oils and placed on the inner surface of the cover of 10cm Pettri-dish. A group of twenty five larvae of varied carpet beetle were put in Pettridish for each experiment and finally, the dishes were made air tight. Each treatment was replicated four times. After 24 hours of fumigation, Larvae mortality was recorded (figure 3).

#### 2.2.9. Data analysis

Mortality data were corrected using Abbott's formula (Abbott, 1925). The observed data were subjected to probit analysis according to probitvb6.

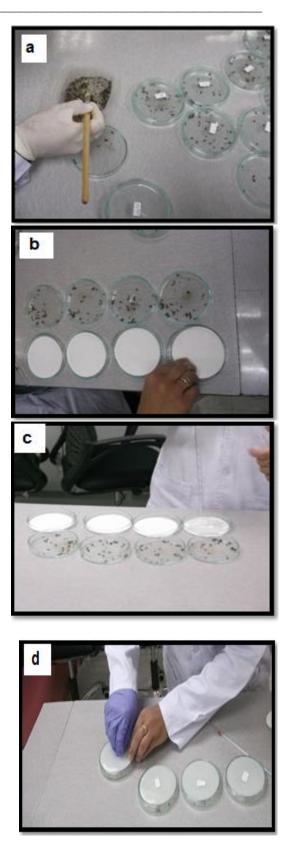


Figure 3. (a, b, c, d) Application of volatile oils and pesticides were added to Ag-NPs with the larva in petri dishes

#### 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 wavelength range from 200 to 800 nm in 4 cm quartz corvette. Abroad peak centered at 432 nm was observed (figure 4). The position of this peak between 410-450 nm suggested the presence of Ag-NPs [39, 40].

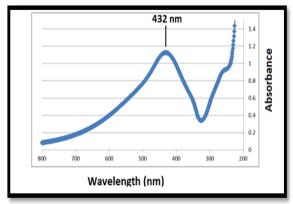


Figure 4. Ultraviolet visible spectrum for the suspended Ag-NPs

# 3.1.2. Transmission Electronic Microscope TEM

The morphology and size of Ag-NPs were measured by using transmission electron microscope. The morphology of Ag-NPs was observed as spheres and the particle size was taken as an average of tenparticles and found to be approximately as 20nm (figure 5).

# **3.2.** Efficacy of volatile oils and chemical insecticides added to silver nanoparticles on mortality of *Anthrenus Verbasci*

# **3.2.1.** Efficacy of Cinnamon and Eucalyptus oils with Ag-NPs on mortality of *Anthrenus Verbasci*

Predetermined weight of Ag-NPs with concentrations 0.5, 1, 1.5% (v/v) of oil was suspended in oil-Acetone solution with concentration 5% aiding by ultrasonic apparatus for 10min. The efficacy of Cinnamon and Eucalyptus oils with Ag-NPs on mortality of Anthrenus Verbasci were tested often 24 hours. The result showed when we added silver nanoparticles to volatile oils it increase the mortality of Larval Anthrenus Verbasci in different concentrations compared to volatile oils only and LC50 of Cinnamon oil - Ag-NPs was 0.442 while that LC<sub>50</sub> of cinnamon was 0.854 indicating that Ag NPs

improved performance of volatile oils in mortality of larva. Also Cinnamon oil - Ag-NPs was 0.442 while that of Eucalyptus oil Ag-NPs was 0.639 indicating that Cinnamon oil – Ag-NPs was the effective Nano oil in different concentrations for controlling *Anthrenus Verbasci*, where it possess the lowest value of  $LC_{50}$  and  $LC_{90}$  as in (table 1, figure 6).

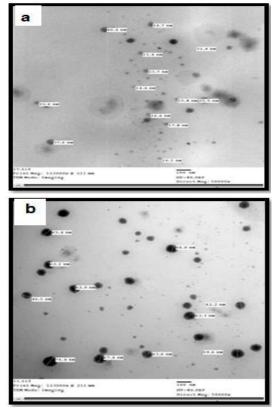


Figure 5. (a, b)TEM images of Silver Nanoparticles

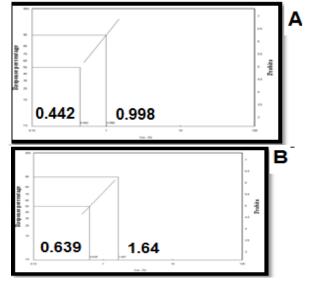


Figure 6. (A) The effect of cinnamon oil Ag-NPs and (B) eucalyptus oil Ag-NPs concentrations on larval bioassay

# **3.2.2.** Efficacy of Carbon disulfide and Paradichlorobenzen added to silver nanoparticles on mortality of *Anthrenus Verbasci*

Predetermined weight of Ag-NPs with concentrations 0.5, 1, 1.5% (v/v) of chemical insecticides was suspended in their acetone solution with concentration 5% aiding by ultrasonic apparatus for 10min. The efficacy of concentration (0.5, 1, 1.5, 2%) of Carbon disulfide and Paradichlorobenzen-Ag-NPs were tested often 24 hours. The result showed when we added Silver Nanoparticles to chemical insecticides it increase the mortality of

Anthrenus Verbasci in different concentrations compared to chemical insecticides only, and  $LC_{50}$  of Carbon disulfide Ag-NPs was 0.712 while that  $LC_{50}$ of carbon disulfide was 1.559 indicating that Ag-NPs improved performance of chemical insecticides in mortality of larva. Also Carbon disulfide - Ag-NPs while that of Paradichlorobenzen- Ag-NPs was 0.781 indicating that Carbon disulfide- Ag- NPs was the effective Nano chemical insecticides in different concentrations for controlling *Anthrenus Verbasci*, where it possess the lowest value of  $LC_{50}$  and  $LC_{90}$  as in (table 2, figure 7).

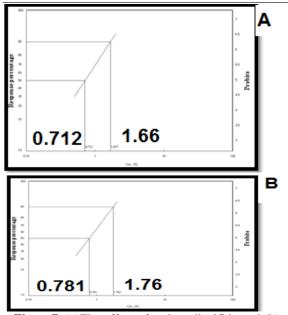
 Table 1. The mortality of Anthrenus Verbasci larvae by Cinnamon and Eucalyptus oils before and after added Ag-NPs

Volatile oils	Concentr	Mortality	LC50	LC90	Slope ±SE
	ation %	%			
Cinnamon oil	0.5	30			
	1	58	0.854	3.147	$2.26\pm0.24$
	2	75			
	3	92			
Cinnamon oil	0.5	60			
Ag-NPs			0.442	0.998	$3.62\pm0.51$
C	1	85			
	1.5	100			
Eucalyptus oil	0.5	20			
	1	45	1.225	4.743	2.18±0.24
	2	60			
	3	85			
Eucalyptus oil	0.5	40			
Ag NPs	1	65	0.639	1.647	$3.12\pm0.41$
	1.5	92			
	2				

Table 2. Mortality of Anthrenus Verbasci larvae by Carbon disulfide and Paradichlorobenzen Ag-NPs

Volatile oils	Concentr ation %	Mortality %	LC50	LC90	Slope ±SE
Carbon disulfide	1	35			
	2	55			
	3	75	1.559	5.527	2.33±0.30
	4	85			
Carbon disulfide	0.5	35			
Ag-NPs	1	60	0.712	1.667	$3.44\pm0.32$
	1.5	85			
	2	100			
Para-	1	15	2.896	8.848	2.64±0.33
dichlorobenzene	2	25			
	3	50			
	4	70			
Paradichloroben	0.5	30	0.781	1.762	$3.63\pm0.32$
zen Ag-NPs	1	55			
	1.5	80			
	2	98			

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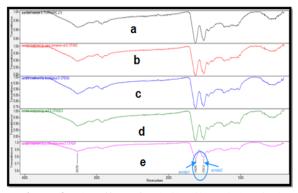


**Figure7.** (a)The effect of carbon disulfide and (b) Paradichlorobenzen Ag-NPs concentrations on larval bioassay

# **3.3.** Effect of volatile oils and chemical insecticides were added to silver nanoparticles on dyed wool samples

#### 3.3.1. FTIR-ART Analysis:

Infrared absorption of wool show characteristic absorption bands assigned to the peptide bonds that are known as amide I, amide II, and amide III.



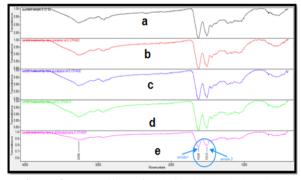
**Figure 8.** The effect of volatile oils and chemical insecticides Ag-NPs on functional groups for dyed woolen samples, (a) untreated wool, (b) cinnamon oil, (c) Eucalyptus oil, (d) carbon disulphide and (e) paraadichlorobenzene

Infrared absorption spectra of wool show characteristic absorption bands assigned to the peptide bonds (-CONH-) that are known as amide I, amide II, and amide III. Amide I is mainly related with the C=O stretching, and it occurs in the range of 1,660-1,600 cm<sup>-1</sup>. Amide II falls in 1,540-1,520 cm<sup>-1</sup>

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range and is assignable to N–H bending and C–N stretching vibrations. Amide III occurs at  $\approx$  1,230 cm<sup>-1</sup> and is related to N–H bending and C–N stretching. N-H stretching and C-H stretching bands occurred at (3200 -3400) cm<sup>-1</sup> and (3100 -2800) cm<sup>-1</sup> ranges respectively [40].

The results in (figure 8) showed the FTIR-ATR spectrum of the untreated wool sample, compared to that of treated samples with volatile oils and chemical insecticides were added to Ag NPs. It is found that amide I of untreated wool sample appeared at 1625 cm<sup>-1</sup> while that of the sample treated with cinnamon oil Ag-NPs was shifted to a higher wavenumber value from 1625 cm<sup>-1</sup> to 1640 cm<sup>-1</sup> indicating breakdown some of hydrogen bonds between protein chains and thus wool in this instance became more susceptible to deterioration. The results also showed no change in the frequencies of amide I bands of treated wool samples with Eucalytus oil Ag-NPs, Carbon disulphide Ag-NPs and Paradichlorobenzene Ag- NPs, 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 shows slight decrease in bands intensities than that of the untreated sample.



**Figure 9.** The effect of volatile oils and chemical insecticides Ag-NPs on functional groups for dyed woolen samples attacking by *Anthrenus verbasci*, (a) untreated wool, (b) cinnamon oil, (c) Eucalyptus oil, (d) carbon disulphide and (e) paraadichlorobenzene

The results in (figure 9) showed no strong effect on the functional groups of wool attacked by *Anthrenus verbasci*, treated with volatile oils and chemical insecticides were added to Ag-NPs. Where, there were no observed changes in amide I, II and III frequencies. All treated woolen samples shows slight decrease in bands intensities than that of the untreated sample.

#### 3.3.2. XRD Analysis:

X-ray diffraction studies lead us to understand the crystalline structure and the degree of crystalline

portion. As any treatment that can change the morphology may sometimes lead to crystallization or decrystallization, it was thought worthwhile to investigate the changes caused by volatile oils and chemical insecticides which were added to Ag NPs treatment on the different fibers [41]. 20 peaks in the diffractogram at 20.8° and 9.7° are corresponded to wool [42, 43].

X-ray diffraction (XRD) analysis of untreated and treated samples is presented in two ways. The first way presents the percentage of the crystallinity index of the untreated samples and of those treated by volatile oils and chemical insecticides were added to Ag-NPs. There is a slight decrease in the crystallinity index of the wool after treatment as presented in (Table 3). The second way presents the peak intensity (counts) in both the amorphous and crystalline regions. The results showed that the treatments of wool samples caused a slight increase in the peak intensity in both the amorphous and crystalline regions. Furthermore, the ratio of the crystalline and amorphous fractions barely changed. Changes in crystallinity by volatile oils and chemical insecticides added to Ag NPs treatments may be indicated by the tensile properties of the samples.

The result also showed that crystallinity index of all treated samples increased compared to that of standard sample, this may be due to the formation of more secondary bonds between protein chains and the chains became orderly arranged.

Table 3. The crystallinity index of volatile oils and chemical insecticides with Ag-NPs

Samples	Crystalline area		Amorphous area		Crystallinity index (%)	
	20	(°) count	<b>2θ</b> ( °)	counts		
Standard sample	21.40	290	9.56	400	55.3	
Sample treated by cinnamon oil Ag NPS 1.5%	20.60	314	8.5	500	58.7	
Sample treated by Eucalyptus oil Ag NPS 1.5%	21.40	320	8.4	500	60.7	
Sample treated by carbon disulfide Ag NPS e 1.5%	21	380	9.2	500	56.2	
Sample treated by Paradichlorobenzen Ag-NPS 1.5%	21	350	9	500	57.1	

## **3.3.3.** Mechanical properties

In order to estimate the effect of volatile oils and Chemical insecticides were added to Ag-NPs on the mechanical properties of dyed woolen samples with Natural turmeric dye and mordant by Potash Alum, tensile strength and elongation % were measured. The results in (table 4) the woolen samples treated

with volatile oils (cinnamon, Eucalyptus oil) and

paradichlorobenzene were added to Ag-NPs showed an increase in tensile strength and elongation % compared to untreated samples. While, Sample treated with carbon disulphide were added to Ag-NPs reduced the mechanical properties compared to the standard sample.

 Table 4. The effect of volatile oils and Chemical insecticides added to Ag-NPs on dyed woolen samples with turmeric, mordant by Potash Alum

Sample code	Elongation (%)	Change %	Tensile strength (kgf/mm <sup>2</sup> )	Change %
Standard sample	0.5689		16.0	
Sample treated with	0.5753	+1.1	17.3	+ 8.3
cinnamon oil Ag- NPS 1.5%				
Sample treated with	0.5844	+ 2.7	20.3	+27.0
Eucalyptus oil Ag- NPS				
1.5%				
Sample treated with carbon	0.4993	- 12.6	15.0	- 6.2
disulfide Ag-NPS 1.5%				
Sample treated with	0.5697	+0.14	19.7	+22.9
Paradichlorobenzen Ag-				
NPS 1.5%				

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	•			
Sample code	Elongation (%)	Change %	Tensile strength (kgf/mm <sup>2</sup> )	Change %
Standard sample	0.3873		11.3	
Sample treated with	0.4766	+23.0	13.0	+ 14.7
cinnamon oil Ag- NPS 1.5%				
Sample treated with	0.4252	+ 9.7	12.7	+11.8
Eucalyptus oil Ag- NPS				
1.5%				
Sample treated with carbon	0.3905	+ 0.8	12.3	+ 8.8
disulfide Ag-NPS 1.5%				
Sample treated with	0.3022	- 22.4	11.0	- 2.9
Paradichlorobenzen Ag-				
NPS 1.5%				

**Table 5.** The effect of volatile oils and Chemical insecticides added to Ag-NPs on dyed woolen samples with turmeric and mordanted by Potash Alum and attacking by *Anthrenus verbasci*

The results in (table 5) Changes in the mechanical properties could be attributed to changes in crystalline orientation. The woolen samples treated with volatile oils and carbon disulfide Ag-NPs showed an increase in tensile strength and elongation % compared to untreated samples. While, Sample treated with paradichlorobenzene Ag- NPs reduced the mechanical properties compared to the standard sample.

# CONCLUSIONS

The present study concluded that 1.5% (v/v) concentration of cinnamon oil were added to silver nanoparticles 5% was the most effective on controlling Anthrenus verbasci, it achieved 100% larval mortality compared to other materials in the same concentration. Volatile oils Ag-NPs improved mechanical properties of dyed woolen textile more than chemical insecticides Ag-NPs compared to standard sample. Both volatile oils and chemical insecticides were added to Silver Nanoparticles increased the crystallization index of dyed woolen textile compared to standard sample and there is a slight increase in the peak intensity (counts) in both the amorphous and crystalline regions. Volatile oils and chemical insecticides were added to Silver Nanoparticles have nearly no effect on the chemical functional groups of dyed woolen textile with natural turmeric dve.

A detailed study should be made on the different colors found in the archaeological textiles to ensure that they were not affected by the materials in this research. There is another research talking about the measurement of color change and the effectiveness of this material on the morphology of textile.

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# **Conflicts of Interest**

The authors declare no conflict of interest.

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