

Preparation of a nano insecticide using dried *Lawsonia Inermis* leaves with TeO₂ and investigate its effectiveness against house flies

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Abstract

Background: *Lawsonia Inermis* (henna) extract and trillium dioxide (TeO₂) has many applications among them is biomedicine and pharmaceutical industry.

Objective: the current study aimed to prepare extract from henna leaves, and synthesize novel nanoparticles derived from henna extract and TeO₂, characterized them and test its bioactivity against house flies.

Materials and Methods: *Lawsonia Inermis* (henna) leaves and TeO₂ were collected from local markets in Baghdad, Iraq. The extraction of henna leaves was prepared and the TeO₂ nano particles was synthesized according to a certain procedures, the nanoparticles of the combination of henna extract and TeO₂ was prepared a characterized successfully using different techniques such UV-Vis, XRD, AFM. The bioactivity of all the compounds was evaluated and tested.

Results: The UV-Vis, XRD and AFM spectra was collected and evaluated. The bioactivity was also examined against house fly and the collected data was clear and very optimistic. It was found that the highest effect was of trillium nanoparticles prepared at a concentration of 500ppm to reach 100% to kill the 1st larval. It also shows clear decreases in the effect at the same concentration above for the 1st larval stage when using only trillium oxide or only henna leaves extract, 30% and 10% respectively,

Conclusions: Many conclusions were found in this work, among them are; extract of *Lawsonia Inermis* leaves was prepared successfully, novel nanoparticles were synthesized from henna extract and TeO₂ nanoparticles (HennaTeO₂NPs) was prepared, characterization of the novel hennaTeO₂NPs was carried out, and the bioactivity of the novel nanoparticles was carried out against house fly and it was noticed that the novel henna nanoparticles shows best activity.

Keywords: Henna; Trillium dioxide; Nanoparticles; House flies

1. Introduction

Musca domestica is considered one of the most common and dangerous pests in our ecosystem because of its ability to transmit more than 100 types of pathogens, and the extermination of this insect means the extermination of many diseases that it spreads and causes, therefore, chemical pesticides were used extensively to try to control this scourge, which was distinguished by its ease of application, speed of impact, and low cost. However, the negative and harmful effects of excessive use of chemical pesticides were very clear on the environment and non-target organisms, especially humans. Thus, it was necessary to find new alternative materials that are not harmful to the environment, but have a clear effect on pests in the ecosystem, so nanopesticides prepared from some parts of plants appeared with inorganic nanoparticles such as TeO₂. [1, 2].

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Tellurium dioxide (TeO₂) nanoparticles are particles of tellurium dioxide with sizes ranging from 1 to 100 nanometers. They have potential applications in various fields such as electronics, optics, catalysis, and biomedicine. In electronics, TeO₂ nanoparticles have been studied as a possible replacement for conventional silicon-based semiconductors due to their unique electrical and optical properties. They have also been explored as potential materials for advanced memory devices and solar cells, while in optics, TeO₂ nanoparticles have been used in the production of optical fibers for telecommunications, due to their high refractive index and low dispersion. They can also be used in the production of optical coatings and lenses. In catalysis, TeO₂ nanoparticles have been investigated for their potential as catalysts for various reactions, including the reduction of nitroarenes and the oxidation of alcohols [3-5].

In biomedicine, TeO₂ nanoparticles have been studied for their potential use in drug delivery and cancer therapy. They can be functionalized with various biomolecules and have been shown to be biocompatible and capable of penetrating cell membranes [6].

Henna leaves are a rich source of organic compounds, such as lawsone, which can act as reducing agents for the synthesis of nanoparticles. The preparation of nanoparticles derived from dried henna leaves can be achieved through various methods, including the following [7, 8]:

- Green synthesis method: This method involves the use of water as a solvent and mild reaction conditions to extract the organic compounds from dried henna leaves and use them as reducing agents for the synthesis of nanoparticles.
- Hydrothermal method: This method involves the reaction of henna leaf extract and a metal salt under high-temperature and high-pressure conditions to form nanoparticles.
- Co-precipitation method: This method involves the simultaneous precipitation of metal ions and a precipitating agent, such as ammonium hydroxide, with the henna leaf extract to form nanoparticles.
- Ultrasonic method: This method involves the use of ultrasonic waves to promote the formation of nanoparticles from the henna leaf extract and a metal salt.

After the synthesis of the henna leaf-derived nanoparticles, their properties can be characterized using various techniques, such as transmission electron microscopy, X-ray diffraction, and Fourier-transform infrared spectroscopy, to determine their size, shape, and crystal structure [9].

The prepared nanoparticles can be used for various applications, such as drug delivery, bioimaging, and catalysis, due to their biocompatibility, stability, and good dispersibility. However, more research is needed to fully understand the properties and potential applications of these nanoparticles [9-10].

The current work is to prepare novel nanoparticles that derived from henna leaves extract and TeO₂, characterized them and test its bioactivity against house fly.

2. Material and methods

2.1. Preparation of *Lawsonia Inermis* (henna) leaves Extract

Henna leaves was collected from local markets in Baghdad, Iraq, the preparation of henna leaf extract involves several steps, which are as follows:

- Collection and drying of henna leaves: The henna leaves are harvested and dried in a well-ventilated area until they are completely dry.
- Grinding of dried henna leaves: The dried henna leaves are ground into a fine powder using a mortar and pestle or a mechanical grinder.
- Extraction of henna powder: The henna powder is mixed with a solvent, such as water or ethanol, and heated for a certain period of time to extract the organic compounds from the henna leaves.
- Filtration of the extract: The extract is filtered through a filter paper or a mesh to remove any impurities or undissolved particles.
- Concentration of the extract: The extract is concentrated using evaporation or other methods to obtain a concentrated extract, pH must be adjusted to 4.1.

Storage of the extract: The henna leaf extract can be stored in a dark and cool place at 4 °C for later use.

The extracted henna leaf extract can be used for various applications, such as in the synthesis of nanoparticles, dyeing hair and skin, and as a natural remedy for various ailments. It is important to note that the properties of the extract, such as its color and concentration, may vary depending on the solvent used and the method of extraction.

2.2. Preparation of Tellurium dioxide nanoparticles (TeO₂NPs)

Tellurium dioxide was bought from the scientific chemical stores in Baghdad, Iraq, the preparation of titanium dioxide nanoparticles (TeO₂NPs) using deionized distilled water is start by preparing a solution of titanium precursor such as titanium isopropoxide (TIP) in deionized distilled water. The concentration of TIP can vary depending on the desired size and concentration of TeO₂NPs. Add a surfactant such as polyvinylpyrrolidone (PVP) to the solution. The surfactant helps to stabilize the nanoparticles and prevent them from agglomerating. Stir the solution at room temperature for a few minutes to ensure complete mixing of the TIP and surfactant. Heat the solution under reflux conditions for several hours. Refluxing involves boiling the solution and continuously condensing the evaporated solvent to keep the reaction mixture at a constant temperature. After refluxing, cool the solution to room temperature and centrifuge it to separate the TeO₂NPs from any unreacted precursor or byproducts. Wash the TeO₂NPs with deionized distilled water to remove any residual surfactant or impurities. Dry the TeO₂NPs in an oven at a low temperature (around 60-80°C) until all the solvent has evaporated, and the colour is changed. The pH must be around 7 [11 12].

2.3. Characterization of TeO₂ nanoparticles using UV-visible Spectroscopy, Atomic Force Microscopy (AFM), and X-Ray Diffraction

- UV-Visible Spectroscopy: This technique can be used to measure the absorption spectrum of TeO₂ nanoparticles. TeO₂ nanoparticles will have characteristic absorption peaks in the UV-Vis range that can be used to determine their size and shape. The absorption spectrum can be measured using a UV-Vis spectrophotometer.
- Atomic Force Microscopy (AFM): This technique can be used to determine the size, shape, and surface morphology of TeO₂ nanoparticles. AFM measures the surface topography of the nanoparticles by scanning a sharp tip over their surface. This produces a three-dimensional image of the nanoparticles that can be used to determine their size and shape.
- X-Ray Diffraction: This technique can be used to determine the crystal structure of TeO₂ nanoparticles. X-ray diffraction patterns will show characteristic peaks that can be used to identify the crystal structure of the nanoparticles. The XRD pattern can be obtained using an X-ray diffractometer.

The Absorption Spectrum: This technique can be used to measure the optical properties of TeO₂ nanoparticles. The absorption spectrum will show characteristic peaks that can be used to determine the bandgap energy of the nanoparticles. The absorption spectrum can be measured using a UV-Vis spectrophotometer [9, 10].

By combining the above techniques, it is possible to fully characterize the size, shape, crystal structure, and optical properties of TeO₂ nanoparticles. This information is important for understanding the properties of the nanoparticles and for optimizing their use in various applications.

2.4. Insect breeding and treatment mechanism with prepared pesticides

Adults of house flies were bred for several generations in the animal house of the Department of Life Sciences at Al-Mustansiriyah University before obtaining the treated larval stages to evaluate the effectiveness of the extracts used in concentrations (100, 300, 500 ppm) for each extract where the prepared concentrations were replaced with water during the food fusion process, and the experiments were carried out in plastic cups, with three replicates for each treatment. As for the treatment of adults, it consisted of preparing a sugar solution with a concentration of 10%, consisting of adding 9 ml of each concentration used, and adding an gram of sugar to it, then we put a cotton soaked in sugar solutions for each concentration used and put it in a glass dish inside a cage and put on each cotton 10 adults at the age of 24 hours [2]

2.5 Bioactivity of Henna TeO₂ Nanoparticles against House Flies

After the synthesis of TeO₂ nanoparticles with henna extract (HennaTiO₂NPs), the nanoparticles should be characterized for size, shape, and purity using techniques such as UV-Vis spectroscopy, X-ray diffraction, and atomic force microscopy.

The bioactivity of the HennaTeO₂NPs was evaluated against house flies. House flies can be placed in a chamber and exposed to the henna extract with nanoparticles. The mortality rate of the house flies can be measured over time. The data collected from the house fly bioassay can be analyzed using statistical methods to determine the effectiveness

against house flies. The LC50 (concentration required to kill 50% of the house flies) can be calculated to determine the potency of the extract with nanoparticles. Then comparison with control that should be included in the study to compare the mortality rate of the house flies exposed to the henna extract with nanoparticles with those exposed to the solvent or nanoparticles alone [9, 10].

3. Results and discussion

Figure 1 shows the colors of the three synthesized solutions

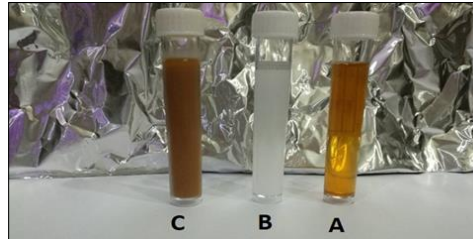


Figure 1 A- The first figure represents an aqueous extract of dried henna leaves; B- The second figure is a solution of trillium oxide; C- The third figure represents the nanoparticles prepared from mixing the two previous compounds

It is clear from the colour changes that confirm the formation of the new nanoparticles (TeNPs), this result match with the findings of (Hameed al., 2019 [11]; Miu and Dinischiotu 2022 [12].

Figure 2 shows the absorption spectrum of UV-Visible of the extract of henna leaves.

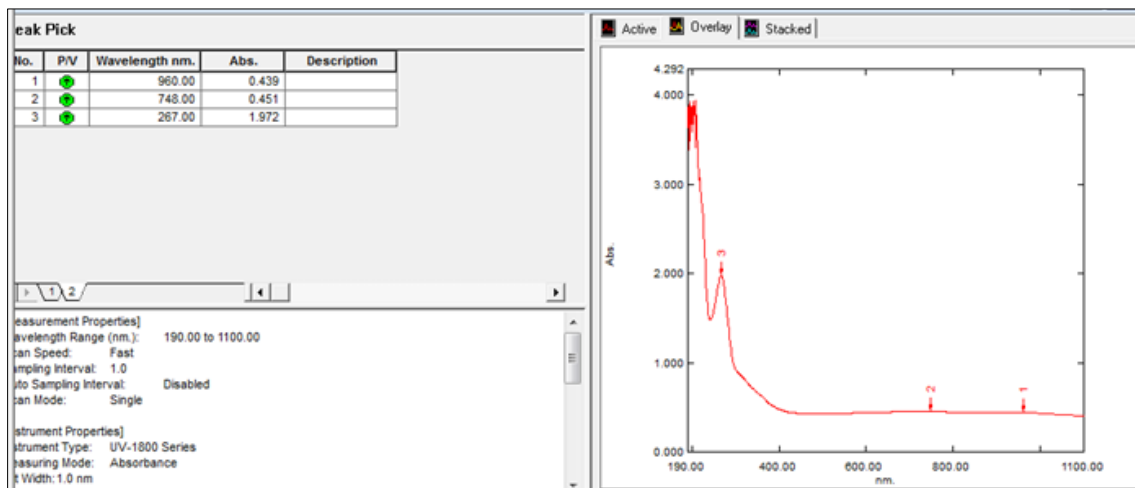


Figure 2 UV-Visible spectrum of Henna Extract

The figure represents the optical absorbance spectrum of the henna solution prepared by the previously used dissolving method, and the absorbance was measured within wavelengths between (190-1100nm), it is noticed that there is stability at the beginning of the region at wavelengths between (190-220nm). Followed by a sharp decrease at wavelengths 267nm, with a value of 1.97, after that, the stabilization phases begin to reach complete stability at 748 nm with a value of 0.45 and at a wavelength of 960nm with a value of 0.43.

Figure 3 shows the Gaussian distribution of the granularity rate of the TeNPs.

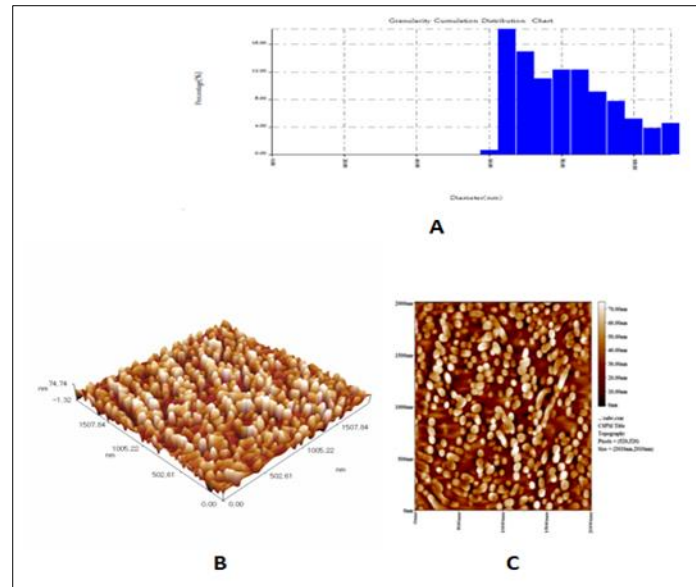


Figure 3 A- The diagram of the cumulative size distribution of granules B and C- Represents the topographic images and volume distribution of the TeNPs

The diagram in figure 3 A of the cumulative size distribution of granules with an average grain size of (78.45nm), which is a very acceptable size for nanoparticles as prescribed by (Sharma et al., 2022) [14] which indicate the perfect size of the nanoparticles must ranged from 10 to 80nm and also explained by (Sundus et al., 2023) [12], while the topographic pictures in figure 3, B and C shows the homogeneity of the nanoparticles, this finding is the same finding of (Abbas et al., 2020 [15] ; Benedis et al., 2023 [16]), while B & C in figure 3 shows the topographic images and volume distribution of nanoparticles which indicates high homogeneity and volume as well as high density, this result match with the findings of (Bolean et al., 2017 [17]; Zheng 2022 [18]).

Figure 4 represents the X-ray diffraction spectroscopy of the henna (*Lawasonia Inermis*) nano-extract deposited on glass by the drop-drop method. It is noticed the appearance of a single peak at the angle of 24.29, and using the Scherrer equation, the crystal size was found at 1nm, while the atomization density was 97.9 and the microcompliance was approximately 0.03×10^{16}

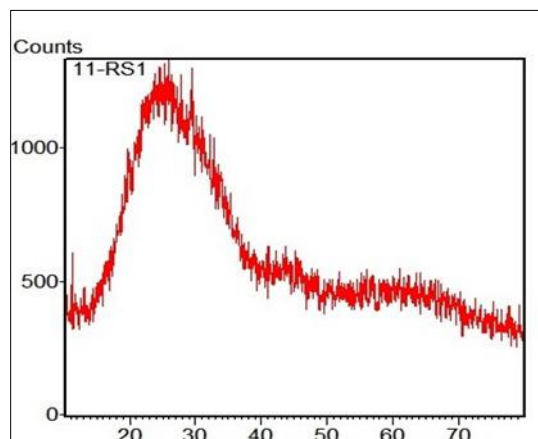


Figure 4 X-Ray diffraction spectrum

Figure 5 below shows the examination result of a solution of nano *Lawasonia Inermis* leaf extract deposited on glass by the drop method and prepared by the melting method at a temperature of 38 °C, it is noted that there are irregular spherical shapes in a granular shape, estimated at an approximate size between (112-66 nm), this result match with the findings of (Gajjar et al., 2021) [19].

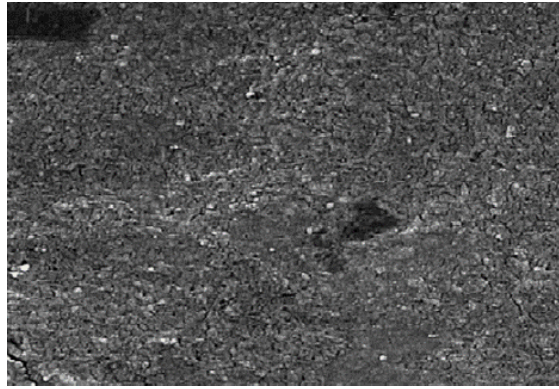


Figure 5 Examination result of nano henna extract

Tables 1, 2, and 3 represent the percentage of fatal in house flies using only henna extract, or only TeO₂, and a solution of henna nanoparticles with TeO₂

Table 1 The percentages of House Flies insects killed by henna plant extract

	100ppm	300ppm	500ppm
1st larva	6.6	10	20
2nd larva	3.3	10	10
3rd larva	0	0	3
Adult	13.3	16	16

Table 2 The percentage of house flies killed by Trillium oxide

	100ppm	300ppm	500ppm
1 st larva	16	23	30
2 nd larva	10	16	26.6
3 rd larva	3.3	6.6	16.6
Adult	13	13	20

Table 3 The percentage of house flies killed by nano extract

	100ppm	300ppm	500ppm
1 st larva	93	96	100
2 nd larva	60	63.3	73.3
3 rd larva	50	63.3	70
Adult	73	76,6	83.3

It appears clear from the results in tables (1, 2, 3) that the highest effect of the concentrations used in controlling house flies in all treatment stages was 500 for nanoparticles prepared for all stages used, respectively. The most sensitive stage was the 1st larval stage, in which the killing rate reached 100% for the same concentrations above (500ppm).

Among the effects are the deformations that appeared in the form of charring of the 2nd larval, as in figure 6 which is attributed to the penetration of nanoparticles into the cuticle layer, causing its destruction, which makes it vulnerable to water loss and charring (Rasha et al., 2019) [9].

It was also observed failures in the backs of the wings and loss of feet for adults emerging from a 3rd larval stage treated with the prepared nanoparticles due to the production of effective forms of oxygen which after the entry of the prepared nanoparticles into the digestive system as a result of feeding causes killing and deformities of the treated larvae, which considered as a major carrier of many pathogens in humans (Sundus et al., 2023) [10].

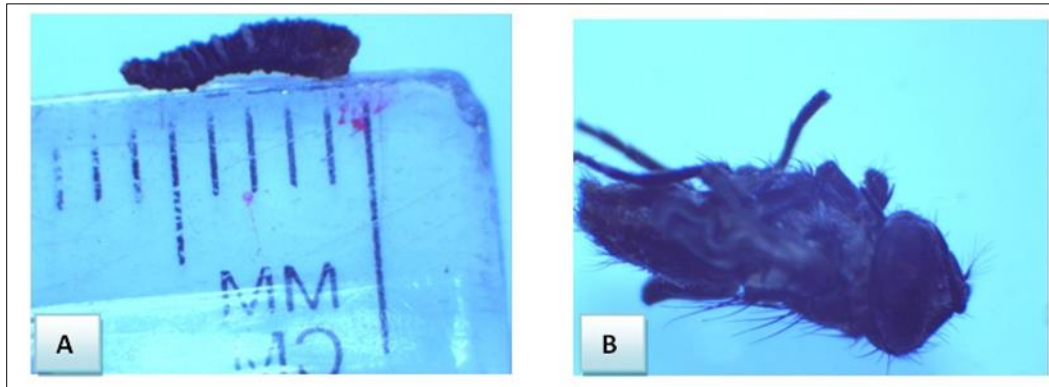


Figure 6 Phenotypic variation that noticed in A:larval Charring; B: The absence of wings and deformities of the limbs

4. Conclusion

- Extract of *Lawsonia Inermis* (henna) leaves was prepared successfully.
- Nano particles were synthesized from Trillium dioxide.
- Novel nanoparticles were prepared from henna extract and TeO₂ nanoparticles (HennaTeO₂NPs).
- Characterization of the novel hennaTeO₂NPs was carried out.
- Bioactivity of the novel nanoparticles was carried out against house fly and it was noticed that the novel HennaTeO₂NPs shows best bioactivity compared with other solutions.

Compliance with ethical standards

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