Comparative Killing Activity of Different Nanoparticles and Nano-composites Based on *Dermanyssus gallinae*

Sameh Ismail, Gehad Mohamed, Aziza Amer, Mohamed Amer

Egypt Nanotechnology Center, Cairo University, El-Sheikh Zayed, 6th October, Giza, Egypt.

Chemistry Department, Faculty of Science, Cairo University, 12613, Giza, Egypt.

Department of Pharmacology, Faculty Veterinary Medicine, Cairo University, 12211 Giza, Egypt.

Department of Poultry Diseases, Faculty Veterinary Medicine, Cairo University, 12211 Giza, Egypt.

Corresponding author. E-mail: drsameheltayer@yahoo.com

Received: Jan. 10, 2020; Accepted: Oct. 10, 2020; Published: Nov. 27, 2020

Citation: Sameh Ismail, Gehad Mohamed, Aziza Amer, and Mohamed Amer. Comparative Killing Activity of Different Nanoparticles and Nano-composites Based on *Dermanyssus gallinae*. *Nano Biomed. Eng.*, 2020, 12(4): 338-350.

DOI: 10.5101/nbe.v12i4.p338-350.

Abstract

Silver (Ag NPs) and magnetite nanoparticles (MANPs) were synthesized and characterized using X-ray powder diffraction (XRD), X-ray fluorescence (XRF), scanning electron microscope (SEM) and transmission electron microscope (TEM), atomic force microscope (AFM) and Raman spectroscopy. XRD, XRF and Raman spectroscopy results of MANPs and Ag NPs confirmed their synthesis without any undesired impurities from synthesis method. TEM, SEM and AFM images of Ag NPs illustrated homogenous spherical particles with the size of about 5 nm, while MANPs had spherical to semi-cubic with the size of 20 nm. Both silver-essential oils and magnetite-essential oils nanocomposites were synthesized by sonochemical method with the ratio of 1:1. In-vitro contact effect of commercial plant essential oil mixture with synthetic silver and magnetite nanoparticles was studied. Comparative killing study was carried out by direct contact spray of groups of saline, silver nanoparticles, magnetite nanoparticles, essential oils, silver-essential oil nanocomposite, and magnetite-essential oil nanocomposite on poultry red mite *Dermanyssus gallinae* (*D. gallinae*). Activity and changes occurred were examined under stereomicroscope for 3 h. Interestingly, highest killing activity was obtained with silver-essential oil nanocomposite and silver nanoparticles in comparison with magnetite nanoparticles, essential oil and magnetite-essential oil nanocomposite. As a result, silver-essential oil nanocomposite and silver nanoparticles could be beneficial and involved in *D. gallinae* (red mite) control strategies in poultry industry.

Keywords: Red mites, Nanosilver, Nanomagnetite, In-vitro study, Contact, Plant oil mixture

Introduction

*Dermanyssus gallinae* (*D. gallinae*) infestation still represents a major threat to the egg production industry, posing serious animal health and welfare problem in poultry industry all over the world; the economic the egg production industry, adversely affecting productivity, impacting public health and their role as a disease vector are well known [1]. The veterinary and human medical impact needs adequate therapeutic measures to control this parasite [2-4]. This blood feeding mite causes production losses due to irritation and anemia, but may even cause death of its host and is also involved in transmission of many pathogenic agents responsible for severe outbreaks in both animals and humans [5,6]. Repeated
use of various synthetic contact acaricides such as permethrin, carbaryl, diazinon, dichlorvos are the most commonly used for control D. gallinae infestation leads to drug chemical pollution. The development of resistance [1] and further, the residues in eggs and meat are a highly important problem for human health [7,8]. Route avoidance of these drawbacks could be the use of vegetable organic pharmaceuticals [9]. There is constant need for alternative control measures to maintain a good animal health in avairy systems [10].

In the last thousand years ago, plant extracts was used as acaricide / insecticide in Asia. Plant essential oils may be an alternative source of products used in the treatment of mite infestations, because they are rich sources of bioactive chemicals [11]. Kim et al. [11] studied the activity of 56 plant essential oils against poultry collected adult D. gallinae using direct contact and fumigation methods and concluded that the effect of these essential oils was largely due to action in the vapour phase. The acaricidal effect of eleven essential oils against poultry red mite D. gallinae was tested in vitro using the direct contact method. The results revealed that oils of sweet basil, coriander peppermint and summer savory were the most effective [12]. Neem oil, pure garlic juice, eucalyptus essential oils, and wood vinegar reduced, killed and control red mite [13-16]. Garlic oil has medicinal use and pesticidal effects [17]. Nanotechnology has become one of the most promising new generations for pest control in the recent years [18], could provide cost-effective solution to some of the most challenging environmental cleanup problems [19], help to produce new pesticides, insecticides and insect repellants [20]. Silver nanoparticles were approved by many agriculture researchers as insecticides which highly affect both adults and larvae [21] and also for insect control [22]. One proposed mechanism by which Ag NPs produced toxicity is by enhancing intracellular levels of reactive oxygen species (ROS). ROS, when formed, produce subsequent cellular damage such as disrupting membrane integrity and damaging proteins and DNA [23]. Consumption of Ag NPs was resulted in cuticular demelanization in Drosophila [24]. Adult Tetranychusurticaespider mite mortality using different concentrations of silver nanoparticles by leaf spray showed that at 100 ppm concentration, more than 50% of mites were died while using leaf dipping methods, spraying with 3000 ppm concentration showed a mortality effect more than 90% [25]. Magnetite nanoparticles have antibacterial properties for both Gram positive and negative strains [26]. The present study was planned to synthesis silver nanoparticles, magnetite nanoparticles, silver-essential oil nanocomposite and magnetite-essential oil nanocomposite and evaluating their killing activity with respect to saline and essential oils by direct contact or spraying them on mites and observing the changes tooks place under stereomicroscope.

**Experimental**

All used chemicals are laboratory grade including ferric chloride hexahydrate (FeCl$_3$·6H$_2$O), ferrous chloride (FeCl$_2$), tri-sodium citrate (TSC), polyethylene glycol (PEG 600) and AgNO$_3$ manufactured by Sigma Company, India. Ammonia solution (34%) was supplied from Al-Nasr Company, Egypt.

**Red mite collection**

Three hundred and sixty red mites (D. gallinae) of different stages were collected from naturally infested 5 poultry lying flocks. Mites were collected with the aid of a brush in plastic jars and were used for tests within 2 days of collection. Until testing, the mites were kept at 24 °C under a photoperiod of 16:8 h (light/dark). The collected mites were used in 3 replicates for testing of efficacy of mixture of vegetable oil extracts (Allisal®) alone or with either silver or magnetite nanoparticles solutions.

**Essential oils**

Allisal® commercial: a liquid supplement product of Envisal GmbH, Eulenbusch 10a, 21391 Repented, Germany. Allisal was an aqua suspension of natural plant oils composed of 2.5% garlic oil, 4.2% rosehip oil, 4.8% rapeseed oil and 14% polysorbate. It was used at a rate of 0.4% dilution in deionized water for testing its in vitro effect on red mites.

**Synthesis**

**Synthesis of Ag NPs**

Synthesis of Ag NPs was done by co-precipitation method using TSC as reducing agent and capping agent in the same time [29]. AgNO$_3$ solution (0.01M) was dissolved in 100 mL deionized water, heated to boiling then was added 0.1 M TSC drop by drop with constant stirring and heated until the color of mixture become pale yellow. The solution was cooled to room temperature under dark condition to avoid lights.
Synthesis of MANPs

MANPs were synthesized also by co-precipitation method using ammonia as reducing agent and PEG 600 as capping agent [30]. FeCl$_3$·6H$_2$O solution (0.2 M) was added to 0.1M FeCl$_2$ solution and the mixture was heated with constant stirring at 60 °C for 15 min until orange solution was formed without any powder precipitate. Then, ammonia was added drop by drop until all solution become black. Finally, the pH was adjusted to 12 by adding more ammonia solution. Wash using deionized water and external magnets several times then adding PEG to the final powder and the mixture was subjected to sonication for 1h in condition of 0.2 cycle and 10% amplitude.

Synthesis of silver-essential oil nanocomposite

3 mL of 75 ppm silver nanoparticles was added to 3 mL essential oil then the mixture was subjected to sonication under condition of 0.5 cycle and 50% amplitude for 20 minutes until pale yellow color was obtained.

Synthesis of magnetite-essential oil nanocomposite

3 mL of 100 ppm magnetite nanoparticles was added to 3 mL essential oil then the mixture was subjected to sonication under condition of 0.1 cycle and 10% amplitude for 30 minutes until brown color was obtained.

Characterization of nanoparticles

The aim of characterization of MANPs and Ag NPs was to determine physiochemical properties which influence their bioactivity [31]. Characterization was classified into three sectors namely composition, microscopic and physical sectors. Composition sector was carried out by XRD (D8 Discovery-Bruker Company) at condition of 40 KV and 40 AM (1600W) at speed scan 0.01 and 2(θ) range from 10° to 80°, XRF (X-Met 8000 Oxford Instruments Company.) and Raman spectroscopy (Lab. RAM-HR Evolution Horiba Co.) with acquisition time 5 sec (for Ag NPs) and 7 sec (for MANPs), accumulations 5 (for Ag NPs) and 1 (for MANPs) without spike filter and delay time and objective was X50 (for Ag NPs) and X100 (for MANPs) with grating 1800 (450-850 nm) and ND filter 50% (for Ag NPs) and 5% (for MANPs). The laser type was green of 532 nm. Microscopic sector was carried out by transmission electron microscope (TEM) model EM-2100 High-Resolution at magnification 25X and voltage 200 kV. Scanning electron microscope (SEM) was carried out by Jol 2000, Japan and atomic force microscope (AFM) was done using AFM 5600LS Agilent Technology Company. Physical sector was carried out to determine the surface area, pore volume pore size and, average particles radius by surface area and pore size analyzer model Nova Touch LX2 manufactured by Quantachrome Company. Killing activity observed under stereo microscope (model M165 C-Leica Biosystems Company). DLS and zeta potential were carried out by instrument manufacture by Malvern instruments Ltd. Model of Nano Sight NS500 to determine the size and zeta potential of nanoparticles and nanocomposites. Optical and magnetic properties for nanoparticles and nanocomposites were measured by spectrophotometer model of Shimadzu d50 and vibrating sample magnetometer (VSM) instrument model 8600 series manufactured by Lake Shore Cryotronics, Inc. Killing activity was observed under stereomicroscope (model M165 C-Leica Biosystems company).

In-vitro study

Grouping and treatment using direct contact with mixture of plant oils and/or MANPs and Ag NPs solutions was described. Sex groups, 20 mites each, were transferred to separate glass petri dishes and exposed to the following treatments: Group 1 kept as non-treated control. Group 2 was sprayed with 4% essential oil. Groups 3 and 4 were sprayed with MANPs (100 ppm/mL) and Ag NPs (75 ppm/mL), respectively. Group 5 was sprayed with magnetite-essential oil nanocomposite (1 mL of 75 ppm silver and 1 mL essential oil). Finally, for group 6, magnetite-essential oil nanocomposite (1 mL of 100 ppm magnetite and 1 mL essential oil) was used. All treated mite groups were observed under stereomicroscope at 1, 2 and 3 h after treatment where non-moving mites considered dead. The number of dead and alive mites was counted in all the plates. The efficacy (E) of each tested material at each observation time was calculated as follows:

$$E\% = \frac{(A - B)}{A} \times 100,$$

where A is the number of mites before spray and B is the number of mites after spray.

The time for parasite death was recorded and photos taken at different times from start of treatment until complete death of the parasites for comparative
evaluation of their acaricidal effect.

Results and Discussion

XRD results of MANPs and Ag NPs showed the formation of MANPs and Ag NPs without any shifting of its 20 peaks position or presence of peaks of another chemicals used through synthesis process which indicated the purity of MANPs and Ag NPs (Fig. 1). The XRD results gave information about crystallinity and crystal form of the materials. The XRD curve of Ag NPs showed best amorphous state without any additional peaks indicated the homogeny of chemical composition (there are no other peaks of chemicals that used in synthesis process). XRD curve of MANPs (Fig. 1) illustrated the best fit 20 peaks position with cubic crystal form for magnetite nanoparticles according to ICCD data base (COD1011032 Fe₃O₄ Magnetite). Elemental analysis using Eva software from Bruker Company using XRD chart showed the composition of MANPs as 27.6% oxygen and 72.4% iron. XRD measure for Silver-essential oil nanocomposite and magnetite-essential oil nanocomposite was carried out to identify if there is any change in chemical composition of silver and magnetite nanoparticles by nanocomposite synthesis. XRD pattern of both silver-essential oil and magnetite-essential oil nanocomposites as shown in Figs. 2 and 3 illustrated unchanged in crystalline or chemical composition of both silver and magnetite nanoparticles by nanocomposite synthesis with essential oils.

XRF charts illustrated the purity of MANPs and Ag NPs (Fig. 4 and 5) with percentage of Ag and Fe more than 97%. The purity allowed evaluating their bioactivity without any influence from any factors.

Raman spectrum of Ag NPs (Fig 6) showed very sharp peak at 56.02 cm⁻¹ and weak one at 146.41 cm⁻¹. Raman spectrum of MANPs showed very sharp peak at 670 cm⁻¹. TEM image showed the mono dispersion of MANPs.
particles with size about 20 nm and shape of spherical to semi-cubic (Fig. 7). TEM image of Ag NPs showed the best spherical shape with homogeneity size about 5 nm (Fig. 8).

Fig. 6 Raman spectra of MANPs and Ag NPs.

Fig. 7 TEM image of MANPs.

Fig. 8 TEM image of Ag NPs.

Fig. 9 pointed out the 2D and 3D AFM images regarding the shape, size, homogeneity of particles distribution and roughness profile of MANPs and Ag NPs, respectively. Thin films of MANPs and Ag NPs were prepared in order to give complete ability study under AFM was done by sonication the MANPs and Ag NPs suspension in deionized water for 1h at condition of 0.1 cycle and 15% amplitude then using Spain coater which make thin films on glass.

Fig. 9 (a) 2D AFM image for MANPs (top view 500 × 500 nm); (b) 3D AFM image for MANPs (500 × 500 nm); (c) 2D AFM image for Ag NPs (top view 500 × 500 nm); and (d) 3D AFM image for Ag NPs (500 × 500 nm).
slid substrate. All images were of the size 500 X 500 nm using contact mode with speed of 0.1 in/sec and servo condition of I gain 0.5, P gain 2.5 and set point zero. 2D and 3D AFM images of Ag NPs and MANPs illustrated the spherical shape with well sorting without any agglomeration and homogenous in size distribution (2-7 nm for Ag NPs and 15-22 nm for MANPs) and shape. 2D and 3D AFM images of MANPs-essential oil and Ag NPs-essential oil nanocomposites illustrated the effect of oils in aggregation and size of silver and magnetite nanoparticles. However, 2D and 3D AFM images (Fig. 10 and 11) illustrated the homogenous of size distribution become bad sorting due to coating of oils to silver and magnetite nanoparticles formed different sizes of them but still aggregate in nano-size.

DLS results as shown in Fig. 12 illustrated the size of nanoparticles effect by nanocomposite formation due to coating of essential oils to nanoparticles lead to agglomeration or concentration of nanoparticles. However, nanoparticles size was 5.3 nm and 21.7 nm for silver and magnetite nanoparticles, respectively. Nanocomposite size was 20.5 nm and 40 nm for Ag NPs-oil nanocomposite and magnetite-oil nanocomposite, respectively. The results of DLS were in good agreement with AFM results.

Remarkable results illustrated by measure zeta potential of nanoparticles and nanocomposites where the zeta potential of Ag NPs and MANPs desperation in distilled water was −30 and −17 mV where Ag NPs-oils nanocomposite and MANPs-oils nanocomposite (nanoparticles desperation on oils) was −55 and −28 mV. Increased value of zeta potential of nanocomposite formed allowed them to increased its stability in oils and more desperation in oils than water and also increase surface charge. However, increased zeta potential value enhancement the killing activity of nanomaterials.

Vibrating sample magnetometer (VSM) was carried out to illustrate the magnetic properties of MANPs and MANPs-essential oil nanocomposite and explain if there are changes in magnetic properties of nanoparticles after nanocomposite synthesis. VSM was measured at room temperature as shown in Fig. 13 and 14. Coercivity (Hci), magnetization (Ms) and retentivity (Mr) were 58.543 G, 39.370 emu/g and 2.1261 emu/g for MANPs, respectively, and 51.241 G, 32.11 emu/g and 2.01 emu/g for MANPs-oil nanocomposite, respectively. Both of them have

Fig. 10 (a) 3D AFM image for Ag NPs-essential oil (500 × 500 nm); (b) top view AFM image for Ag NPS-essential oil (500 × 500 nm).

Fig. 11  (a) 3D AFM image for MANPs-essential oils (500 × 500 nm); (b) top view AFM image for MANPS-essential oils (500 × 500 nm).
S-shape which indicated the super para magnetic nature of them. However, nanocomposite synthesis does not effect on magnetic properties which explain why MANPs and MANPs-oil nanocomposites has almost the same killing activity.

Mites were directly subjected to different treatments and examined under stereomicroscope until complete stop movements and their features were recorded at 1 and 2 h after treatments (Fig. 15). The non-treated mites showed active movement under stereomicroscope with pale light brown color at 1 and 2 h after treatment. The sprayed mites with 0.4% solution of essential oil were completely stopped movements which considered dead within 1 and 2 h after treatment with completely stretched legs and white bead like spots of oils accumulation on legs and bodies. Mites treated with MANPs (100 ppm) stopped movements after 2 h treatment and showed bent legs under their bodies with dark brown color on their bodies. Mites treated with Ag NPs (75 ppm) were directly stopped movement after treatments for 1 and 2 h with detached legs and white coloration and pale hit...
film surround the mites. Mites treated with magnetite-
essential oil nanocomposite showed stop movement,
detached legs and abnormal keratin surface of dark
brown color after 2 h treatment. While, mites treated
with magnetite-essential oils nanocomposite suddenly
stop movements after spraying and showed complete
distraction and detachment of legs and distracted body
keratin with white coloration.

Results of essential oil, Ag NPs, MANPs, magnetite-
essential oil nanocomposite and silver-essential oil
nanocomposite sprayed on *D. gallinae* at with different
doses were listed in Table 1. For the individual
0.4% essential oil, Ag NPs and silver-essential oil
nanocomposite after 1, 2 and 3 h of contact showed
strong acaricide effect of 100% red mites’ death at 1, 2
and 3 h. While, magnetite-essential oil nanocomposite
showed 70.33 ± 0.47% at 1 hour and 100% at 2 and
3 h contact. MANPs (75 ppm) alone showed slower
acaricide effect started with 40.66 ± 0.49% at 1 h
followed by 100% at 2 and 3 h contact.

XRD, XRF and Raman spectra results of MANPs
and Ag NPs confirmed their synthesis without any
contamination or undesired chemicals during synthesis
methods, so the silver and magnetite nanoparticles
used individual or in synthesis of nanocomposite with
essential oil were pure. TEM, SEM and AFM images
showed the mono desperation of MANPs particles size
about 20 nm with spherical to semi-cubic shape and
Ag NPs showed spherical shape with homogeneity size
about 5 nm. The sharp peak at 56.02 cm⁻¹ and weak one
at 146.41 cm⁻¹ found in the Raman spectrum of Ag NPs
were characteristic to Ag lattice vibrational mode while
other week peaks for oxygen bond due to high intensity
of laser (ND filter 50%) may be led to formation of
silver oxide. Raman spectrum of MANPs was carried
out using acquisition time 7 sec, accumulation 1
without spike filter and delay time. Objective was
X100 with grating 1800 (450-850 nm), ND filter 5%
and the laser type was green of 532 nm. It was obvious
from the MANPs Raman chart that the peak at 670
cm⁻¹ was characteristic to magnetite lattice vibrational
mode. TEM image showed the mono desperation of
MANPs particle size about 20 nm and TEM image of

---

**Table 1** Acaricidal effect of different materials against *D. gallinae* collected from layers farms (average of 3 replicates)

| Group | Treatment                          | Dose          | Mites mortality (%) |
|-------|------------------------------------|---------------|---------------------|
|       |                                    |               | 1 h       | 2 h       | 3 h       |
| 1     | Non treated                        | Saline        | 0        | 0        | 0        |
| 2     | Oils                               | 0.4%          | 100     | 100     | 100     |
| 3     | MANPs                              | 100 ppm/mL   | 40.66 ± 0.49 | 100     | 100     |
| 4     | Ag NPs                             | 75 ppm/mL    | 100     | 100     | 100     |
| 6     | Magnetite-essential oils nanocomposite | 1 mL        | 70.33 ± 0.47 | 100     | 100     |
| 6     | Silver-essential oils nanocomposite | 1 mL        | 100     | 100     | 100     |
Ag NPs showed spherical shape with homogeneity size about 5 nm. The 2D and 3D images of Ag NPs illustrated the spherical shape, while MANPs particles were of spherical to semi-cubic in shape.

*D. gallinae* or the poultry red mite is a cosmopolitan hematophagous mite, parasitic on birds. *D. gallinae* life cycle possessed five stages (egg, larva, protonymph, deutonymph and adult). Infestation can triple its numbers in only 10 days where nymphs need a blood meal for metamorphosis and adult females need blood meals for egg maturation. The mites are small and grey in color, but may appear if they have filled with blood after feeding on the bird. The susceptibility of recent field collected *D. gallinae* from naturally infested commercial chicken houses to essential oil mixture and/or prepared nanoparticles as acaricide was evaluated. Normal color is grey in color and appears red after feeding on the bird. The outer part of the mite exoskeleton, known as the epicuticle, consists of a layer of wax, which further limits water loss, and a cement layer, which protects the cuticle from external abrasion [32].

The susceptibility of collected *D. gallinae* to essential oil, MANPs, Ag NPs, magnetite-essential oil nanocomposite and silver-essential oil nanocomposite as acaricide was carried out by direct spray to fulfill contact toxicity [33]. The non-treated mites showed active movement under stereomicroscope with pale grey color at 1 and 2 h. The mites are grey in color, but protonymph, deutonymph and adult appeared red after feeding on the bird. The mites sprayed with 0.4% oil solution were completely stopped movements and considered dead after 1 and 2 h contact with completely stretched legs with white bead like spots of oils accumulation on legs and bodies. The used essential oil mixture (*Allisal®*) contains garlic oil, rosehip oil and rapeseed oil. Researchers [34-36] stated that plant-derived essential oils are shown to have a lethal characteristic, where garlic and thyme oils were the most effective. In the other side, the synergistic action of essential oil for cuticular penetration was reported [37, 38]. Mites treated with MANPs of 75 ppm concentration as spray were stopped movements after 2 h and showed bent legs under their bodies with dark brown color. Silver nanoparticles have a significant impact on insect [39]. Also, Ag NPs were resulted in cuticular depenalization in Drosophila. The dark brown spot can be attributed to accumulation of Ag NPs as they were apo plastically transported in the cell wall and found aggregated at plasmodesmata [40] or can die due to Ag NPs induced oxidative stress as indicated by histochemical staining of superoxide radical and hydrogen peroxide that was manifested in terms of DNA degradation and cell death [41]. Mites treated with 75 ppm Ag NPs directly stopped movement (5 sec) and lasted at 1 and 2 h with detached legs and white coloration and pale hit film surround the mites, while those treated with magnetite-essential oils nanocomposite showed stop movement and detached
legs and abnormal keratin surface of dark brown color at 2 h. While, mites treated with silver-essential oils nanocomposite suddenly (2 sec) stopped movements after spraying and showed complete distraction and detachment of legs and distracted body keratin with white coloration. This result proved the synergistic acaricial action of essential oil and nanoparticles. Regarding the acaricial effect measured by percent of *D. gallinae* deaths in relation to time of exposure. The 0.4% essential oils solution (garlic oil, rosehip oil and rape seed) showed strong acaricide effect with 100% deaths at 1, 2 and 3 h. This result was previously reported by many researchers. Carvacrol (essential oil of Origanum vulgare), garlic extract, cinnamon, eucalyptus and mint extract reduction in mite control with 92%, 96%, 66.97, 80.85 and 90.19%; respectively. Essential oils were tested for the movable *D. gallinae* acaricial activity regardless of the stage of their development. The results were 80% rosemary oil (>90% mortality), 50% lavender (75% mortality), 100% thyme (80% mortality) and 80% clove oil (73% mortality) as measured after 48h [42]. Garlic extract was 96% effective after two successive sprays. Acaricial activity of plant bioactive components was assessed against *D. gallinae* by contact toxicity carvacrol and thymol were found to be toxic to *D. gallinae* with LD₅₀ values of 1 and 3.15 μg/cm, respectively [42]. Combination of carvacrol-thymol in 4:1 ratio at 2% concentration displayed good residual toxicity and was effective against *D. gallinae* till 14 days post spraying and a synergistic effect can be suggested for the control of *D. gallinae* [43]. The main chemical constituent of intact garlic is the amino acid allicin, an alkyl derivative of cysteine alkyl sulfoxide, which may vary from 0.2 to 2.0% fresh weight [44-47]. Garlic contains at least 100 sulfur-containing compounds basic to medicinal uses. Allicin represents 70-80 % of the total thiosulfates [47-50]. Garlic essential oil was toxic to *T. molitor* larva, followed by pupa and adult. Diallyl disulfide was the most toxic than diallyl sulfide for pupa > larva > adult, respectively, and showing lethal effects at different time points. Garlic essential oil and their compounds have the potential for pest control [51]. The rosehip seed contained valuable phytochemicals such as phenolic compounds (2554 μg/g), carotenoids (2.92 μg/g), and ascorbic acid (1798 μg/g). Furthermore, the rosehip-seed oil was rich in polyunsaturated fatty acids, linoleic acid (54.05%), linolenic acid (19.37%), and phytosterols, mainly β-sitosterol (82.1%) [52]. Comparing the toxicity of the plant oils, it was affirmed that rose volatile oils was efficient natural phyto compounds against the treated larvae of cotton leaf-worm and combination of oils has synergistic action against the 4th larval instar of *S. littoralis* [53]. Rapeseed oil is plant-based oil extracted from the seeds of the rapeseed plants contained higher levels of total tocopherols and carotenoids. It is often added to animal feeds, when used in spray treatment against spider mites, green peach aphid and pear psylla resulted in efficacy rate 97.4% (7 days after treatment) against the summer population of *P. ulmi*, efficacy was 84.1% and 94.6%, 11 days after treatment against *T. urticae* and against *M. persicae* on pepper revealed high (> 96%, 7-14 days) efficacy of as and in a greenhouse achieved 86.9% efficacy after 8 days [54]. The synergistic action of essential oils mixture was also reported in antimicrobial activity [55-57]. Ag NPs (75 ppm) showed rapid 100% death at the 1st hour and no movement was observed till the 3rd hour. The result indicated highly and rabid effect of Ag NPs under the used concentration and particle size. Silver nanoparticles have a significant impact on insect antioxidant and detoxifying enzymes, leading to oxidative stress and cell death [58]. Ag nanoparticles also reduced acetylcholinesterase activity, while polystyrene nanoparticles inhibited CYP450 isoenzymes. Metal nanoparticles can bind to S and P in proteins and nucleic acids, respectively, leading to a decrease in membrane permeability. In the other hand, Quantitative proteomics studies on human colon cancer cell lines indicated that some cellular responses triggered by Ag NPs are driven by the size of NPs. The 100 nm NPs exerted indirect effects via serine/threonine kinase (PAK), mitogen-activated protein kinase (MAPK), and phosphatase 2A pathways, while the 20 nm NPs induced indirect effects on cellular stress, including generation of ROS, protein carbonylation and up-regulation of proteins [59]. The *Origanum vulgare* essential oil and silver nanoparticles have potent antibacterial activity [60]. MANPs (100 ppm) had slower acaricide effect starts with 40.66 ± 0.49% at 1 h followed by 100% at 2 and 3 h. The slow killing activity of MANPs can be attributed to its very low toxicity which considered very safe environmental nanoparticles. The tested silver-essential oils nanocomposite showed acaricide effect 100% after 1, 2 and 3 h contact. This is the same obtained result from each compound alone. While, magnetite-essential oils nanocomposite showed 70.33 ± 0.47% at 1 hour and 100% at 2 and 3 h so MANPs reduced the activity of essential oil.
Conclusions

Silver nanoparticles, magnetite nanoparticles, silver-essential oil nanocomposite and magnetite-essential oil nanocomposite were synthesized and their morphological, identification and index properties were characterized. According to killing time and effect on D. gallinae bodies, the highest killing activity was shown by silver nanoparticles, followed by silver-essential oil nanocomposites, essential oil, magnetite nanoparticles, and lastly magnetite-essential oil nanocomposites. Apart from that, silver-essential oil nanocomposites and silver nanoparticles presented 100% killing activity after a few seconds, while essential oil, magnetite-essential oil nanocomposites and silver nanoparticles showed 100% killing activity after 1 to 2 h. If not considering the time of killing, magnetite-essential oil nanocomposites were more recommended to be used than silver-essential oil nanocomposites and silver nanoparticles due to their very low toxicity. However, if taking time into account, silver-essential oil nanocomposite and silver nanoparticles could be beneficial and involved in D. gallinae (red mite) control strategies in poultry industry.

Acknowledgments

Synthesis, characterization and observed killing activity of nanoparticles and nanocomposites carried out in Egyptian Nanotechnology Center-Cairo university (EGNC).

Funding

The authors declare that the work was self-funded.

Authors’ contributions

Aziza M. Amer and Mohamed M. Amer designed, planned the study samples and all laboratory tests. Sameh H. Ismail and Gehad G. Mohamed synthesized and characterized nanoparticles and nanocomposite. All authors shared performed experimental work, manuscript writing, drafted, revised the manuscript and approved the final manuscript.

Conflict of Interests

The authors have no conflict of interests to declare regarding the publication of this paper.

References

[1] A.S. Flochlay, E. Thomas, and O. Sparagano. Poultry red mite (Dermanyssuss gallinae) infestation: A broad impact parasitological disease that still remains a significant challenge for the egg-laying industry in Europe. Parasites & Vectors, 2017, 10 (1): 357.
[2] B. Meyer-Kuhling, K. Pfister, J. Muller-Lindloff, et al., Field efficacy of phoxim 50% (ByeMite) against the poultry red mite Dermanyssuss gallinae in battery cages stocked with laying hens. Vet. Parasitol. 2007, 147: 289-296.
[3] L. Roy, C. Chauve, J. Delaporte, et al., Exploration of the susceptibility of AChE from the poultry red mite Dermanyssuss gallinae (Acari: Mesostigmata) to organophosphates in field isolates from France. Exp. Appl. Acarol. 2009, 48: 19-30.
[4] O. Sparagano, A. Pavličević, T. Murano, et al., Prevalence and key figures for the poultry red mite Dermanyssuss gallinae infections in poultry farm systems. Exp. Appl. Acarol., 2009, 48: 3-10.
[5] J. Chirico, R. Tauson. Traps containing acaricides for the control of Dermanyssuss gallinae. Vet. Parasitol., 2002, 110: 109-116.
[6] M.C. Valiente, J. Thioulouse, C. Chauve, et al., Bacterial taxa associated with the hematophagous mite Dermanyssuss gallinae detected by 16S rRNA PCR amplification and TTGE fingerprinting. Res. Microbiol., 2009, 160: 63-70.
[7] L.C. Cernea, E. Suteu, M. Cernea, et al., Realization of an experimental model for in vitro testing of the acaricidal effect of the vegetal extracts [in Romanian. Rev. Sci. Parasitol., 2006, 7: 35-40.
[8] S.I. Kim, Y.E. Na, J.H. Yi, et al., Contact and fumigant toxicity of oriental medicinal plant extracts against Dermanyssuss gallinae (Acari: Dermanyssidae). Vet. Parasitol., 2007, 145: 377-382.
[9] S. Lee, R. Tsao, C. Peterson, et al., Insecticidal activity of monoterpenoids to Western corn rootworm (Coleoptera: Chrysomelidae), twospotted spider mite (Acari: Tetranychidae) and house fly (Diptera: Muscidae). J. Econ. Entomol., 1997, 90: 883-892.
[10] R.Z. Abbas, D.D. Colwell, Z. Iqbal, et al., Acaricidal drug resistance in poultry red mite (Dermanyssuss gallinae) and approaches to its management. World’s Poul. Sci. J., 2014, 70: 113-124.
[11] S.I. Kim, J.H. Yi, J.H. Tak, et al., Acaricidal activity of plant essential oils against Dermanyssuss gallinae(Acari: Dermanyssidae). Vet. Parasitol., 2004, 120: 297-304.
[12] M. Magdaş, M. Cernea, H. Baciu, et al., Acaricidal effect of eight essential oils against the poultry red mite Dermanyssuss gallinae (Acari: Dermanyssidae). Sci. Parasitol., 2010, 11(2): 71-75.
[13] J. Lundh, D. Wiktelius, and J. Chirico, Azadirachtin-impregnated traps for the control of Dermanyssuss gallinae. Vet.Parasitol., 2005, 130: 337-342.
[14] V. Maurer, E. Perler, and F. Heckendorn. In vitro efficacies of oils, silicas and plant preparations against the poultry red mite Dermanyssuss gallinae. Exp. Appl. Acarol., 2009, 48: 31-41.
[15] D.R. George, D. Masic, O.A. Sparagano, Variation in chemical composition and acaricidal activity against Dermanyssuss gallinae of four eucalyptus essential oils. Exp. Appl. Acarol., 2009, 48: 43-50.
[16] K. Yamauchi, N. Manabe, Y. Matsumoto, et al., Exterminating Effect of Wood Vinegar to Red Mites and its Safety to Chickens. J. Poult. Sci., 2015, 51: 327-332.
[17] V.K. Singh, D.K. Singh, Pharmacological Effects of Garlic (Allium sativum L.). ARBS Annu. Rev. Biomed
Nano Biomed. Eng., 2020, Vol. 12, Iss. 4

http://www.nanobe.org

[18] A. Bhattacharyya, A. Bhaumik, P.U. Rani, et al., Nanoparticles - A recent approach to insect pest control. Afr. J. of Biotechnol., 2010, 9: 3489-3493.

[19] C.R. Chinnamuthu, P. Murugesu Boopathi, Nanotechnology and Agroecosystem. Madras Agri. J., 2009, 96(1-6): 17-31.

[20] O.F. Owolade, D.O. Ogunleti, and M.O. Adenekan, Titanium Dioxide affects disease development and yield of edible cowpea. Electronic J. of Env. Agri. and Food Chem., 2008, 7(50): 2942-2947.

[21] M. Rouhani, M.A. Samih, and S. Kalantari, Insecticidal effect of silica and silver nanoparticles on the cowpea seed beetle, Callosobruchus maculatus F. (Col.: Bruchidae). J. of Entomological Res., 2012, 4(4): 297-305.

[22] U. Suresh, K. Murugan, C. Panneerselvam, et al., Suedamaritimata-based herbal coills and green nanoparticles as potential biopesticides against the dengue vector Aedes aegypti and the tobacco cutworm Spodoptera luristica. Physiol. Mol. Plant Pathol., 2018, 101: 225-235.

[23] C. Völker, M. Oetken, and J. Oehlmann, The Biological Effects and Possible Modes of Action of Nanosilver. Reviews of environmental contamination and toxicology, volume 223. Springer, 2013: 81-106.

[24] N. Armstrong, M. Ramamoorthy, D. Lyon, et al., Mechanism of Silver Nanoparticles Action on Insect Pigmentation Reveals Intervention of Copper Homeostasis. PLoS ONE, 2013, 8(1): e53186.

[25] A. Jalalizand, S. Gavanji, J.K. Esfahani, et al., The effect of Silver nanoparticles on Tetranychus urticae. Intl. J. Agri. Crop Sci., 2013, 5 (8): 820-827.

[26] Y.T. Prabhu, K.V. Rao, B.S. Kumari, et al., Synthesis of FeO2 nanoparticles and its antibacterial application. Int Nano Lett, 2015, 5: 85.

[27] S. Sajjad, A. Nasseri, Synthesis and stabilization of Ag nanoparticles on a polyamide surface and its antibacterial effects. Int Nano Lett, 2011, 1: 22.

[28] J. Tang, M. Myers, K.A. Bosnick, Magnetite FeO2 nanocrystals: Spectroscopic observation of aqueous oxidation kinetics. J. Phys. Chem. B, 2003, 107: 7501.

[29] F. Bosi, U. Halenius, and H. Skogby, Crystal chemistry of the magnetite-ulvospinel series Locality: synthetic Note: Mgt100. American Mineralogist, 2009, 94: 181-189.

[30] J. Pritchard, T. Kuster, O. Sparagano, et al., Understanding the biology and control of the poultry red mite Dermanyssus gallinae: A revie. Avian Pathology, 2015, 44(3): 143-153.

[31] J. Zdybel, J. Karamon, and T. Cencek, In vitro effectiveness of selected acaricides against red poultry mites (Dermanyssus gallinae, De Geer, 1778) isolated from laying hen battery cage farms localised in different regions of Poland. Bull. Vet. Inst. Pulawy., 2011, 55: 411-416.

[32] A. Bhattacharyya, A. Bhaumik, P.U. Rani, et al., Nanoparticles - A recent approach to insect pest control. Afr. J. of Biotechnol., 2010, 9: 3489-3493.

[33] D.R. George, G. Olautunji, J.H. Guy, et al., Effect of plant essential oils as acaricides against the poultry red mite, Dermanyssus gallinae, with special focus on exposure time. Vet. Parasitol., 2010, 169: 222-225.

[34] D.R. George, O.A.E. Sparagano, G. Port, et al., Environmental interactions with the toxicity of plant essential oils to the poultry red mite Dermanyssus gallinae. Med. and Vet. Entomol., 2010, 24: 1-8.

[35] D.R. George, B.D. Finn, P.M. Graham, et al., Present and future potential of plant-derived products to control arthropods of veterinary and medical significance. Parasit Vectors, 2014, 7: 28.

[36] F. Tong, J.R. Bloomquist, Plant essential oils affect the toxicities of carbaryl and permethrin against Aedes aegypti (Diptera: Culicidae). J. Med. Entomol., 2013, 50: 826-832.

[37] G. Benelli, Mode of action of nanoparticles against insects. Environ. Sci. Pollut. Res., 2018, 25: 12329.

[38] J. Geisler-Lee, Q. Wang, Y. Yao, et al., Phytotoxicity, accumulation and transport of silver nanoparticles by Arabidopsis thaliana. Nanotoxicol, 2013, 7(3): 323-337.

[39] K. Vishwakarma, K. Shweta, N. Upadhyay, et al., Differential Phytoxic Impact of Plant Mediated Silver Nanoparticles (AgNPs) and Silver Nitrate (AgNO3) on Brassica sp., Front Plant Sci., 2017, 8: 1501.

[40] M. Roczeń-Kaczmarek, M. Demkowska-Kutrzepa, J. Zdybel, et al., In vitro effect of plant essential oils as acaricides against Dermanyssus gallinae. Proceedings of the 3rd Cost Conference. Oeiras, Portugal, Sep. 20-21, 2017.

[41] M.A. Tabari, M.R. Youssefi, A. Barimani, et al., Carvacrol as a potent natural acaricide against Dermanyssus gallinae. Parasitol. Res., 2015, 114(10): 3801-3806.

[42] F. Masoumi, M.R. Youssefi, and M.A. Tabari, Combination of carvacrol and thymol against the poultry red mite (Dermanyssus gallinae). Parasitol. Res., 2016, 115(11): 4239-4243.

[43] E. Michailles. Über Neue wirkstoffeausknoblauch (Allium sativum) und kuchenzwiebel (Allium cepa L.) (dissertation). München: Ludwig-Maximilians Univeristät, 1974.

[44] J. Lutomski, Inhalts-u. wirksstoffeiniger Allium Arten. Report at Allium Conf. Mendel-Frimmel Gesellschaft für Angewandte Genetic e.v.c - Freising (FRG), 1983: 19-23.

[45] J. Lutomski, Das wichfigste uber knoblauch und knoblan-ch-praparate. Dtsch. Apoth. Ztg, 1983, 123: 623-624.

[46] J. Lutomski, Components and biological properties of some Allium species. Poznan: Institute of the Medicinal Plants, 1987: 1-58.

[47] L.D. Lawson, B.G. Hughes, Characterization of the formation of allicin and other thiosulfonates from garlic. Planta Med., 1992, 58: 345-350.

[48] L.D. Lawson, Bioactive organosulfur compound of garlic and garlic products: role in reducing blood lipids. Human medicinal agents from plants. American Chemical Society, ACS Symposium Series, 1993, 534: 306-330.

[49] K.C. Srivastava, A. Bordia, and S.K. Verma, Garlic (Allium sativum) for disease prevention. South Afr. J. Sci., 1995, 91: 68-77.

[50] A. Plata-Rueda, L.C. Martinez, M.H. Dos Santos, et al., Insecticidal activity of garlic essential oil and their constituents against the mealworm beetle, Tenebrio molitor Linnaeus (Coleoptera: Tenebrionidae). Sci. Rep., 2017, 7: 46406.

[51] G. Benelli, Mode of action of nanoparticles against insects. Environ. Sci. Pollut. Res. 2018, 25: 12329.

[52] H. Ilyasoğlu. 2014. Characterization of Rosehip (Rosa canina L.) Seed and Seed Oil. Int. J. of Food Properties, 2014, 17(7): 1591-1598.

[53] H.A. Mesbah, A.K. Mourad, and A.Z. Rokaia, Efficacy of some plant oils alone and/or combined with different insecticides on the cotton leaf-worm Spodopteralitoralis (Boisd.) (Lepidoptera: Noctuidae) in Egypt. Commun Agric Appi Biol Sci, 2006, 71(2) Pt B: 305-328.

[54] D. Marčić, P. Perić, M. Prijović, et al., Insecticidal activity of garlic essential oil and their constituents against the mealworm beetle, Tenebrio molitor Linnaeus (Coleoptera: Tenebrionidae). Sci. Rep., 2017, 7: 46406.

[55] I.H.N. Bassolé, H.R. Juliani, Essential oils in combination with insecticides. Environ. Sci. Pollut. Res. 2018, 25: 12329.
[56] M. Hyldgaard, T. Mygind, and R.L. Meyer, Essential oils in food preservation: mode of action, synergies, and interactions with food matrix components. Front. Microbiol, 2012, 3: 12.

[57] T. Verano-Braga, R. Miethling-Graff, K. Wojdyla, et al., Insights into the cellular response triggered by silver nanoparticles using quantitative proteomics. ACS Nano, 2014, 8: 2161-2175.

[58] S. Scandorieiro, L.C. de Camargo, C.A.C. Lancheros, et al., Synergistic and Additive Effect of Oregano Essential Oil and Biological Silver Nanoparticles against Multidrug-Resistant Bacterial Strains. Front Microbiol., 2016, 7: 760.

Copyright © Sameh Ismail, Gehad Mohamed, Aziza Amer, and Mohamed Amer. This is an open-access article distributed under the terms of the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original author and source are credited.