



APHIDICIDAL POTENTIAL OF GREEN SYNTHESIZED MAGNESIUM HYDROXIDE NANOPARTICLES USING *Olea europaea* LEAVES EXTRACT

Alaa Y. Ghidan¹, Tawfiq M. Al-Antary¹, Akl M. Awwad² and Muhand W. Akash³

¹Department of Plant Protection, School of Agriculture, the University of Jordan, Amman, Jordan

²Department of Materials Science and Nanotechnology, Royal Scientific Society, Amman, Jordan

³Department of Horticulture and Crop Science, College of Agriculture, the University of Jordan, Amman, Jordan

E-Mail: akl.awwad@yahoo.com

ABSTRACT

Magnesium hydroxide (MgHNPs) nanoparticles were synthesized using *Olea europaea* aqueous extract at ambient temperature and in one-single step. X-ray diffraction (XRD) analysis showed that synthesized MgHNPs have spherical shape with an average size diameter 40nm. Fourier transform infrared (FT-IR), scanning electron microscopy (SEM) and UV-vis spectrophotometer were used to characterize the synthesized magnesium hydroxide nanoparticles. This study determined the mortality efficacy of different concentrations of the synthesized MgHNPs against early and late nymphal instars of the green peach aphid. There were significant differences in the aphid mortalities between the different concentrations of the MgHNPs nanoparticles. In addition, the differences between the different concentrations and the control were significantly obvious.

Keywords: green synthesis, magnesium hydroxide nanoparticles, *Olea europaea* leaf extract, mortality, green peach aphid.

INTRODUCTION

The green peach aphid (GPA) *Myzus persicae* (Sulzar) (Homoptera: Aphididae) is considered as a key peston peach and as a primary host of this tree (Emanuele and Piero, 2002). It is considered as a globally important pest of a broad range of arable and horticultural crops, including Jordan (Al-Antary and Khader, 2013). This aphid is able to transmit more than 100 plant viruses (Blackman and Easto, 2000). This global pests responsible of several important economic losses on more than 50 plant families causing losses to agro industrial crops, including potato, sugar beet and tobacco, horticulture crops, including plants of Brassicaceae, Solanaceae and Cucurbitaceae families and stone fruits including, peach, apricot and cherry. Insecticide resistance is one of the best examples of micro-evolution, or evolution occurring on an ecological time scale. The study of insecticide resistance is important, both because it leads to a better understanding of evolutionary mechanisms operating in real time, and because of its economic relevance.

Long-term reliance on synthetic chemical insecticides for aphid control has resulted in high resistance among many aphid species and environmental problems. The incidences of increasing insect resistance to the chemical insecticides shortens their longevity, facilitating a search a search for potential green methods that can be exploited for insect pest control. Recently, green peach aphid control was evaluated by using alkalization of bamboo tar (Tong and Feng, 2016); Piperonylbutoxide (Panini *et al.*, 2017); bornyl acetate (Buchoiz *et al.*, 2017); effect of nitrogen fertilizers (Rousselin *et al.*, 2016); mineral oil (Samara *et al.*, 2016); (E)- β -farnesene (Qin *et al.*, 2016); Aphelinusasychis (Wang *et al.*, 2016); protein extracted from bulbs of *Phycellaaustralis* Ravenna (Zapata *et al.*, 2016); and plant aqueous extracts of fruits, leaves and flowers (Birgücü *et*

al., 2015; Madanat *et al.*, 2016; Erdoğan & Yıldırım, 2016). Thereby the new approach is not only green, economic, eco-friendly to environment and non-toxic by-products, but also easy to scale up. This is the first work on synthesis of magnesium hydroxide nanoparticles using an eco-friendly, non-toxic aqueous extract of *Olea europaea* leaves at ambient temperature and one single step reaction to evaluate its effect on the green peach aphid.

Magnesium hydroxide [Mg(OH)₂] is important inorganic materials with many applications such as adsorbent, fire retardant, catalyst support, advanced ceramics, toxic waste remediation, photo electronic materials, antacid excipient in pharmaceuticals and refractory materials. Therefore various techniques and routes for synthesis magnesium hydroxide and oxide nanoparticles have been reported in the literature such as hydrothermal route (Dhaouadi *et al.*, 2011; Yan *et al.*, 2008; Hanlon *et al.*, 2015); water-in-oil microemulsion (Wu *et al.*, 2008); ultrasound-assisted method (Baidukova & Skorb, 2016), batch reaction crystallization (Song *et al.*, 2011); precipitation and co-precipitation processes (Zhou *et al.*, 2014; Wang *et al.*, 2011; Pilarska *et al.*, 2013); Chen *et al.*, 2009; Jiang *et al.*, 2009; Xu *et al.*, 2006); microwave reaction (Yousefi, 2014), via a facile template-free solvothermal route (Jinghui *et al.*, 2009); stream-rotating packed bed reactor (Shen & Liu, 2016); and spinning desk reactor (Tai *et al.*, 2007). These approaches have many disadvantages due to the difficulty of scale up the process, separation and purification of nanoparticles from the oil, surfactant, and organic solvents. Developing green methods for synthesizing magnesium hydroxide and oxide nanoparticles are of importance and still a challenge for materials researchers. If the synthesis and production cost of magnesium hydroxide and oxide nanoparticles could be decreased greatly through using very cheap raw non-toxic



materials, ambient temperature, large scale preparation, the practical application of magnesium hydroxide and oxide nanoparticles would be accessible. Magnesium hydroxide were synthesized by green methods using non-toxic and eco-friendly such as *Neem* leaves extract (Moorthy *et al.*, 2015); *Citrus limon* leaves extract (Awwad & Ahmad, 2014); *acacia* gum (Srivastava *et al.*, 2015); *Brassica oleracea* and *Punicagranatum* peels (Sugirtha *et al.*, 2015); and *Clitoriaternatea* (Sushma *et al.*, 2016). In this research work *Olea europaea* leaves aqueous extract was used to synthesis magnesium hydroxide nanoparticles and to study their effects on the green peach aphid.

MATERIALS AND METHODS

Anhydrous magnesium sulfate ($MgSO_4$) and potassium hydroxide (KOH) were obtained from Aldrich and used as received without further purifications. De-ionized distilled water was used in all experimental work. Freshly *Olea europaea* leaves were collected from different fields in Jordan and washed several times with distilled water to remove the dust particles and then dried in shade to remove the residual moisture. The leaves extract was prepared by placing 20 g of dried powder in 500 mL glass beaker along with 400 mL of sterile distilled water. The mixture was then boiled for 10 minutes, the colour of the aqueous solution changed to yellow. Afterwards, the extract was cooled to room temperature and filtered by Whatman paper No. 1 before centrifuging at 1200 rpm for 5 minutes to remove the heavy biomaterials. The extract was stored at room temperature in order to be used for the experimental work. In a typical reaction mixture, 1.2g of magnesium sulfate, $MgSO_4$ was dissolved in 200mL of aqueous solution of *O. europaea* leaves under stirring at room temperature for 10 min to achieve brown solution. Then potassium hydroxide solution (0.5g/100mL) was added drop by drop to the mixture to adjust the pH = 12-14, yellow suspended particles appeared, indicating the formation of magnesium hydroxide nanoparticles. The suspended particles were purified by dispersing in sterile distilled water and centrifugation three times. Afterwards, yellowish particles of magnesium hydroxide were dried at 80°C. The above procedure was repeated by using different amounts of *O. europaea* leaves extract and magnesium ions concentration at room temperature.

Magnesium hydroxide and oxide nanoparticles synthesized by this green method were characterized by X-ray diffractometer, (XRD-6000, Shimadzu) equipped with $CuK\alpha$ radiation source ($\lambda = 0.154056$ nm) using Ni as filter at a setting of 30 kV/30mA. All XRD data were collected under the experimental conditions in the angular range $3^\circ \leq 2\theta \leq 80^\circ$. FT-IR spectra of *O. europaea* leaves extract, synthesized MgHNPs were obtained in the range 4000-400 cm^{-1} with IR-Prestige 21 spectrophotometer (Shimadzu) using KBr pellet method. Scanning electron microscopy (SEM) images were taken using a field emission scanning electron microscopy (Hitachi S4700, 15 kV). UV-vis spectra of MgHNPs nanoparticles were recorded by a UV-vis, SPUV-26, Sco-tech

spectrophotometer in the wavelength region 200 to 700 nm operated at a resolution of 1 nm.

RESULTS AND DISCUSSIONS

X-ray diffraction analysis (XRD) pattern of the synthesized magnesium hydroxide nanoparticles is shown in Figure-1. The diffraction peaks with 2θ values of 17.89°, 32.45°, 37.49°, 50.22°, 58.24°, 61.58°, 67.70°, and 71.65° correspond to reflections from (001), (100), (101), (102), (110), (111), (103) and (201) planes Bragg reflections, respectively (JCPDS No. 00-044-1482). No characteristic peaks of other phases are detected, which indicates a high purity of the synthesized MgHNPs.

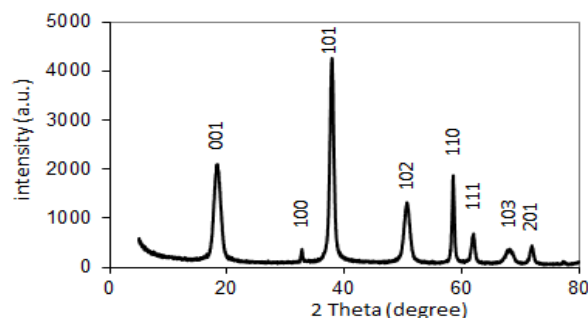


Figure-1. X-ray diffraction pattern of the synthesized MgHNPs.

The average crystallite size of the synthesized magnesium hydroxide nanoparticles calculated using Debye-Scherrer (Klug & Alexander, 1959) equation:

$$D = \frac{K\lambda}{\beta \cos\theta}$$

Where, D is the crystallite size of magnesium hydroxide nanoparticles, λ represents wavelength of x-ray source 0.1541 nm used in XRD, β is the full width at half maximum of the diffraction peak, K is the Scherrer constant with value from 0.9 to 1 and θ is the Bragg angle. The average crystallite size of the synthesized magnesium hydroxide powders calculated to be 40nm.

Fourier transform infrared spectroscopy (FT-IR) pattern of *O. europaea* leaves extract; Figure-2 displayed a number of absorption peaks, reflecting its complex nature. The broad band at 3541-3336 cm^{-1} could be ascribed to the stretching absorption bands of attributed to hydrogen bonded -OH groups of alcohols and phenols and also to the presence of amines -NH of amide. The strong absorption peaks at 2924 cm^{-1} and 2845 cm^{-1} could be assigned to the asymmetric and symmetric stretching of CH_2 and CH_3 functional groups of aliphatic. The strong bands at 1743 cm^{-1} and 1612 cm^{-1} are characteristic of amide carbonyl group in amide I and amide II. The band 1423 cm^{-1} is assigned to the methylene scissoring vibrations from the proteins. C-N stretch of aromatic amines and carboxylic acids. The band at 1253 cm^{-1} is due to C-O vibrations of alcohols, phenols and C-N stretching



vibrations of amine. The band at 1041 cm^{-1} is assigned to the C-O stretching vibrations of alcohols. The peaks at 655 cm^{-1} are assigned to aromatic compounds. These functional groups act as dispersing, capping and stabilizing agents for MgHNPs during the process of synthesis. FT-IR spectrum indicated a new chemistry linkage on the surface of magnesium hydroxide nanoparticles. This suggests that *O. europaea* leaves extract can bind to MgHNPs. FT-IR analysis of

synthesized magnesium hydroxide nanoparticles, Figure-3, the strong peaks at 3699 cm^{-1} and 451 cm^{-1} are assigned to Mg-O-H stretching vibration. The strong absorption peak at 3699 cm^{-1} corresponds to O-H stretching mode arising from the absorption of water on the surface of MgHNPs and N-H stretching absorption of amines. The structural changes in FT-IR spectra indicated that the capping and stabilization of magnesium hydroxide nanoparticles via the coordination with OH, -NH, C=O, C=N.

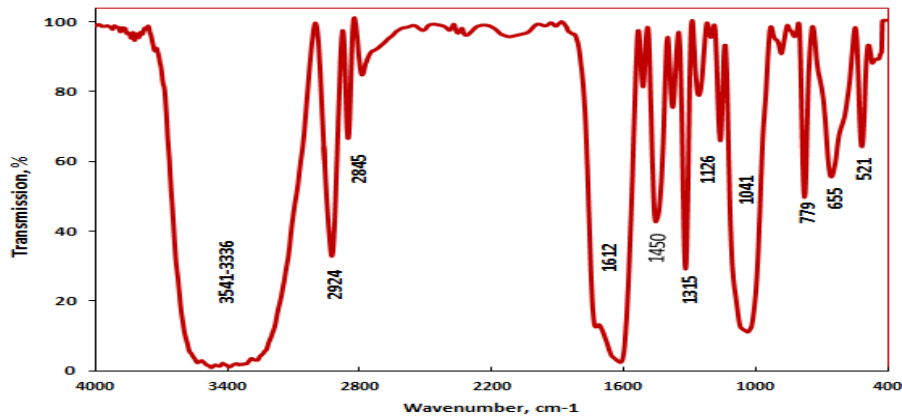


Figure-2. FT-IR spectrum of *O. europaea* leaves extract.

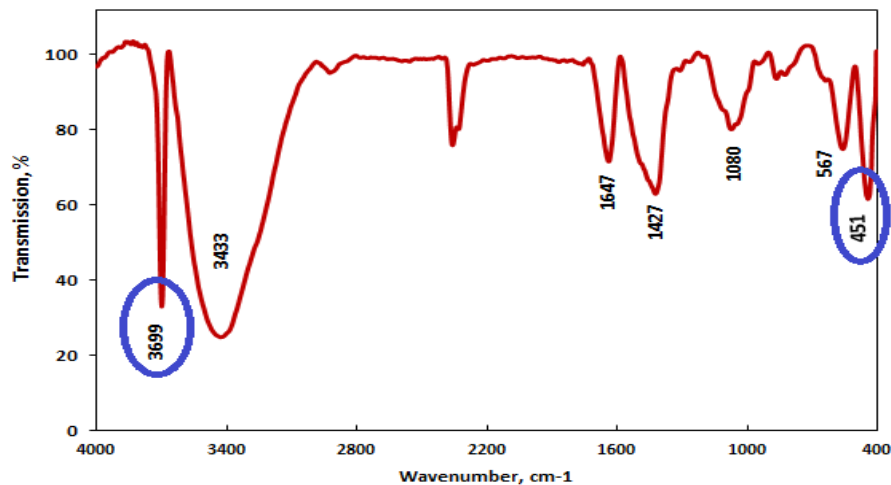


Figure-3. FT-IR spectrum of synthesized MgHNPs.

Formation and stability of MgHNPs in sterile distilled water is confirmed using UV-vis spectrophotometer in a range of wavelength from 200 to 700 nm. UV-vis spectra were recorded as function of reaction time. We observe that there is no peak showing

no sign for the synthesis of magnesium hydroxide nanoparticles but after 2 min the surface Plasmon resonance of magnesium hydroxide occur at 300 nm and steadily increasing with the time of reaction without much change in the peak wavelength (Figure4).

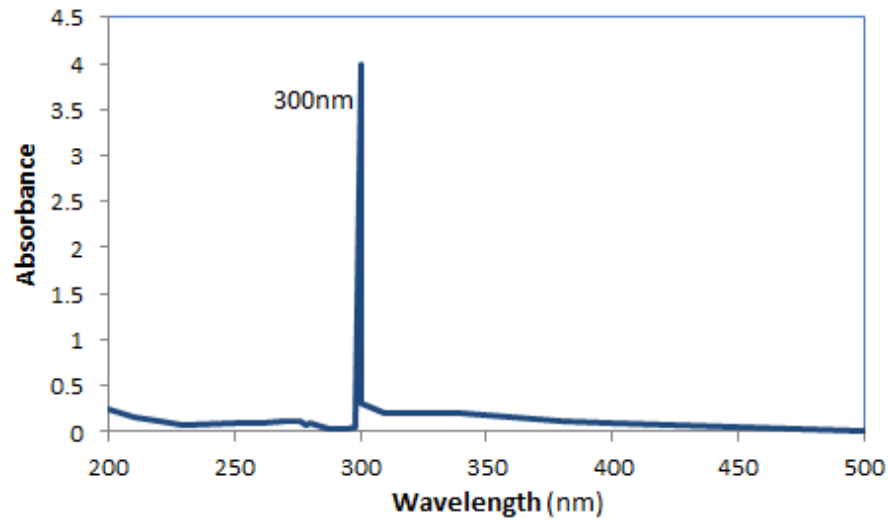


Figure-4. UV-vis spectrum of synthesized MgHNPs.

Scanning electron microscopy image of magnesium hydroxide nanoparticles is shown in Figure-5. It can be seen that the sample consists entirely of Nanoparticles with an average size 40nm and it appears that the nanoparticles tend to assemble into nanoparticles bundles and each bundle consists of several nanoparticles with random orientations. Knowing that $Mg(OH)_2$ can be transformed into MgO due to heating from the electron

beam, the electron dosage was kept low to minimize the sample heating. However, no visible change in the SEM images indicating the high stability of $Mg(OH)_2$ in vacuum and under the electron. Energy-dispersive (EDS) analysis of synthesized MgHNPs Figure-6 showed that traces of potassium and sulfur suggesting related to crystalline and amorphous organic phase of *O. europaea* leaves.

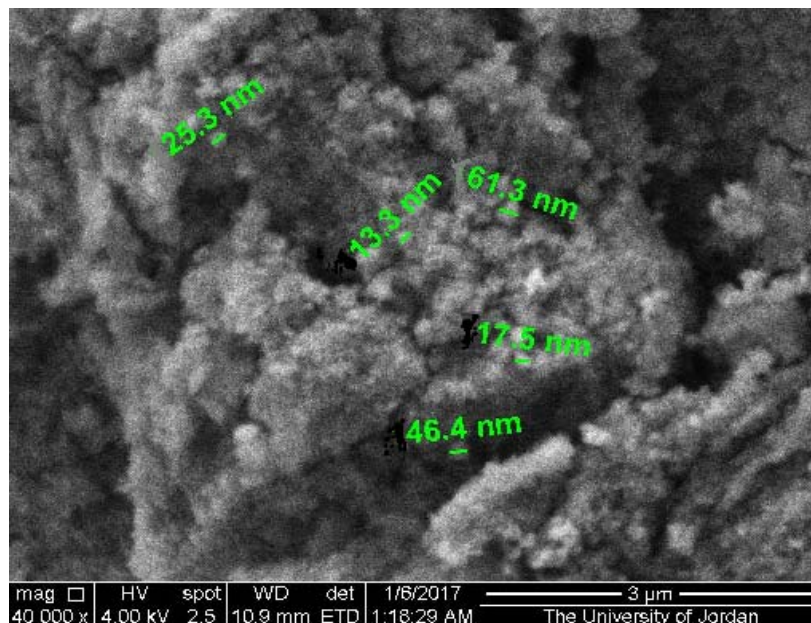


Figure-5. SEM micrograph of MgHNPs.

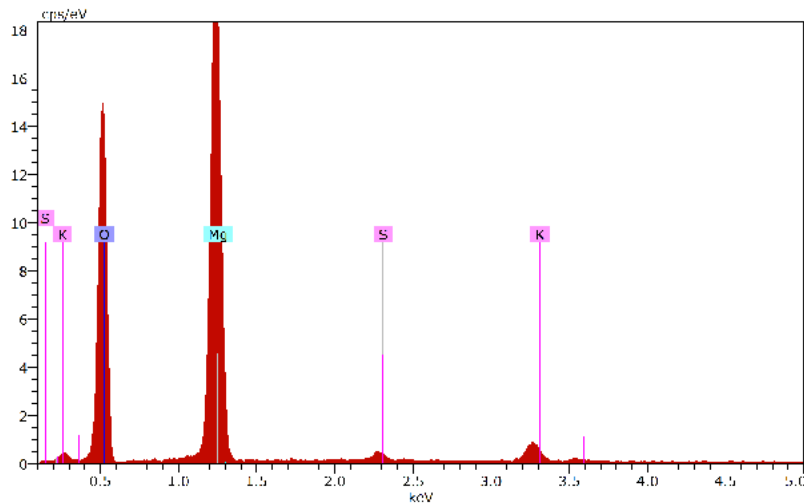


Figure-6. EDS pattern of synthesized MgHNPs.

Effect of synthesized $Mg(OH)_2$ nanoparticles onto the green peach aphid (GPA) was carried out at the University of Jordan and the Royal Scientific Society. The effect of different concentrations of MgHNPs on GPA is shown in Table-1, Table-2, Figure-7 and Figure-8). Means of mortalities % of the 1st and 2nd nymphal instars caused by the five concentrations were different significantly. Means of mortalities % of 3rd and 4th nymphal instars of the same aphid caused by the same concentrations were also different significantly. However, the mortalities

percent (%) of both aphid categories in all concentrations were greater significantly than the control treatment. The same trend for CuONPs and ZnONPs in separated experiments on early and late nymphal instars of the green peach aphid was obtained (Ghidan *et al.*, 2016; 2017). However, silver and zinc nanoparticles have been tested against the oleander aphid (*Aphis nerii*) and showed insecticidal effect (Rouhani and Samih, 2011; Rouhani *et al.*, 2012).

Table-1. Means of mortalities percent (%) of 1st and 2nd nymphal instars of the green peach aphid by five different concentrations of MgHNPs.

Concentration ($\mu\text{g/ml}$)	Mortality % 1 st and 2 nd nymphal instars \pm SE after		
	24h	48h	72h
control	4e \pm 0.6	5 d \pm 0.5	5b \pm 0.7
250	28d \pm 2.7	33 c \pm 1.7	92a \pm 8.3
1000	50 c \pm 4.0	90b \pm 2.9	95 \pm a 5.0
2000	64 b \pm 2.0	100a \pm 0.0	100a \pm 0.0
4000	67 b \pm 2.0	100a \pm 0.0	100 a \pm 0.0
8000	82a \pm 0.7	100a \pm 0.0	100a \pm 0.0

*Means within the same column of the same period of time sharing the same letter do not differ significantly at 5% level using Frisher Protected LSD test.

The highest mortality percent (%) of 1st and 2nd nymphal instars was in 8000 ($\mu\text{g/ml}$) concentration. It was 82 % after 24 hours, then reached 100% after 48 and 72 hours, while mortality increased at 4000($\mu\text{g/ml}$) concentration reached 100 after 48 and 72 hours. The lowest mortality was in case of using 250 ($\mu\text{g/ml}$) after 24 hours then increased to 33 and 92 % after 48 and 72 hours, respectively. The mortalities in case of control were

significantly the least after 24, 48 and 72 hours compared with the other treatments. However, in case of testing of CuONPs on 1st and 2nd nymphal instars of the green peach aphid, the highest mortalities for the 8000 ($\mu\text{g/ml}$) concentration was 86% (Ghidan *et al.*, 2016). However, nanoparticles of zinc at 700 mL^{-1} had the highest insect mortality effect (Rouhani *et al.*, 2012).

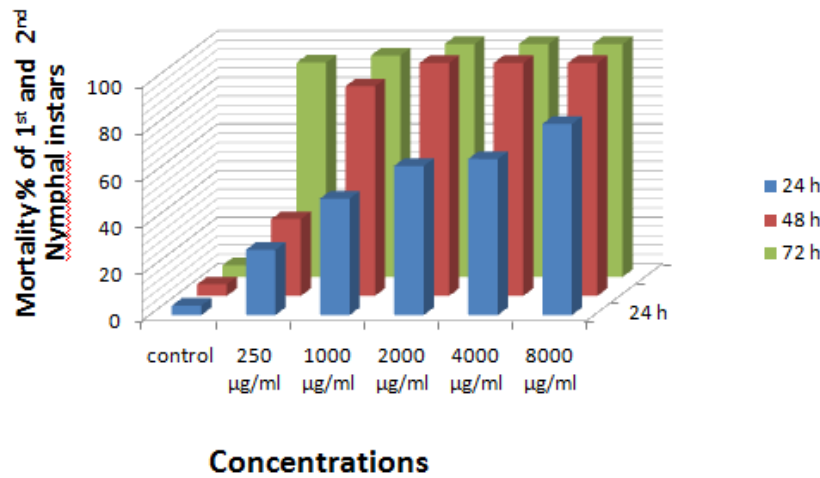


Figure-7. Mortalities percent (%) of 1st and 2nd nymphal instars of the green peach aphid by five different concentrations of MgHNPs.

Table-2. Means of mortalities percents (%) of 3rd and 4th nymphal instars of the green peach aphid by five different concentrations of MgHNPs.

Concentration (µg/ml)	Mortality % of 3 rd and 4 th nymphal instars ± SE after		
	24h	48h	72h
control	4d± 0.6	4 d± 0.7	5 b± 0.9
250	25c ± 2.0	27c± 1.7	94 a± 10
1000	36b ± 3.3	80b± 1.5	93 a± 4.7
2000	47a ± 3.3	81b± 1.7	92 a± 4.0
4000	48a ± 2.3	85ab± 2.6	93a± 1.8
8000	50a ± 0.0	87a ± 2.0	94a± 5.7

*Means within the same column of the same period of time sharing the same letter do not differ significantly at 5% level using Frisher Protected LSD test.

The highest mortality % of 3rd and 4th nymphal instars was in 8000 (µg/ml) concentration. It was 50.0 % after 24 hrs, then reached 87,94% after 48 and 72 hrs, respectively. While mortality increased at 4000(µg/ml) concentration reached 85, 93 % after 48 and 72hrs, respectively. The lowest mortality was in case of using 250 (µg/ml) after 24 hour then increased to 27 and 94% after 48 and 72 hours, respectively. The mortalities in case of control were significantly the least after 24, 48 and 72 hours compared with the other treatments. However, in case of testing of CuONPs on 3rd, and 4thnymphal instars of the green peach aphid, the lowest mortalities for the 250 (µg/ml) concentration was 14% (Ghidan *et al.*, 2016).

The toxicity of five concentrations of MgHNPs on the GPA are shown in Tables 1 and 2. Means of mortalities % of the 1st and 2ndnymphal instars of the green peach aphid by five different concentrations of MgHNPs were different significantly. Means of mortalities % of the 3rd and 4th nymphal instars of the same aphid caused by the same concentrations were also different significantly. However, mortality % of both aphid categories in all concentrations were greater significantly than the control treatment. However, the same trend for CuONPs and ZnONPs which were tested against the same aphid was obtained where there were significant differences between mortality for both aphid categories than control.

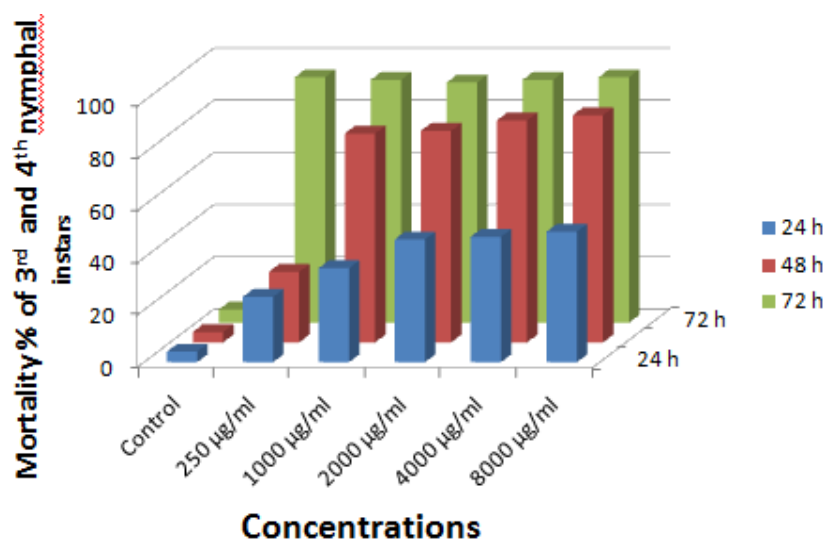


Figure-8. Mortalities percent (%) of 3st and 4^{ed} nymphal instars of the green peach aphid by five different concentrations of MgHNPs.

CONCLUSIONS

A new and green route has been developed for synthesis magnesium hydroxide nanoparticles in one-pot reaction process at ambient temperature. Our synthesis approach employed an environmental friendly biomaterial *O. europaea* leaves extract, which act as dispersing agent and prevents the agglomeration of magnesium hydroxide nanoparticles (MgHNPs) formed during synthesis. XRD and SEM data showed that the size and shape of the magnesium hydroxide nanoparticles could be controlled by the amount of *O. europaea* leaf extract and the concentration of Mg(II) ions. The method in the present study offers several important advantageous features. 1. The synthesis method is eco-friendly and economical, because it involves inexpensive and non-toxic materials, 2. Size-controlled magnesium hydroxide nanoparticles are produced easily by different amounts of *O. europaea* leaf extract, 3. Scale up the process and 4. The activity of biologically synthesized magnesium hydroxide nanoparticles was in different concentrations evaluated against the mortality of green peach aphid. However, nanoparticles can be used as a valuable tool in pest management programs of *Aphis nerii* (Rouhani and Samih, 2011). From the present study, it can be concluded that there were significant differences in the aphid mortalities between the different concentrations of the MgHNPs nanoparticles. In addition, the differences between the different concentrations and the control were significantly obvious. This will lead to the main conclusion that MgHNPs is a potential aphicide to decrease the population. However, further studies are needed to be done on this issue in the greenhouse and the field.

ACKNOWLEDGEMENTS

Authors acknowledge the support of Royal Scientific Society (RSS) and the University of Jordan, Amman, Jordan

REFERENCES

- Al-Antary T., Khadar B. 2013. Toxicity of four insecticides on longevity and fecundity of three populations of green peach aphid *Myzus persicae* Sulzer (Homoptera: Aphididae) for three generations. *Jordan Agricultural Science*. 9: 52-62.
- Awwad A.M., Ahmad, A.H. 2014. Biosynthesis, characterization, and optical properties of magnesium hydroxide and oxide nanoflakes using *Citrus limon* leaf extract. *Arab J. Phys. Chem*. 1: 65-70.
- Baidukova O., Skorb E.V. 2016. Ultrasound-assisted synthesis of magnesium hydroxide nanoparticles from magnesium. *Ultrasonica Sonochemistry*. 31: 423-428.
- Birgücü A.K., Satar, S., Karaca I. 2015. Effects of Some Plant Extracts on *Aphis gossypii* Glover (Homoptera: Aphididae) and *Bemisia tabaci* (Gennadius) Takahashi (Homoptera: Aleyrodidae). *Asian Journal of Agriculture and Food Sciences*. 3: 149-154.
- Blackman R.L., Eastop V.F. 2000. Aphids on the world's crops, an identification and information guide. Chichester, UK: John Wiley & Sons Ltd.
- Bucholz E., Frampton J., Jetton R., Tilotta D., Lucia L. 2017. Effect of different headspace concentrations of bornyl acetate on fecundity of green peach aphid and balsam woolly adelgid. *Scandinavian Journal of Forest Research*. 1-9, in Press.
- Chen D., Zhu L., Liu P., Zhang H., Xu X. 2009. Rod-like morphological magnesium hydroxide and magnesium oxide via a wet coprecipitation process. *J. Porous Mater*. 16: 13-18.



- Dhaouadi H., Chaabane H., Touati F. 2011. Mg (OH)₂ nanorods synthesized by a facile hydrothermal method in the presence of CTAB. *Nano Micro Lett.* 3: 153-159.
- Erdoğan P., Yıldırım A. 2016. Insecticidal Activity of Three Different Plant Extracts on the Green Peach Aphid [(Myzus persicae Sulzer) (Hemiptera: Aphididae)]. *Journal of the Entomological Research Society.* 18: 27-35.
- Hanlon J.M., Diaz L.B., Balducci G., Stobbs B.A., Bielewski M., Chung P., MacLaren I., Gregory D.H. 2015. Rapid surfactant-free Hanlon synthesis of Mg(OH)₂nanoplates and pseudomorphic dehydration to MgO. *Cryst. Eng. Comm.* 17: 5672-5679.
- Ghidan A.Y., Al-Antary T.M., Awwad A.M. 2016. Green synthesis copper oxide nanoparticles using Punicagranatumpeels extract: Effect on green peach Aphid. *Environmental Nanotechnology, Monitoring & Management.* 6: 95-98.
- Ghidan A.Y., Al-Antary T.M., Salem N.M., Awwad A.M. 2017. Facile Green Synthetic Route to the Zinc Oxide (ZnONPs) Nanoparticles: Effect on Green Peach Aphid and Antibacterial Activity. *Journal of Agricultural Science.* 9: 131-138.
- Jiang W., Hua X., Han Q., Yang X., Lu L., Wang X. 2009. Preparation of lamellar magnesium hydroxide nanoparticles via precipitation method. *Powder Technology.* 191: 227-230.
- Jinghui W., Xin L., Junwei Y., Yuan L., Guiling N. 2009. Facile controlled synthesis of Mg(OH)₂ nanoparticles with novel shapes. *Adv. Mater. Res.* 58: 55-58.
- Klug H.P., Alexander L.E. 1959. X-ray diffraction procedures, Wiley, New York, USA.
- Madanat H.M., AlAntary T.M., Musa H. Abu Zarqa M.H. 2016. Toxicity of six ethanol plant extract against the green peach aphid *MYZUS persicae* Sulzer (HOMOPTERA: APHIDIDAE). *Fresenius Environmental Bulletin.* 25: 706-718.
- Mazzoni E., Cravedi P. 2002. Analysis of insecticide-resistant Myzus persicae (Sulzer) populations collected in Italian peach orchards. *Pest Manag. Sci.* 58: 975-980.
- Moorthy S.K., Ashok CH., Rao K.V., Viswanathana C. 2015. Synthesis and characterization of MgO nanoparticles by Neem leaves through green method. *Materials Today: Proceedings.* 2: 4360-4368.
- Pilarska A., Wysokowski M., Markiewicz E., Jesionowski T. 2013. Synthesis of magnesium hydroxide and its calcinates by a precipitation method with the use of magnesium sulfate and poly (ethylene glycols). *Powder Technology.* 235: 148-157.
- Panini M., Tozzi F., Bass C., Zimmer C.T., Field L., Borzatta V., Mazzoni E., Moores G. 2017. The interactions of piperonylbutoxide and analogues with the metabolic enzymes FE4 and CYP6CY3 of the green peach aphid Myzus persicae (Hemiptera: Aphididae). *Pest Management Science.* 73: 3371-379.
- Qin Y., Zhang J., Song D., Duan H., Li W., Yang X. 2016. Novel (E)-β-farnesene analogues containing 2-nitroimino-hexahydro-1, 3, 5-triazine: Synthesis and biological activity evaluation. *Molecules.* 21: 825.
- Rouhani M. and Samih, M. 2011. Entomotoxic effect of nanoparticles of ZnTiO₂ - Ag against oleander aphid, *Aphis nerii*. *Plant Protection Journal.* 3: 15- 25.
- Rouhani M., Samih, M. and Kalantari S. 2012. Insecticide effect of silver and zinc nanoparticles against *Aphis nerii* Boyer de Fonscolombe (Hemiptera: Aphididae). *Chilean Journal of Agricultural Research.* 4: 590-594.
- Rousselin A., Sauge M.H., Vercambre G., Bevacqua D., Lescorret F., Jordan M.O. 2016. Effects of nitrogen and vegetative growth on the population dynamics of the aphid, Myzus persicae, in the peach tree, Prunus persica. *Acta Horticulturae.* 1137: 137-144.
- Samara R., Lowery D.T., Stobbs L.W., Vickers P.M., Bittner L.A. 2016. Horticultural mineral oil influences Plum pox virus transmission by Myzus persicae. *Journal of Applied Entomology.* 140: 688-696.
- Shen H., Liu Y. 2016. In situ synthesis of hydrophobic magnesium hydroxide nanoparticles in a novel impinging stream-rotating packed bed reactor. *Journal of Chinese Chemical Engineering.* 24: 1306-1312.
- Song X., Sun S., Zhang D., Wang J., Yu J. 2011. Synthesis and characterization of magnesium hydroxide by batch reaction crystallization. *Frontiers of Chemical Science and Engineering.* 5: 416-421.
- Srivastava V., Sharma Y.C., Sillanpää M. 2015. Green synthesis of magnesium oxide nanoflower and its application for the removal of divalent metallic species from synthetic wastewater. *Ceramics International.* 41: 6702-6709.
- Sugirtha P., Divya R., Yedhukrishnan R., Suganthi K.S., Anusha N., Ponnusami V., Rajan K.S. 2015. Green synthesis of magnesium oxide nanoparticles using *Brassica oleracea* and *Punicagranatum* peels and their anticancer and photocatalytic activity. *Asian Journal of Chemistry.* 27: 2513-2517.
- Sushma N.J., Prathyusha D., Swathi G., Madhavi T., Deva B., Raju P., Mallikarjuna K., Kim H-S. 2016. Facile approach to synthesize magnesium oxide nanoparticles by using Clitoria ternatea - characterization and in vitro antioxidant studies. *Applied nanoscience.* 6: 437-444.



Tai C.Y., Tai C-T., Chang M-H., Liu H-S. 2007. Synthesis of magnesium hydroxide and oxide nanoparticles using a spinning disk reactor. *Ind. Eng. Chem. Res.* 46: 5536-5541.

Tong S-M, Feng M-G. 2016. Aphidicidal activity of a novel botanical insecticides made by alkalization of bambo tar. *Crop Protection.* 87: 85-89.

Wang P., Li C., Gong H., Wang H., Liu J. 2011. Morphology control and growth mechanism of magnesium hydroxide nanoparticles via a simple wet precipitation method. *Ceramics International.* 37: 3365-3370.

Wu J., Yan H., Zhang X., Wei L., Liu X., Xu B. 2008. Magnesium hydroxide nanoparticles synthesized in water-in-oil microemulsions. *J. Colloid. Interface Sci.* 324: 167-1671.

Xu H., Deng X-R. 2006. Preparation and properties of superfine Mg(OH)₂ flame retardant. *Trans. Nonferrous Met. Soc. China.* 16: 488-492.

Yan H., Zhang X-h., Wu J-m., Wei L-q., Liu X-g., Xu B-s. 2008. The use of CTAB to improve the crystallinity and dispersibility of ultrafine magnesium hydroxide by hydrothermal route. *Powder Technology.* 188: 128-132.

Yousefi M. 2014. A fast method for synthesis magnesium hydroxide nanoparticles, thermal stable and flame retardant poly vinyl alcohol nanocomposite. *Journal of Nanostructures* 4: 383-388.

Zhou W., Qiao X., Zhang L., Guo J., Zheng J. 2014. Synthesis and hemolytic activity of magnesium hydroxide nanoparticles. *Advanced Materials Research.* 971-973: 228-231.

Zapata N., Van Damme E.J.M., Vargas M., Devotto L., Smaghe G. 2016. Insecticidal activity of a protein extracted from bulbs of *Phycellaaustralis* Ravenna against the aphids *Acyrtosiphonpisum* Harris and *Myzuspersicae* Sulzer. *Chilean Journal of Agricultural Research.* 76: 188-194.