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Green synthesis of copper oxide nanoparticles using *Punica granatum* peels extract: Effect on green peach Aphid

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ABSTRACT

Copper oxide nanoparticles (CuONPs) were synthesized by a simple and green method using *Punica granatum* peels extract at room temperature. Biosynthesized copper oxide nanoparticles were characterized by Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), UV–vis absorption spectroscopy, and X-ray diffraction (XRD). The particles are crystalline in nature with average size 40 nm. The morphology of the copper oxide nanoparticles could be controlled by tuning the amount of *Punica granatum* peels aqueous extract and copper ions. This study determined the mortality efficacy of the synthesized CuONPs against green peach Aphid.

1. Introduction

Copper oxide (CuO) is an important metal oxide semiconductor with a narrow band gap of 1.7 eV. Further it can be used in pesticides formulation, and antibacterial agents. Copper oxide nanoparticles are synthesized through different techniques and methods including precipitation (Sahooli et al., 2012), sonochemical route (Safarifard and Morsali, 2012), sol-gel (Pandiyarajan et al., 2013), hydrothermal approach (Mohamed et al., 2014; Outokesh et al., 2011), chemical bath deposition (Jiang et al., 2015), chemical reduction (Karthik and Geetha, 2013), non-vacuum and spin coating sol-gel techniques (Yahia et al., 2016) and reflux contestation (Bouazizi et al., 2015). These methods have many disadvantages due to the difficulty of scale up the process of synthesis, separation and purification of the nanoparticles, energy consumption and using hazardous chemicals. Recently, there have been related works employed the potential of green methods to synthesize copper oxide nanoparticles using plant aqueous extracts such as *Gundelia tournefortii* leaves and stems (Nasrollahzadeh et al., 2015), *Tinospora cordifolia* (Udayabhanu et al., 2015), *Calotropis gigantean* leaf (Sharma et al., 2015), *Aloe barbadensis* leaves (Gunalan et al., 2012; Kumar et al., 2015), *Carica papaya* leaves (Sankar et al., 2014), *Gloriosa superba* L (Naika et al., 2015), *Citrus limon* juice (Mohan et al., 2015), *Tabernaemontana divaricate* leaf (Sivaraj et al., 2014), carob leaf (Awwad and Ibrahim, 2015), and *Malva sylvestris* leaf (Awwad et al., 2015).

Aphids are insects able to attack several plants, secreting honey dew and transmitting viral diseases economic plants (Al-Antary and Khadir, 2013). The green peach aphid *Myzus persicae* Sulzer (Homoptetra: Aphidae) is worldwide distributed, polyphagous and with wide host range. In Jordan, the green peach Aphid attacks several economic plants and vegetables particularly sweets and hot peppers.

The present study was designed with a novel, rapid, and cost-effective route for biosynthesis of copper oxide nanoparticles (CuONPs) using *Punica granatum* peels extract. The synthesized copper oxide nanoparticles obtained by the green method are under investigation of their effect on green peach Aphid.

2. Experimental

2.1. Materials

Copper acetate monohydrate [$Cu(CH_3COO)_2{\cdot}H_2O$] is analytical grade purchased from Merck, Darmstadt, Germany and used without further purification. Deionized distilled water was used in all experimental work.

2.2. Preparation of Punica granatum peels extract

Fresh peels of healthy *Punica granatum* fruits were collected from local market, Jordan. Peels were washed several times with water to remove dust particles and then dried in shade for two weeks to remove the residual moisture. *Punica granatum* peels aqueous extract was prepared by placing 10 g of dried fine powder in 500 ml glass beaker along with 400 ml of sterile distilled water.

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The mixture was boiled for 10 min until the color of aqueous solution changed from watery to brown-yellow. Then the mixture was cooled to room temperature and filtered with Whatman No. 1 filter paper before centrifuging at 1200 rpm for 5 min to remove biomaterials. The extract was stored at room temperature in order to be used for further experiments.

2.3. Green synthesis of copper oxide nanoparticles (CuONPs)

In a typical reaction mixture, 2.8 g of copper acetate monohydrate was dissolved in 500 ml of the deionized water and stirred magnetically at room temperature for 5 min. Afterwards, *P. granatum* peels aqueous extract was added dropwise under stirring, as soon as, the peels extract comes in contact copper ions spontaneous change the blue color of copper ions to green color. The obtained green mixture was left under stirring at room temperature. After 10 min, the green mixture started changing to a brown suspended mixture, indicating the formation of water soluble monodispersed copper oxide nanoparticles.

2.4. Characterization techniques

Scanning electron microscopy (SEM) analysis of synthesized copper oxide nanoparticles was done using a Hitachi S-4500 SEM machine. Powder X-ray diffraction was performed using a X-ray diffractometer, Shimadzu, XRD-6000 with CuKα radiation λ = 1.5405 Å over a wide range of Bragg angles ($20^\circ \leq 2\theta \leq 80^\circ$). Fourier transform infrared spectroscopic measurements were done using Shimadzu, IR-Prestige-21 spectrophotometer. UV–vis spectrum of copper oxide nanoparticles was recorded, by taking 0.1 ml of the sample and diluting it with 2 ml deionized water, as a function of time of reaction using a Schimadzu1601 spectrophotometer in the wave length region 300–700 nm operated at a resolution of 1 nm.

3. Results and discussion

3.1. X-ray diffraction analysis

Fig. 1 shows the X-ray diffraction (XRD) pattern of the synthesized CuONPs powder. The XRD pattern revealed the orientation and crystalline nature of copper oxide nanoparticles. The peaks position with 2θ values of 35.22°, 38.36°, 48.05°, 52.4°, 56.56°, 60.78°, 65.4°, and 73.89° are indexed as (0 0 2), (1 1 1), (2 0 2), (0 2 0), (202), (113), (311), (113), planes, which are in good agreement

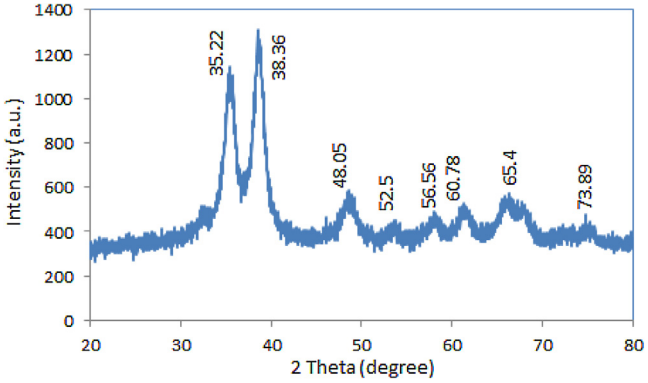

Fig. 1. XRD pattern of the synthesized CuO nanoparticles using *P. granatum* peels extract.

with those of powder CuO obtained from the International Center of Diffraction Data card (JCPDS-45-0937) confirming the formation of a crystalline structure. No extra diffraction peaks of other phases are detected, indicating the phase purity of CuONPs. The average crystallite size of the synthesized copper oxide nanoparticles was calculated using Debye-Scherrer equation (Sankar et al., 2014; Vidhu et al., 2011):

$$D = Lambda\ K/cos\theta$$

where

D – The crystallite size of copper oxide nanoparticles,

λ – Represents wavelength of X-ray source 0.15406 nm used in XRD,

β – The full width at half maximum of the diffraction peak,

K – The Scherrer constant with value from 0.9 to 1 and θ is the Bragg angle.

The average particle size of CuONPs was calculated 40 nm by using above Debye-Scherrer·s formula.

3.2. Fourier infrared spectroscopy (FT-IR) analysis

Fourier transform infrared spectroscopy is used to identify and get an approximate identification of the possible biomolecules in plant extract. FT-IR spectrum of *P. granatum* peels extract is shown in Fig. 2. FT-IR displays a number of absorption peaks, reflecting its complex nature due to biomolecules. Strong and broad peak at 3379 cm^{-1} attributed to hydrogen bonded O—H groups of alcohols and phenols and also to the presence of amines N—H of amide. The

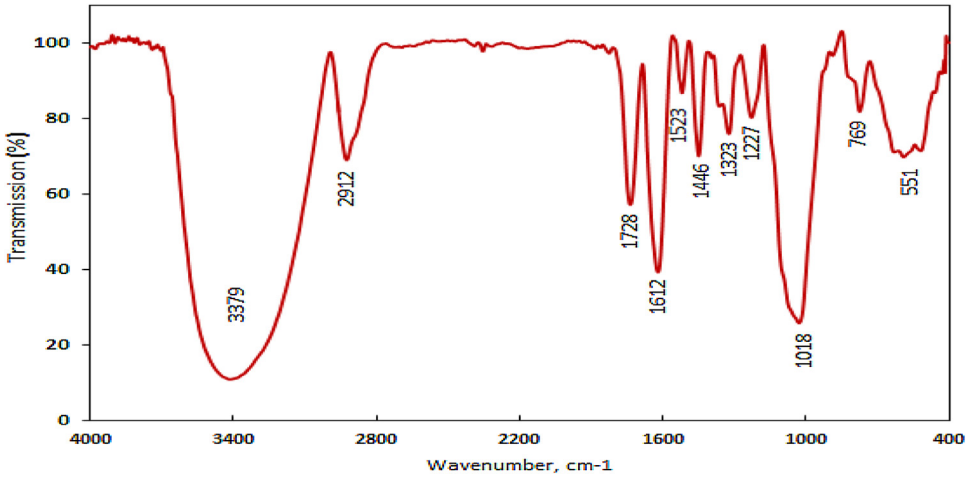

Fig. 2. FT-IR spectrum of *Punica granatum* peels extract.