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Green synthesis and characterization of ZnO nanoparticles using *Solanum rantonnetii* leaves aqueous extract and antifungal activity evaluation

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ABSTRACT

In view of advantages of green synthesis, a novel green route for synthesis and stabilization of zinc oxide nanoparticles (ZnONPs) using aqueous extract of *Solanum rantonnetii* leaves at room temperature. The formation of ZnONPs is monitored by recording the UV–vis absorption spectra for surface Plasmon resonance (SPR) peak (~374 nm). X-ray diffraction (XRD) pattern of the ZnONPs agrees with the reported data for Zn metal and the crystallite average size is 12 nm. Scanning and transmission electron microscopic (SEM and TEM) show uniform spherical particles obtained by this green method. The antifungal activity is found to be effective of ZnONPs. Results revealed that the green synthesis is an efficient for the preparation of ZnONPs as an active antifungal agent for practical applications.

Capsule Summary: *Solanum rantonnetii* leaves aqueous extract for green synthesis of zinc oxide nanoparticles at room temperature, were leaves extract act as reducing and stabilizing agent. The ZnONPs have spherical shape and well dispersed with average size of 12 nm.

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INTRODUCTION

Different structures of ZnONPs as antibacterial activity can be synthesized by chemical and physical methods such as a chemical precipitation method (Kumar et al., 2013; Ghorbani et al., 2015), sol-gel method (Hasnidawani et al., 2016; Chung et al., 2015; Modwi et al., 2021), sonochemical synthesis (Khanna et al., 2012), microwave-assisted combustion (Kooti and Sedeh; 2013), microemulsion method (Yildirim and Durucan., 2010), hydrothermal synthesis (Baruwati et al., 2006; Gerbreders et al., 2020). Recently researchers concentrated their research work on using plants extract such as leaves, fruits, flowers as green methods for synthesis ZnONPs, using plants, i.e., fruits extract of *Ananas Comosus* (Ahmad et al., 2019*)*, aqueous extract of *Garcinia mangostana* fruit pericarp (Aminuzzaman et al., 2018), *Ailanthus altissima* fruit extracts (Awwad et al., 2020), aqueous fruit extracts of *Myristica fragrans* (Faisal et al., 2021), *Hibiscus rosasinensis* leaf extract (Divya et al., 2013) *Azadirachta indica* leaf extract (Bhuyan et al., 2015), leaf extract of *Mentha pulegium* (Rad et al., 2019), *Aloe vera* and *Cassava* starch (Primo et al., 2020), *Deverra tortuosa* aqueous extract (Selim et al., 2020), leaf extracts of *Cassia fistula* and *Melia azadarach* (Naseer et al., 2020), *walnut* leaf extract (Saemi et al., 2021), *Aqueous Piper betle leaf extract* (Tran et al.,

2021), *Lippia adoensis* leaf extracts (Demissie et al., 2020), *Mentha spicata* L. leaves (Chikkanna et al., 2019), plant extract of *chamomile* flower, *olea* europaea leaves and red tomato fruit (Ogunyemi, et al., 2019), aqueous extracts of *Origanum majorana*, *Ziziphus jujube, Elaeagnus angustifolia* fresh leaves, cucumber fruit and *pomegranate* peel (Mohammadian et al., 2018), *Veronica multifida* leaf extract (Doğan et al., 2020), *Laurus nobilis* plant extract (Chemingui et al., 2019), *Papaver somniferum* aqueous extract (Muhammad et al., 2019), *Euphorbia heterophylla* L (Lingaraju et al., 2019), Moringa oleifera (Matinise et al., 2017).

Similarly, *Cassia auriculata*can leaf extract (Ramesh et al., 2021), *salvia officials* extract *(*Alrajhi et al., 2021*), Cannabis Jatropha curcasa Alovera and Tinosporacordifolia* leaves (Thakur et al., 2020), *Prosopis juliflora plant leaf* (Mydeen et al., 2020), aqueous extract of *Becium grandiflorum* (Kahsay et al., 2021), leaf extract of *Citrus reticulata* (Shah et al., 2021) *Syzygium cumini* leaf extract (Arumugam et al., 2021), *Cayratia pedata* leaf extract (Jayachandran et al., 2021), *Phoenix dactylifera* waste (Rambabu et al., 2021), loquat seed extract (Shabaani et al., 2020) and *Ruellia. tuberosa* extract (Vasantharaj et al., 2021), *Trifolium pratense* flower (Dobrucka and Dlugaszewska, 2016) have also been utilized for the green synthesis of nanoparticles.

In view of importance of green synthesis, in the present study, *Solanum rantonnetii* leaves aqueous extract was used for the synthesis zinc oxide nanoparticles (ZnONPs), which were characterized by advanced techniques and antifungal activity was evaluated.

MATERIAL AND METHODS

Chemical and reagents

Zinc acetate dihydrate Zn $(CH_3COO)_2.2H_2O$ (Purum ≥98%) was purchased from Sigma-Aldrich, Germany. De-ionized water was used in all experimental work. *Solanum rantonnetii* leaves were collected from trees at gardens, Amman, Jordan, and washed with water to remove dust particles. Leaves were then dried at ambient temperature for 6 days and then ground to obtain fine powder. The powder was then sieved with 350 mesh.

Aqueous extract preparation

Solanum rantonnetii leaves powder was mixed with 100mL de-ionized water and boiled for 10 min at 80°C to obtain red aqueous extract and then left to cool at ambient temperature. Afterward, aqueous extract was obtained by filtration on Whitman's filter paper and kept for our research work.

Synthesis of zinc oxide nanoparticles (ZnONPs)

A 10 g of zinc acetate dihydrate Zn $(CH_3COO)_2.2H_2O$] was dissolved in 100 mL de-ionized water under stirring with magnetic bar at ambient temperature (27 °C). Afterward, an aqueous extract of *S. rantonnetii* leaves aqueous extract was added drop by drop to zinc solution till the solution started changing from colorless color to white and the formation of suspended particles. The mixture was left overnight and filtered to obtain the suspended particles, which dried in an oven at 80 °C for 4 h (Scheme 1). The powder obtained was subjected to analysis by X-ray diffraction (XRD).

Characterizations

Fourier infrared spectroscopy (FT-IR; IR Prestige-21, Shimadzu) was used to identify the different chemical functional groups present in the *Solanum rantonnetii* leaves aqueous extract. FTIR analyses also used to determine the functional groups are responsible for the reduction of zinc acetate to zinc oxide nanoparticles. The analysis was carried out using KBr and the spectral range varying from 4,000 to 400 cm^{-1}. X-ray diffractometer, XRD-6000 (Shimadzu, Japan) equipped with Cu K α radiation source using Ni as filter at a setting of 30 kV/30mA. All XRD data were collected under the experimental conditions in the angular range $3^o \leq 2\theta \leq 80^o$. Transmission electron microscopy (TEM) analysis of synthesized ZnO nanoparticles was done by TEM machine (Hitachi, Japan). Formation and stability of ZnONPs in sterile distilled water is confirmed using UV-vis spectrophotometer in a range of wavelength from 200 to 700 nm.

Scheme 1: Presentation for ZnONPs green synthesis using *Solanum rantonnetti* leaves aqueous extract

RESULTS AND DISCUSSION

UV-vis analysis

Zinc oxide nanoparticles (ZnONPs) synthesized using *Solanum rantonnetii* leaves aqueous extract were studied.

Formation of zinc oxide nanoparticles were confirmed by UV-vis spectrophotometer (Fig. 1). UV–vis absorption spectrum of synthesized zinc oxide nanoparticles was recorded for the sample in the range of 200–500 nm. The spectrum showed that the absorbance peak at 374 nm corresponding to the characteristic band of zinc oxide nanoparticles.

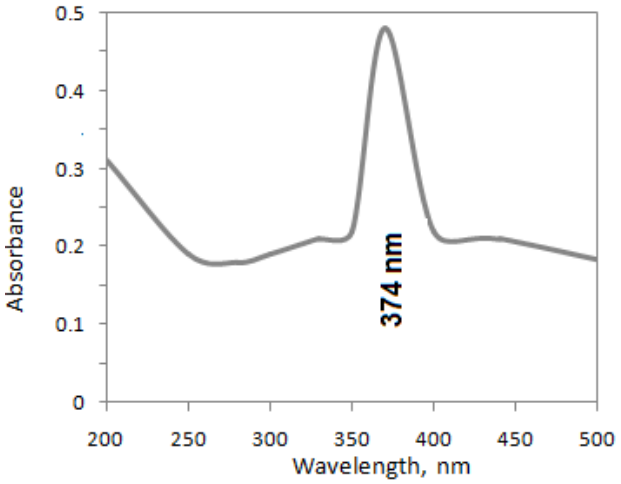

Fig. 1. UV-vis spectrum of ZnONPs synthesized using *Solanum rantonnetti* leaves aqueous extract

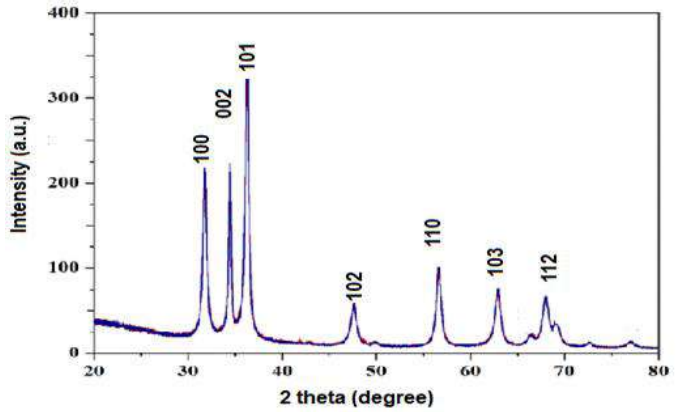

Fig. 2. XRD pattern of ZnONPs synthesized using *Solanum rantonnetti* leaves aqueous extract

XRD analysis

The X-ray diffraction (XRD) pattern of green synthesized ZnONPs is illustrated in Fig. 2. The peak position with 2θ values of 31.7°, 34.4°, 36.2°, 47.5°, 56.6°, 62.8°, and 68.8° are indexed as (100), (002), (101), (102), (110), (103) and (112) planes, which are in good agreement with those of powder ZnO obtained from the International Center of Diffraction Data card (JCPDS-361451) confirming the formation of a crystalline monoclinic structure. No extra diffraction peaks of other phases are detected, indicating the phase purity of ZnONPs. The average crystallite size of the D of synthesized nanoparticles was calculated using the well-known Scherrer formula as shown in Eq. 1.

$$D = 0.9\lambda/\beta\cos\theta \quad (1)$$

Where, λ is the wavelength of X-ray source (Cu-Kα line 0.1541 nm), β is the full width at half maximum (FWHM) in radians and θ is Bragg's diffraction angle. The calculated value of D was 15 for financial support and having given feasibilities to carry out the research work nm.

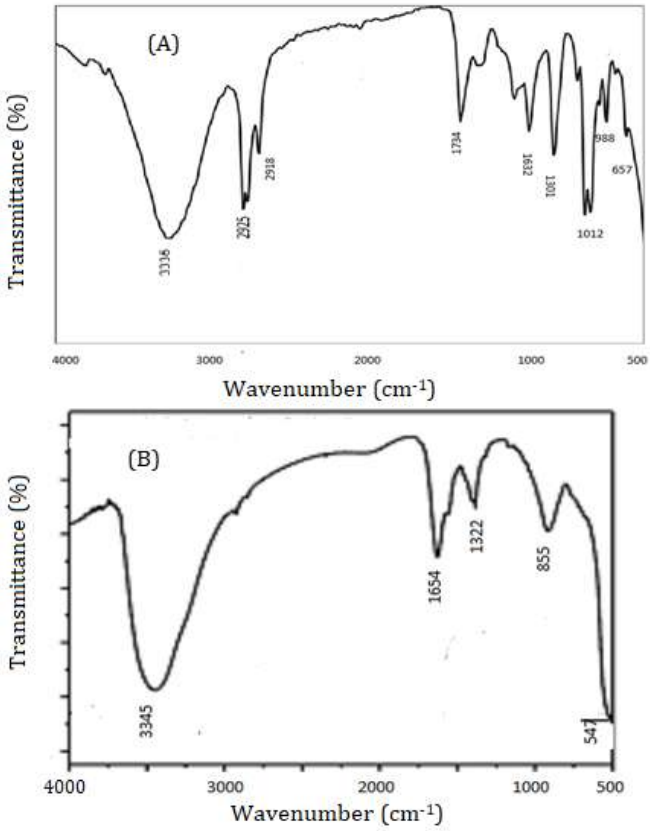

Fig. 3. FT-IR analysis, (A) *Solanum rantonnetti* leaves and green synthesized ZnONPs

FT-IR analysis of extract and ZnONPs

FT-IR analysis of *Solanum rantonnetti* aqueous extract showed that the main absorption peaks at 3336, 2925, 1734, 1632, 1301, 1012, 988, 657 cm^{-1} (Fig. 3A). Strong peak appeared at 3382 refers to the absorption of N-H and O-H stretching. The peaks in FT-IR analysis at 1734 and 1632 cm^{-1} refers to C=O stretching and at 1301 cm^{-1} indicated the O-H bending. This FT-IR analysis showed that *Solanum rantonnetti* leaves extract have the ability to reduce zinc acetate to zinc oxide nanoparticles. Fig 3B. represents the FT-IR of green synthesized ZnONPs and spectrum revealed that the bioactive present in the extract are responsible for nanoparticles formation, which appears in the FTIR of nanoparticles.

Transmission electron microscopy (TEM) analysis

Transmission electron microscopy (TEM) of the synthesized of zinc oxide nanoparticles is illustrated in Fig. 4. It was observed that the ZnONPs have spherical shape with an average diameter of 12 nm. Transmission electron microscopy (TEM) analysis revealed that ZnONPs particles have the average particle size 12 nm. The particle size distribution of the synthesized zinc oxide nanoparicles is ranging between 5 nm and 12 nm A narrow size distribution of the zinc oxide nanoparticles (ZnONPs) was observed with average size 12 nm.

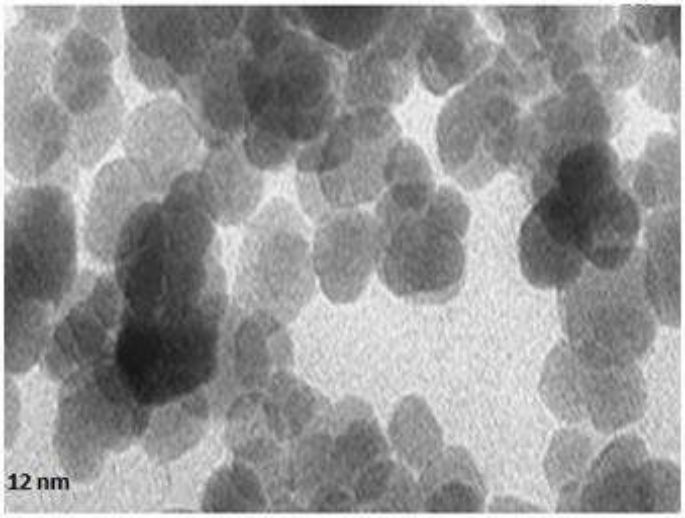

Fig. 4. TEM image of green synthesized ZnONPs using *Solanum rantonnetti* leaves aqueous extract

Antifungal activity of ZnONPs

The antifungal inhibitory effect on the growth was investigated under different concentrations of ZnONPs by a well diffusion assay on a Potato Dextrose Agar (PDA) plate. It was observed that zinc oxide nanoparticles have antifungal activities at different concentrations. The maximum inhibitory activity of 80ppm ± 10ppm ZOI was obtained and increased slightly at 100 and 120ppm concentrations of ZnONPs, No inhibition zone were observed using the aqueous extract of *Solanum rantonnetti* leaves aqueous extract. Present study observed results reveal that the green synthesized ZnONPs showed a significant effect as antifungal towards plant pathogen *F. oxysporum* compared with positive drug control. It could be explained by large surface area of ZnONPs and partially its decomposition in wet medium to $Zn(OH)_2$ which gives better contact with microorganisms thus alter the microbial metabolism and penetrated inside the microorganisms.

CONCLUSIONS

A fast, eco-friendly and convenient green method for the synthesis of ZnONPs nanoparticles from zinc acetate in aqueous extract of *Solanum rantonnetti* leaves at our laboratory temperature. Spherical of particles shape obtained for ZnONPs with sizes ranging from 5 to 12 nm are obtained. These results could be used in developing novel antifungal agent, which may find potential applications in different fields with more safety since NPs could be prepared using green route and the same route can be adopted for the fabrication of NPs based on different NPs metals.

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