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Green Synthesis of Silver Nanoparticles Using Aqueous Extract of *Citrus limon* Peel

Mona Ghoorchibeigi[1], Kambiz Larijani[1,✉], Parviz Aberoomand Azar[1], Karim Zare[1] and Iraj Mehregan[2]

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ABSTRACT

The silver nanoparticles were fabricated by reduction of Ag^{+} to Ag^{0} using aqueous extract of *Citrus limon* peel which grown in Ramsar, province of Mazandaran, North of Iran. Synthesized silver nanoparticles were characterized by means of UV-visible spectroscopy, FT-IR, XRD and SEM for determination size and shape of synthesized silver nanoparticles. The silver nanoparticles in average size of 20-50 nm were fabricated phytochemically using aqueous extract of *Citrus limon* peel.

KEYWORDS

Citrus limon, Silver nanoparticles, Green synthesis.

Author affiliations:

[1]Department of Chemistry, Science and Research Branch, Islamic Azad University, Tehran, Iran
[2]Dertment of Biology, Science and Research Branch, Islamic azad University, Tehran, Iran

✉To whom correspondence to be addressed:

Tel: +98 9123057581
E-mail: klarijani@yahoo.com; larijani_k@yahoo.com

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INTRODUCTION

Synthesis of silver nanoparticles (Ag-NPs) which have the size from 1 to 100 nm is an interested subject in nano materials science and nanotechnology due to its specific application in biology and material science such as solar energy conversion, photonics [1], catalysis [2], microelectronics [3], antimicrobial function-alities [4] and water treatment [5]. Here is a report that Ag-NPs are non-toxic to humans and most effective against bacteria, virus and other eukaryotic microorganism at low concentrations and without any side effects [6]. Silver nanoparticles are prepared using some methods such as physical, chemical [7,8] and biochemical methods [9]. In chemical synthesis of silver nanoparticles often use a reducing agent such as sodium borohydride so these methods are discouraged as they involve the use of toxic chemicals and solvents [10]. So biological methods are considered as safe and ecofriendly for the nanomaterial fabrication as an alternative to conventional chemical methods [11]. The genus *Citrus*, belonging to the *Rutaceae* comprises of about 140 genera and 1,300 species. *Citrus limon* peel extract oils are reported to be one of the rich sources of bioactive compounds namely coumarins, flavonoids, carotenes, terpenes and linalool, *etc*. [12]. In this paper, we report the phytochemical screening and an eco-friendly method to fabrication of silver nanoparticles using the aqueous extract of *Citrus limon* peel.

EXPERIMENTAL

The fruit of *C. limon* was collected at December 2013 from Ramsar, Provice of Mazandaran in North of Iran. The plant material was identified in Citrus Research Institute of Ramsar. The *C. limon* peel was dried in shade for 1 week, crushed and kept in 4 °C for further experiment.

Extract of water soluble constituents: 10 g of *Citrus limon* powdered peel was immersed in 200 mL deionized water in a 250 mL Erlenmeyer flask and heated 80 °C in a water bath for 30 min. The aqueous extract was filtered using a Whatman filter paper No. 1 and the filtered solution dried by freeze drier (CHRIST alpha1-4 LD plus- Germany). The dried extract was kept in 4 °C.

Phytochemical screening of aqueous extract for flavonoids and carbohydrate: 0.1 g of dried extract was dissolved in 50 mL deionized water. Chemical tests were carried out using aqueous extract to identify carbohydrates and flavonoids due to their ability in reduction of Ag^+ ions to Ag^0 nanoparticles, using standard methods.

Test for flavonoids: To 1 mL of aqueous extract, 1 mL of 10 % lead acetate solution was added. The formation of a yellow precipitate was taken as a positive test for flavonoids.

Tests for carbohydrates: Equal volumes of Benedict's reagent and 1 mL of test solution were mixed in a test tube. The mixture was heated in boiling water bath for 5 min. The change of solution colour to green shows the presence of carbohydrate.

Green synthesis of silver nanoparticles: 10 mL of *C. limon* aqueous extract was added to 90 mL of 0.001 M solution of silver nitrate (16.9 mg $AgNO_3$ in 100 mL deionized water) and kept in ambient temperature. After 10 min a brownish red solution can be seen because of reduction of Ag^+ cations to Ag^0 by *C. limon* extract [13].

Structural studies of silver nanoparticles: UV-visible spectral analysis was used to ascertain the formation of silver nanoparticles in aqueous solution based on excitation of surface plasmon resonance (SPR) changes at the wavelength range from 350-550 nm The UV-visible analyses were performed using a Varian Car 300 spectrophotometer (Fig. 1).

The silver nanoparticle solution obtained was centrifuged at 12,000 rpm for 15 min. The sediments were washed by deionized water and re-centrifuged. This step was repeated twice. The dried pellets of Ag-NPs were used for XRD, SEM and EDX analyses for determination of particle size and structural analysis. XRD analysis was performed using a 3003 PTS Seifert (Germany) XRD instrument. The diffracted intensities were recorded from 20° to 80° at 2θ angles. The XRD pattern was showed in Fig. 2. Scanning electron microscopy (SEM) and energy-dispersive microanalysis (EDX) were used to morphological and elemental analysis. The freeze-dried silver nanoparticles were mounted on specimen stubs with double-sided taps, coated with gold in a sputter coater (BAL-TEC SCD-005) and examined under a Philips XL-30 SEM at 12-16 kV with a tilt angle of 45°.

RESULTS AND DISCUSSION

Phytochemical screening on the aqueous extract of *C. limon* peel revealed the presence of flavonoids due to formation of yellowish sediment after adding 10 % lead acetate solution. Any carbohydrate was found in extract due to no colour change after adding Benedict's reagents. The water extract of *C. limon* was used for the synthesis of silver nanoparticles. The reaction started at the first 30 min after adding the extract to 1 mM $AgNO_3$ solution. This was confirmed by the appearance of reddish brown colour in the reaction mixture (Fig. 1) and SPR absorbance of silver nanoparticles in 430-460 nm (Fig. 1).

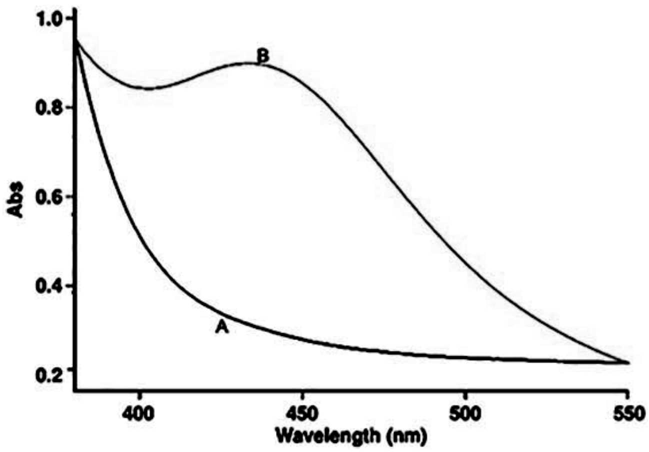

Fig. 1. UV spectra of extract solution + $AgNO_3$ 1 mM solution (A) 0 min, (B) 30 min

XRD pattern of silver nanoparticles (Fig. 2) synthesized by *C. limon* peel aqueous extract shows four intensive peaks at 2θ of 38.74° (111), 45.95° (200), 64.12° (311) and 76.99° (222) which shown the silver nanoparticles were appeared in a face center cubic (fcc) lattice system (Fig. 2).

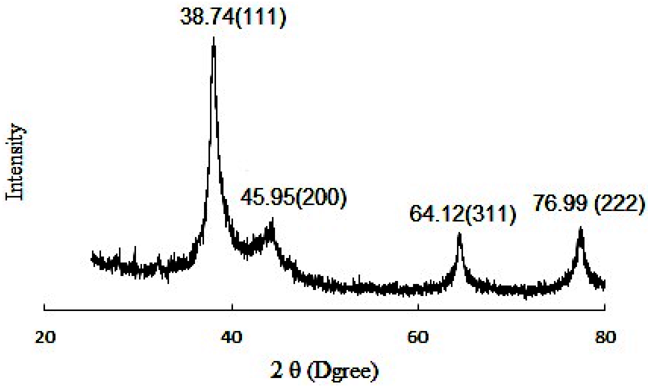

Fig. 2. XRD pattern of synthesized silver nanoparticles

Analysis through energy dispersive X-ray (EDX) spectrometers confirmed the formation of elemental silver (Fig. 3). Identification lines for the major emission energies for silver (Ag) are displayed and these correspond with peaks in the spectrum, thus confirming that silver has been correctly identified with 68.2 %.

The morphology and size of silver nanoparticles was analyzed by SEM. SEM image had shown individual silver nanoparticles (Fig. 4). It shows the particles are predominantly cubic in shape. The SEM image shows the size of the silver nanoparticles ranging from 20-50 nm.

Conclusion

In this study, silver nanoparticles were synthesized using aqueous extract of *Citrur limon* which belong to Rutaceae family instead of hazardous chemical reducing agent. This

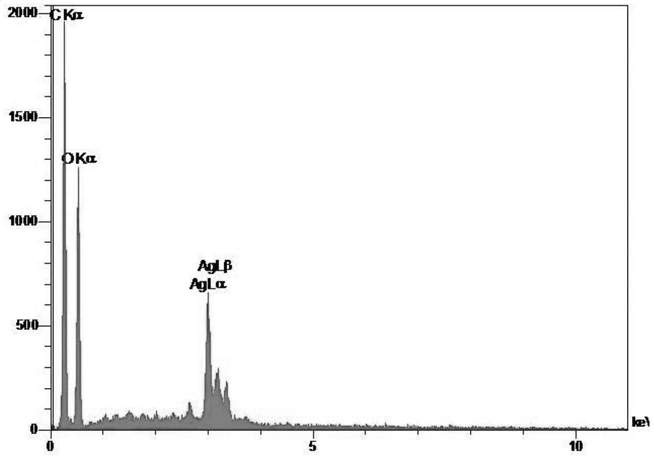

Fig. 3. EDX spectrum of silver nanoparticles

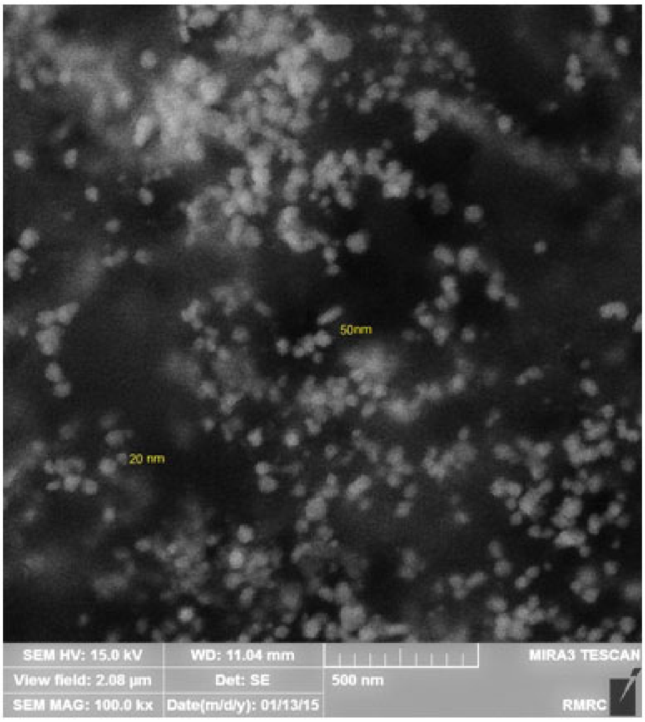

Fig. 4. SEM image of phytochemical synthesized silver nanoparticles

method is an eco-friendly and low cost way to fabrication of silver nanoparticles. The synthesized silver nanoparticles were characterized by analytical instruments such as UV-visible, FT-IR, XRD, EDX and SEM. The obtained data were revealed that these nano-particles are in face center cubic (fcc) lattice system with the average size between 20-50 nm.

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