

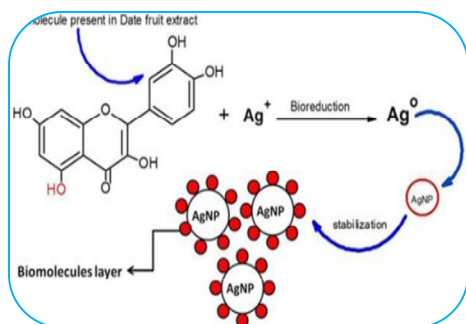


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silver nanoparticles from peel extract of *Citrus Sp.* and characterization biosyn the sized silver nanoparticle. In our study silver nanoparticles were synthesized with peel extract of *Citrus Sp.* fruit and aqueous solution of 1 mM silver nitrate solution and then characterized using UV-Visible, Fourier transform infra red spectroscopy (FTIR), X-ray diffraction (XRD), and Scanning electron microscopic (SEM) methods. Absorption maxima for silver colloidal solution showed at 420 nm in a UV-visible spectrum. The functional biomolecules such as carboxyl groups responsible for the silver nanoparticles formation were characterized by FT-IR. The x-ray diffraction results revealed that the crystallization of the bio-organic phase occurs on the surface of the silver nanoparticles or vice versa. The broadening of peaks in the x-ray diffraction patterns was ascribed to particle size effects. In our study, presence of elemental silver was proven by EDX analysis and x-ray diffraction evidenced that the silver ions had been reduced to elemental silver. In conclusion, our study illustrates the simple benign, cost-effective biosynthesis methods of silver nanoparticles using peel extract of *Citrus Sp.* fruits. Our findings could be explored for industrial production of nanoparticles and their use in biomedical and pharmacy applications.

Keywords : *Citrus Sp.*, Silver nanoparticles, Peel extract, Characterization.

INTRODUCTION

In the past decade there has been a marked increase in the field of fabrication of nanoparticles with controlled morphologies and remarkable features making it an extensive area of research. The synthesis of nanoparticles with control over particle size,

shape and crystalline nature has been one of the main objectives in chemistry that could be used for potential applications, such as bio-medical, biosensor, catalyst for bacterial biotoxin elimination and lower cost electrode.^{1,2,3} Different synthetic methods have been employed for the preparation of nanoparticles with diverse morphology and size. Although these methods have resulted in superior nanoparticles but still a

key understanding of improved manufacturing process is required which could be exploited at the industrial and commercial level to have better built, long lasting, cleaner, safer and smarter products such as home appliances, communication technology, medicines, transportation, agriculture and industries. Therefore, the main focus is to design nanoparticles using environmentally benign approaches. These provide

BIOSYNTHESIS AND CHARACTERIZATION OF SILVER NANOPARTICLES USING *CITRUS SP.* PEEL EXTRACTS

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ABSTRACT

Nanoparticles are prepared by using variety of hazardous chemical methods which are not environmentally friendly. Hence, the present study was designed to synthesize

solutions to growing challenges related to environmental issues.

Nature has provided ways and insight into the synthesis of advanced nanomaterials. It has now been reported in the literature that biological systems can act as the 'bio-laboratory' for the production of pure metal and metal oxide particles at the nano meter scale using biomimetic approach. Various microorganisms, such as bacteria^{4,5}, fungi^{6,7}, yeast⁸, plant extracts⁹ and waste materials,¹⁰ have acted as eco-friendly precursors for the synthesis of nano particles with potential applications. With these regards the present study was conducted with the objective to synthesize silver nanoparticles from peel extract of *Citrus Sp.* and characterize biosynthesized silver nanoparticle.

MATERIALS AND METHODS

Collection of Plant Material and Chemicals

The fruits of *Citrus Sp.* were collected from agricultural field located at Mysore, India. All the chemicals were obtained from Sigma-Aldrich. All the experiments were done in triplicates. Double distilled water was used for the experiments.

Preparation of Peel Extract

Fresh peels of *Citrus Sp.* fruit was washed thoroughly with double distilled water and incised into small pieces. Finely cut peels (20 g) were weighed and transferred to 500 mL flask containing 100 mL of deionized water, mixed well, and boiled for 5 min. The extract obtained was filtered through Whatman No. 1 filter paper and the filtrate was collected in 250 mL Erlenmeyer flask and stored at 4°C for further use.

Synthesis of Silver Nanoparticles Using the Peel Extract

To synthesize silver nanoparticles, 5 mL of peels of *Citrus Sp.* fruit was mixed with 50 mL of an aqueous solution of silver nitrate (AgNO_3) (1 mM) and stirred for 10 min at 30°C. Reduction occurs rapidly as indicated by a reddish brown color after 40 mins, indicating the formation of the silver nanoparticles. The silver nanoparticles obtained were purified by centrifuging at 10,000 rpm for 20 mins. And dispersing the pellets obtained in deionized water three times to remove water solubles.

Characterization of Silver nanoparticles

UV-Vis spectral analysis: The reduction of pure Ag^+ ions was monitored by measuring the UV-Vis spectrum of the reaction medium after 30 min. A small aliquot of the sample was taken for UV-Vis spectrum analysis (350-750 nm). The maximum absorbance spectrum of Ag-Ag nanoparticles was observed at 455 nm.

Fourier Transform Infra Red Spectroscopy (FT-IR): FT-IR measurements were carried out using Perkin (8300 FT-IR Shimadzu) spectrophoto meter, the range from 4000 cm^{-1} to 400 cm^{-1} . The silver nanoparticles pellet obtained using peel extract of *Citrus Sp.* fruits was air dried. The dried nanoparticles were mixed with potassium bromide (KBr) to make thin pellets and were used for FT-IR analysis in transmittance mode.

X-Ray Diffraction (XRD) Analysis: Resulting solution of the developed nanoparticles of silver was centrifuged at 10,000 rpm for 30 min. The solid residues of silver nanoparticles were washed twice with double distilled water and then dried at 80°C to obtain powder of silver nanoparticles used for X-ray diffraction measurements. The X-ray diffraction (XRD) patterns were recorded on (Shimadzu XRD-6000) with copper radiation ($\text{Cu K}\alpha$ 1.5406 Å) at 40 kV and 30 mA.

SEM Analysis of Silver Nanoparticles: Scanning electron microscopic (SEM) analysis was done using (Inspect S 50) SEM machine. Thin films of the sample were prepared on a carbon coated copper grid by just dropping a very small amount of the sample on the grid.

RESULTS AND DISCUSSION

Nanoparticles are prepared by using variety of hazardous chemical methods which are not environmentally friendly. Our study reported, convenient and extra cellular method for the synthesis of

silver nanoparticles by reducing silver nitrate with the help of peel extracts of *Citrus Sp.* The nanoparticles were characterized using UV-Visible, Fourier transform infra red spectroscopy (FTIR), X-ray diffraction (XRD), and Scanning electron microscopic (SEM) methods.

The formation of metal nanoparticles by reduction of the aqueous metal ions during exposure of peel extract of *Citrus Sp.* fruits was followed by UV-Vis spectroscopy (UV- Shimadzu spectrophotometer). The surface plasmon resonance peaks in absorption spectra for silver colloidal solution showed an absorption peak at 420 nm in a UV-visible spectrum, suggesting that the nanoparticles were dispersed in the aqueous solution with no evidence for aggregation in UV-Vis absorption spectrum. (Fig. 1)

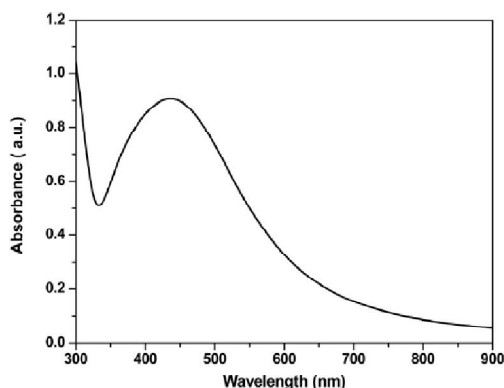


Fig. 1: UV-Vis Spectra of silver nanoparticles synthesized with peel extracts of *Citrus Sp.* and 1 mM silver nitrate solution

The results of FTIR analysis of our study shown different stretches of bonds shown at different peaks; 3327.21—N—H stretch, 1641.42 —C=C, and 1211.30—C=O. Peaks near 3440 cm^{-1} , and 2968 cm^{-1} assigned to OH stretching and aldehydic C—H stretching, respectively.¹¹ The weaker band at 629 cm^{-1} corresponds to amide I arising due to carbonyl stretch in proteins. The peak at 1051 cm^{-1} corresponds to C—N stretching vibration of the amine. The peak near 1743 cm^{-1} corresponds to C=C stretching (non conjugated). The peak near 866 cm^{-1} assigned to C=CH₂ and the peaks near 678 cm^{-1} and 638 cm^{-1} assigned to CH out of plane bending vibrations are substituted ethylene systems —CH=CH. FTIR spectra of silver nanoparticles exhibited prominent peaks at 1641, and 1382 cm^{-1} . The spectra showed sharp and strong absorption band at 1641 cm^{-1} assigned to the stretching vibration of (NH) C=O group. (Fig. 2)

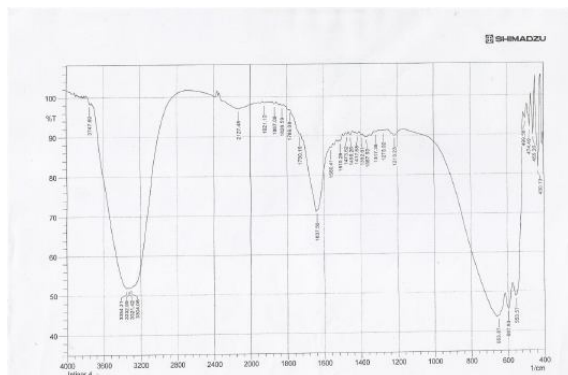


Fig. 2: Fourier transform infra red (FTIR) spectroscopy image of silver nanoparticles synthesized with peel extracts of *Citrus Sp.* and 1 mM silver nitrate solution.

The X-ray diffraction patterns revealed that all the reflections correspond to pure silver metal with face centered cubic symmetry. The reflections were indexed as (111), (200) and (220) with the corresponding 2θ values of 39.128, 43.315 and 63.468 respectively. The intensity of peaks reflected the high degree of crystallinity of the silver nanoparticles. (Fig. 3)

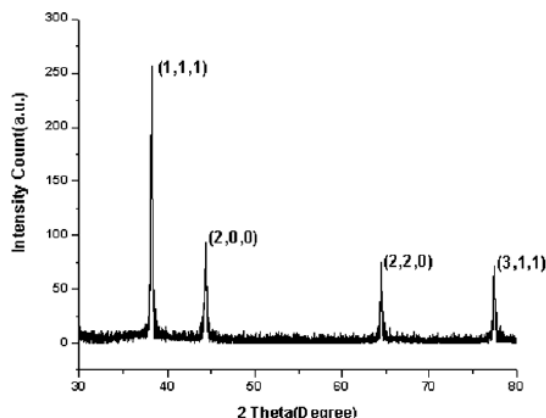


Fig.3: X-ray diffraction of Silver nanoparticles synthesized with peel extracts of *Citrus Sp.* and 1 mM silver nitrate solution.

The scanning electron micrographic study of silver nanoparticles synthesized using peel extracts of *Citrus Sp.* revealed that silver nanoparticles seem to be spherical in morphology and particles form cluster. It is easy to notice that the examined particles consist of a number of smaller objects of a few micro meters in size. (Fig. 4) The EDX spectrum of biosynthesized silver nanoparticles revealed that the middle part of the spectrum shown a strong peak located at 3 kV. This maxima is directly related to the silver characteristic. The maxima located on the left part of the spectrum at 0.2 kV clearly comes from carbon. (Fig. 5) Quantitative analysis proved high silver contents (100%) in the examined samples. (Table 1).

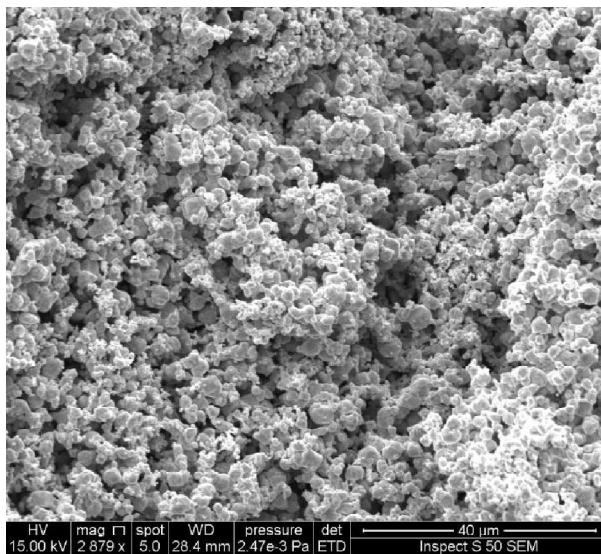


Fig. 4: SEM micrographs of silver nanoparticles synthesized with peel extracts of *Citrus Sp.* and 1 mM silver nitrate solution.

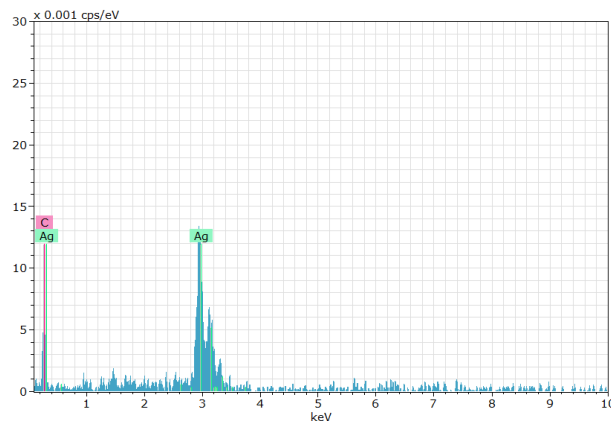


Fig.5: EDX characteristic spectrum obtained for silver powder synthesized with peel extracts of *Citrus Sp.* and 1 mM silver nitrate solution.

Table 1: Elements in Biosyn the sized Silver Nanoparticles

| Element | AN | Series | [Wt.%] | [Norm. wt.%] | [Norm. at.%] |
|---------|----|----------|----------|--------------|--------------|
| Carbon | 6 | K-series | 0 | 0 | 0 |
| Silver | 47 | L-series | 74.13765 | 100 | 100 |
| | | Sum | 74.13765 | 100 | 100 |

Metallic silver nanoparticles generally show typical absorption peak approximately at 3 keV due to surface Plasmon resonance¹² and some weak signals in spectra were due to atoms of molecules attached to the nanoparticles surface. The other signal obtained at 8 keV was derived from the CuTEM support grids.¹³ All the reflections in the X-ray diffraction pattern corresponds to pure silver metal with face centered cubic symmetry. The reflections were indexed as (111), (200) and (220) with the corresponding 2θ values of 39.128, 43.315 and 63.468 respectively. The intensity of peaks reflected the high degree of crystallinity of the silver nanoparticles. However, the diffraction peaks were broad indicating that the crystallite size is very small. Similar character is that peaks have been reported by other researchers.^{14,15,16} In our study, presence of elemental silver was proven by EDX analysis and XRD results showing that silver ions had been reduced to elemental silver.

CONCLUSION

In conclusion, our study illustrates the simple benign, cost-effective biosynthesis methods of silver nanoparticles using peel extract of *Citrus Sp.* fruits. Our findings could be explored for industrial production of nanoparticles and their use in biomedical and pharmacy applications.

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