



Green Synthesis of Silver Nanoparticles from *Solanum xanthocarpum*: Synthesis and Characterization

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Abstract

Green synthesis is now considered as an alternative to chemical and physical synthetic procedures for nanoparticles by using sustainable and eco-friendly materials instead of harsh and toxic chemicals. The aim of the present study describes a cost effective and environment safe technique for green synthesis of silver nanoparticles by using the *Solanum xanthocarpum* leaf extract as reducing agent. Further, characterization such as UV-Visible Spectrophotometer, FTIR and XRD analysis were carried out for the synthesised silver nanoparticles. The synthesised silver nanoparticles have maximum absorption at 410nm with the average size of 05 to 42nm. The FTIR data showed prominent peaks in 3445.05, 2924.34, 1637.35, 1384.25, 1109.60 and 669.74. The XRD data showed 2 intense values with various degrees such as 10.239°, 27.3969° and 35.7712°. It could be concluded that the biosynthesis of silver nanoparticles with leaf aqueous extracts of *S. xanthocarpum* provides potential source for the preparation of pharmacologically useful drugs.

Keywords: *Solanum xanthocarpum*, Silver nanoparticles, FTIR, XRD

Introduction

Nanoparticles have multifunctional properties and very interesting applications in various fields such as medicine, nutrition and energy (Chandran *et al.*, 2006). Nanoparticles have created remarkable advantages in the pharmacological industry to cure various bacterial and viral diseases (Song and Kim, 2009). Among the all noble metal nanoparticles, silver nanoparticles (AgNPs) are an arch product from the field of nanotechnology because of their unique properties such as chemical stability, catalytic, antibacterial, anti-viral, antifungal and anti-inflammatory activities (Ahmad *et al.*, 2015).

A number of techniques are available for the synthesis of silver nanoparticles like ion sputtering, chemical

reduction, and sol gel etc., unfortunately many of the nanoparticles syntheses methods involve the use of hazardous chemicals or high energy requirements, which are rather difficult and including wasteful purification (Ahmed *et al.*, 2015). Green synthesis is now considered as an alternative to chemical and physical synthetic procedures for nanoparticles by using sustainable and eco-friendly materials instead of harsh and toxic chemicals. The rich biodiversity and easy availability of plant entities have been highly explored for the nanomaterials synthesis (Monda *et al.*, 2011). Plant crude extract contains novel secondary metabolites such as phenolic acids, flavonoids, alkaloids and terpenoids. These compounds are mainly responsible for the reduction of

ionic into bulk metallic nanoparticles formation (Aromal and Philip, 2012). Present study is the first for the green synthesis of silver nanoparticles by using *Solanum xanthocarpum* leaves.

Solanum xanthocarpum Schradt & Wendal belongs to Solanaceae family. This plant is rich in many phytoconstituents like alkaloids, carbohydrates, proteins, saponins, flavonoids, phenolic compounds, tannins, terpenoids and steroids. This plant is known for its medicinal benefits for times immemorial. Roots, stem, leaves, flowers and fruits are useful parts of this herb as Siddha medicinal herb (Shree Devi *et al.*, 2014). Various studies indicated that *S. xanthocarpum* possesses antiasthmatic (Khare, 1995), hepatoprotective (Verma *et al.*, 2015), antimicrobial, anthelmintic and DPPH radical scavenging activity (Chhajed *et al.*, 2018), antibacterial (Rana *et al.*, 2016), immunomodulation (Pandey *et al.*, 2018), anti-inflammatory, antiallergic, antianaphylactic and antitumor (Bhatt and Reddy, 2018), antithrombotic (Lugun *et al.*, 2018), Antiurolithiatic (Patel *et al.*, 2012), antidiabetic and insect repellent properties. In this study, the plant mediated synthesized AgNPs were characterized and studied in details with all of their properties significant to current science and prevailing technologies.

Materials and Methods

Preparation of plant extracts

Fresh leaves of *S. xanthocarpum* were collected from the Kalvai village, Tuticorin district and was authenticated by The flora of the Tamilnadu Carnatic (Mathew, 1983). The voucher specimen (Voucher No. VV-BOT-VOCC-1) was also maintained in the Department of Botany, V.O. Chidambaram College, Tuticorin, Tamil Nadu, India. The leaves were surface cleaned with running tap water to remove debris and other organic contents, followed by double distilled water and air dried at room temperature. 50g of fresh chopped leaf materials were added in a conical flask containing 100ml of double distilled water and boiled it for 20min. After cooling, conical flask was kept under stirring on orbital shaker for 2h for complete extraction. The aqueous extract was filtered through Whatman filter paper No. 1 and the filtrate was collected in a Erlenmeyer flask and was used for the reduction process of Ag^+ to Ag^0 (Banerjee *et al.*, 2014).

Green synthesis of silver nanoparticles

For the synthesis of silver nanoparticles, 5ml of plant extract was mixed with 95ml of 1mM aqueous silver nitrate solution. This setup was incubated in a dark chamber to minimize photo-activation of silver nitrate at room temperature. Reduction of Ag^+ to Ag^0 was confirmed by the colour change of solution from greenish yellow to red (Raja *et al.*, 2017). 50ml of colloidal silver nanoparticle suspension was stored in the refrigerator (4°C) for further studies like UV-Visible spectrophotometer spectral analysis. The remaining suspension was poured into a Petridis and kept at $80^\circ C \pm 2$ for 12h in the hot-air oven for drying. The dried sample was scraped for FTIR analysis.

Characterization of silver nanoparticles

To observe the optical property of biosynthesized silver nanoparticles, 1ml of the colloidal silver nanoparticle suspension was taken in a test tube and was diluted with 2ml of deionized water. Then the sample was scanned in UV-visible spectrophotometer (Shimadzu UV 1800 UV-VIS spectrophotometer) between wavelengths of 350 to 750nm (Das *et al.*, 2017).

Fourier-transform Infrared (FTIR) spectroscopic analysis of the dried silver nanoparticles was carried out by the potassium bromide (KBr) pellet method. 1mg of silver nanoparticles were mixed with 100mg of dry potassium bromide (1:100 ratio) and then the mixture was compressed into a pellet using hydraulic press (5000-10000 PSI). The compressed pellet was put into the sample holder and the FTIR (Systronics 166) spectra were recorded in the range of 400-4000 cm^{-1} . To alleviate the moisture content in the sample, a blank disc was put in the reference beam (Vijayabasker and Shiyamala, 2012).

The colloidal silver nanoparticle suspension stored in the refrigerator was centrifuged at 15000rpm for 10min. The supernatant was discarded and the pellet was retained. The pellet was re-dissolved in 10ml of de-ionized water. While preparing samples for X-Ray Diffraction (XRD) analysis, a thin film of sample (100 μ l) was applied on a glass slide and allowed to dry for 30min. The XRD pattern was recorded using X'Pert PROP Analytical-PW 3040/60 X-ray Diffractometer with operating voltage of 30kV at a 20mA current strength. The sample was subjected to

Cu K radiation with nickel monochromator in the 2 range of 20–80° (Inaba, 2008). The size of the silver nanoparticle was calculated by Debye–Scherrer equation (Cullity, 1978) as follows:

$$S = \frac{k\lambda}{\beta_{0.5} \cos\theta}$$

Where, S is the crystallite size of silver nanoparticle, k is the wavelength of the X-ray source (1.54056Å) used in XRD, $\beta_{0.5}$ is the full width at half maximum (FWHM) of the diffraction peak in radian, k is the Scherrer constant that varies from 0.9 to 1 and θ is the Bragg angle in radian.

Results and Discussion

In this present investigation, we developed an inexpensive, versatile and very reproducible method for the synthesis of silver nanoparticles using the plant extract of *S. xanthocarpum*. Aqueous solution of 1mM silver nitrate when mixed with extract of

S. xanthocarpum, a visible colour change from greenish yellow to brown was noted within 5min at room temperature. Reduction of silver ions exhibited brown colour in aqueous solution due to surface plasma vibration in silver nanoparticles. When the plant extract was added to aqueous solution of silver ion complex, the colour started to change from green to brown due to the reduction of silver ions (Shanker *et al.*, 2003).

The formation of silver nanoparticles was confirmed by UV-visible spectroscopy analysis. UV-visible spectrum analysis for the biosynthesized silver nanoparticles using the extracts of *S. xanthocarpum* show speak at 410nm (Fig.1). In an earlier study, characteristic and well defined surface plasmon resonance (SPR) band at 414nm obtained for the silver nanoparticles synthesized by using *Calliandra haematocephala* extract as reducing agent confirms the formation of silver nanoparticles (Raja *et al.*, 2017).

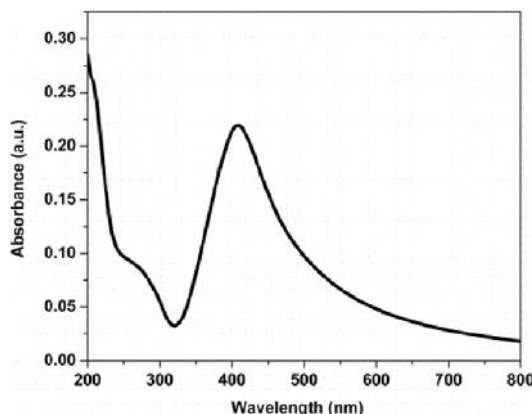
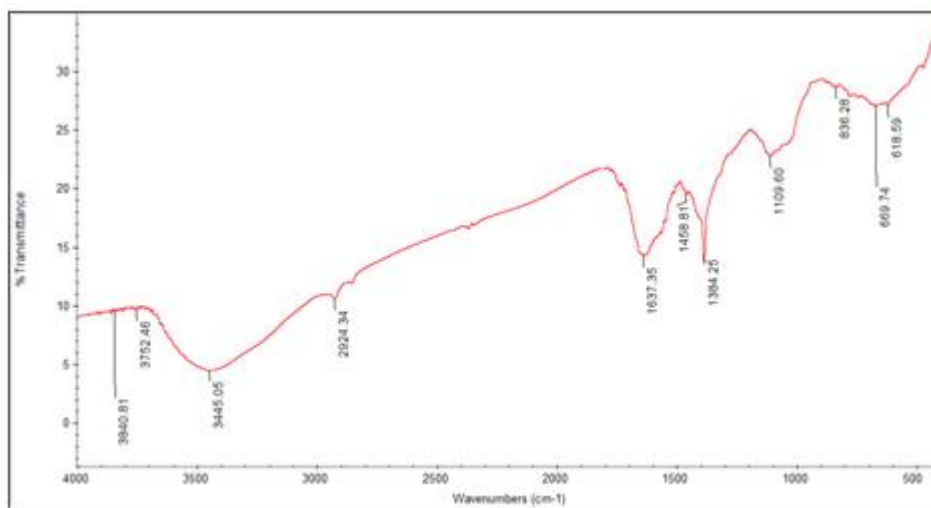


Fig. 1. UV-Vis absorption spectrum of *Solanum xanthocarpum* AgNPs

The FTIR spectroscopy analysis of *S. xanthocarpum* extract with 1mM silver nitrate solution shows peaks at 3445.05 cm^{-1} , 2924.34 cm^{-1} , 1637.35 cm^{-1} , 1458.81 cm^{-1} , 1384.25 cm^{-1} , 1109.60 cm^{-1} , 836.28 cm^{-1} , 669.74 cm^{-1} , and 618.59 cm^{-1} absorption peaks are

known to be associated with the stretching vibration for O-H stretch, C-H stretch, Alkenyl C=C stretch, Methyl C-H asym/sym. Bend, C-H plane bend, C-N stretch (aryl), C-H bend para and C-Cl stretch (Fig. 2 and Table 1).

Fig .2. FTIR spectrum of *Solanum xanthocarpum*Table 1 FTIR spectral qualities interpretation of the comparative shift in functional peaks of critical value (*Solanum xanthocarpum*)

Frequency range cm^{-1}	Compound type
3840.81	---
3752.46	---
3445.05	O-H stretch
2924.34	C-H stretch
1637.35	Alkenyl C=C stretch
1458.81	Methyl C-H asym/sym. bend
1384.25	C-H plane bend
1109.60	C-N stretch (aryl)
836.28	C-H bend para
669.74	C-Cl stretch
618.59	C-Cl stretch

The absorbance bands observed with *S. xanthocarpum* extract at around 3445.05cm^{-1} (amide I arising due to carbonyl stretch in proteins), suggest the presence of proteins on the surface of Ag-core particles, and plant proteins in the NPs shell. As plant molecules get absorbed onto the AgNPs surface, the amide groups intend to form stronger bonds with Ag atoms, which will break most of the H-bonds between the N-H groups and lead the narrowing and blue shifts of the amide bond. These results confirm the presence of possible proteins acting as the reducing and stabilizing agents (Sathyavani *et.al*, 2010).

X-ray diffraction is a method of determination of crystallinity of a compound. Here, to find the crystalline nature of the biosynthesized silver nanoparticles with *S. xanthocarpum* extract, the XRD analysis was done. The XRD pattern clearly showed that the plant extract mediated synthesized silver nanoparticles were crystalline in nature and the average size of nanoparticles was calculated as 19nm (Fig. 3 and Tables 2 & 3).

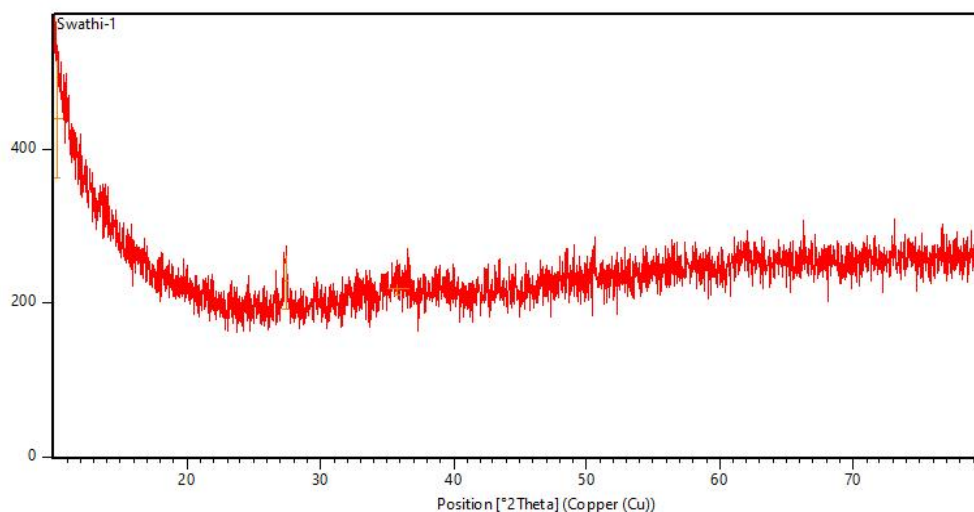


Fig.3. XRD analysis of biosynthesised silver nanoparticles using *Solanum xanthocarpum*

Table 2 Peak indexing from XRD Spectra (*Solanum xanthocarpum*)

2		Sin	Sin ²	$\frac{3 \times \text{Sin}^2\theta}{\text{Sin}^2\theta_{\text{min}}}$	h ² +k ² +l ²	h k l
10.239	5.1159	0.0892	0.0079	$\frac{3 \times 0.0079}{0.0079} = 3$	1 ² +1 ² +1 ²	1 1 1
27.3969	13.6984	0.2368	0.0561	$\frac{3 \times 0.0561}{0.0079} = 21$	4 ² +2 ² +1 ²	4 2 1
35.7712	17.8856	0.3071	0.0943	$\frac{3 \times 0.0943}{0.0079} = 35$	5 ² +3 ² +1 ²	5 3 1

The X-ray diffractogram of *S. xanthocarpum* extract mediated synthesized silver nanoparticles showed peaks in the whole spectrum of 2 ° values of 10.239°, 27.3969° and 35.7712°. The three distinct peaks at 2 ° = 10.239°, 27.3969° and 35.7712° were understood to be (1 1 1), (4 2 1) and (5 3 1) lattice planes respectively, to the face-centered cubic (fcc) structure

of metallic silver. This is in accordance with the standard metallic silver XRD pattern JCPDS No. 04-0873. The intense diffraction peak of (111) substantiated that the synthesized silver nanoparticles might be enriched with (111) facets (Raja et al., 2017). Non-appearance of other peaks confirmed the purity of silver nanoparticles used in the analysis.

Table 3 Particle size derived from XRD spectra (*Solanum xanthocarpum*)

S. No	h k l	2		FWHM (°)	(radian)	Size (nm)
1	1 1 1	10.2319	5.1159	0.8029	0.0140	10
2	4 2 1	27.3969	13.6984	0.2007	0.0034	42
3	5 3 1	35.7712	17.8856	1.6059	0.0279	05
					Mean	19

Conclusion

A plant-mediated, green method of synthesizing silver nanoparticles was successfully performed by employing the leaf extract of *S. xanthocarpum*. The synthesized nanoparticles were characterized by UV-Vis spectrophotometer, FTIR and XRD methods of analysis. These analysis confirmed the reduction of Ag⁺ ions to Ag⁰ which is supposed through the plant extract as capping agents i.e., the phytochemical constituents found in this plant are acting as the reducing agents. It could be concluded that the biosynthesis of silver nanoparticles with leaf aqueous extracts of *S. xanthocarpum* provides potential source for the preparation of pharmacologically useful drugs.

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