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A Green Chemistry Approach to the Synthesis and Characterization of Silver Nanoparticles

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

Traditional synthesis of nanoparticles employed only physical and chemical methods. More often the chemical synthesis methods employed in this production usually makes presence and absorbance of some toxic chemical substance on their surface and this may result in an adverse effect in several medical and other applications. No such adverse effects appear if they are biosynthesized using technology of green synthesis. This paper emphasizes on the synthesis and characterization of silver nanoparticles that can be applied in cleaning up of an environment such as an anti-biofouling compound synthesis by an environmentally friendly green synthesis technique. Aqueous extracts from the leaves of plant *S. siamea* were used as the reducing agents to produce the nanoparticles. UV-Visible spectroscopy monitored the synthesis of colloidal silver Nanoparticles

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and it showed a peak at 429 nm corresponding to the Plasmon absorbance of the silver nanoparticles. SEM analysis and FTIR and EDS XRD were employed to characterize other aspects like that of the size and shape.

Keywords: Green synthesis; SEM; S. siamea; nanoparticles; FTIR.

1. INTRODUCTION

Nanotechnology is believed to be the profound area of research in science related to modern material synthesis [1,2]. The nanoparticles possess completely improved properties with respect to various characteristics like Morphology, distribution as well as size etc. Although new technologies are evolving the various applications of these nanomaterials or nanoparticles, still there is requirement of technologies that synthesize these particles keeping in view the economic commercially viable ecofriendly process that enroute to the clean synthesis. The familiar methods employed for the production of silver nanoparticles include the following reduction in solutions [3] chemical and photochemical reactions in reverse micelles [4] thermal decomposition of silver compounds [5] Radiation assisted [6] electrochemical [7] sono-chemical [8] microwave assisted process [9] and recently via green chemistry route [10-20].

The use of environmentally benign materials like plant leaf extract [21] bacteria [22] fungi [23] and enzymes [24] for the synthesis of silver nanoparticles offers numerous benefits of eco-friendliness and compatibility for pharmaceutical and other biomedical applications as they do not use toxic chemicals for the synthesis protocol. Chemical synthesis methods lead to presence of some toxic chemical absorbed on the surface that may have an adverse effect in the medical applications.

Green synthesis is preferred over physical and chemical methods because it is ecofriendly, inexpensive, can be easily scaled up for mass production and also this method does not use high pressure, temperature, energy, and toxic chemicals. Several studies related to medical and industrial process have reported long back that silver has an inhibitory effect on microorganisms [25,26]. The current paper has chosen the plant *S. siamea* as a reducing agent to form silver nanoparticles. This plant is available in abundance and also possess high medicinal values [27].

2. MATERIALS AND METHODS

2.1 Selection of Plant Material and Preparation of Aqueous Extract

The collection of the plant material was from the Andhra University Campus, its identification as well as authentication as *Senna siamea* (*Lam*) was by Dr. Padal Faculty of Department of Botany, A.U. Visakhapatnam. The herbarium number assigned to it was A.U. (B.D.H) No. 22285.

The leaves of *S. siamea* plant were utilized to prepare the aqueous extract. About 5g of these leaves were washed gently using distilled water, they were cut into fine pieces and then subject to boiling in 100 ml of sterile distilled water. This was followed by filtration using Whatman No.1 filter paper.

2.2 Production of Silver Nanoparticles

In an attempt to synthesize silver nanoparticles, 5mM aqueous solution of $AgNO_3$was prepared. To a 90 ml of aqueous solution of 5 mM silver nitrate about10 ml of *S. siamea* leaf extract was added in order to facilitate the reduction into Ag^+ ions and was then placed at room temperature for about 4 hours.

2.3 UV-Visible Spectroscopy Analysis

UV-Visible spectrum of the reaction medium was measured at 3 hours after performing the dilution of little aliquot of the sample into distilled water to monitor the reduction of pure Ag^+ ions. This was performed using UV-Visible spectrophotometer.

2.4 XRD Analysis

XRD technique is employed to analyze the structure and size of the crystals. Siemens D5000 diffractometer at the Advanced Analytical Laboratory of Andhra University was preferred to carry out the XRD analysis. Also, this XRD analysis gave much more information relating to the size of the particle size and crystal structure.

2.5 SEM-EDS

A carbon coated copper grid was used to drop a minute of the sample on this grid which facilitated the dispersion of the silver nanoparticles. This was followed by air drying of the sample for some time, sample coated on the grid were subjected to attachment to SEM stubs using double-sided conductive tape and sputter-coated with gold and subjected to SEM analysis. An energy dispersive analysis of X-ray spectroscopy (EDS) coupled to the Scanning Electron Microscope was utilized for analyzing the elemental composition of the sample.

2.6 FTIR Analysis

Nicolet 6700 FT-IR, Thermo Scientific spectrophotometer was employed to record the Fourier transform infrared (FTIR) spectrum of the sample. By creating a KBr pellet containing AgNPs, the FTIR spectra varied from 4000 to 500 cm^{-1} at a resolution of 4 cm^{-1}.

2.7 Characterization of Nanoparticles

The average crystallite size was calculated and the crystal phases were identified using X-ray diffraction (XRD). A PANalytical PW 3040/60 X'Pert PRO device was used to detect diffraction patterns using Ni-filtered Cu-Kα radiation with a wavelength of 1.54056A^0. 40 mA of emission current and 45 kV of accelerating voltage were used. A scintillation counter detector was employed, along with a 2θ scan range of 10 to $80^0$and a scanning step size of 0.01^0. Philips X'Pert high score plus proprietary software was used for curve fitting and integration. Using Bragg's law, the Ag NPs' size was determined based on the PXRD peak positions.

On a copper grid coated with carbon, thin films of the sample were created by dropping the sample onto the grid for one minute. The Quanta 200 FEG-SEM machine (TEQIP, Centre for Nanotechnology A.U.) was used to measure the film on the FE-SEM grid after it had been dried for five minutes under a mercury lamp.

EDS confirmed the presence of elemental Silver. TheTEQIP, AUCEN, (JOEL Model JSM-7100F) were employed to carry out the EDS observations. The EDS spectrum captured in spot profile mode from a location on the film's surface where silver nanoparticles are densely concentrated. Quanta 200 FEG was used to analyze the nano crystallites.

The aqueous solution of the samples of silver nanoparticles (AgNPs) was subjected to centrifugation at 10,000 rpm for 30min. The obtained pellet was lyophilized and used to carry out the FT-IR analysis by KBR pellet (FT-IR grade) method and the recording of the spectrum was performed between the spectral range of 4000~500cm^{-1}(Thermo Nicolet, Avatar370).

Fourier transform infrared spectroscopy (FTIR) was used to analyze the interaction between *S. siamea* extract and silver nanoparticles in the diffuse reflectance moderate solution of 4 cm^{-1} in the KBr pellets. The spectra were obtained in the wavelength interval of 4000 to 500nm^{-1}. The biomolecules that cause the reduction of Ag+ ions and capping of the bio-reduced AgNPs produced by acacia extract were identified using FTIR measurements. The acacia filtrate was combined with KBr powder for a comparison analysis, dried, and then pelletized before being measured.

3. RESULTS AND DISCUSSION

The various plants often used to produce silver nanoparticles were *Pelargonium graveolens* [28] *Medicago sativa* [29] *Azadirachta indica* [30] *Lemon grass* [31] *Aloe vera* [32]. There is no evidence of silver nanoparticle synthesis using *S. siamea* as reducing agent to the best of our knowledge.

Silver nanoparticles exhibit a yellowish-brown color in aqueous solution which is a well-known process that occurs due to excitation of surface plasmon vibrations in silver nanoparticles [30]. The reduction of the silver ion caused by the *S. siamea* leaf extract as it was combined with the silver ion complex's aqueous solution caused the extract to turn from watery to yellowish brown, signifying the creation of silver nanoparticles. It is widely acknowledged that controlled nanoparticles in aqueous solutions can be examined using UV–Vis spectroscopy [33]. The UV-Vis spectra obtained from the reaction media after four hours are displayed in Fig. 1. The absorbance peak at 420 nm in the absorption spectra of silver nanoparticles generated in the reaction media broadens with increasing particle dispersion.

XRD measurements in this investigation verified the successful production of silver nanoparticles. Additionally, energy dispersive spectroscopy (EDS), field emission-scanning electron microscopy (FE-SEM), X-ray powder diffraction

(XRD), and microstructure, morphology, and pore characteristics of the samples produced by this technique were assessed.

The distinctive peaks seen in the XRD image (Fig. 2) and the structural view under the scanning electron microscope (Fig. 3) further illustrated and validated the biosynthesized silver nanostructure created by using *S. siamea* leaf extract. Throughout the complete 2θ value range of 10 to 80, the XRD pattern revealed five strong peaks. The spectrum's five sharp peaks line up with reports of silver nanocrystals' Bragg's reflection in the literature [34]. It is discovered that the common pattern of green-synthesized AgNPs has an FCC structure by comparing JCPDS (file no: 89-3722). The Debye-Scherrer equation (Eq. 1) was used to estimate the average crystalline size of the silver nanoparticles.

$$D = 0.9\lambda/\beta \cos\theta.$$

By determining the width of (111) Bragg's reflection, the estimated average size of the particles synthesized was 25 nm with a size range of 20 to 50nm with the cubic and hexagonal shape.
The sample has a mixed phase (cubic and hexagonal) of silver nanoparticle formations, according to the normal XRD pattern. The FWHM of the peak that corresponds to the 111 plane was used to estimate the average particle size of this sample, which came out to be 25 nm. The growth of silver nanostructures was further validated by the SEM image displaying the high density of silver nanoparticles produced by the *S. siamea* leaf extract. Silver was strongly detected in the EDS spectrum (Fig. 4) obtained from silver nanoparticles.

The size, shape, and morphologies of produced silver nanoparticles have been characterized using scanning electron microscopy. Samples' SEM photos at various magnifications are displayed accordingly. The pictures clearly show that the silver nanoparticles' morphology is almost spherical, which is consistent with the UV-visible spectra's surface plasma resonance band's form [29]. In good agreement with the particle size determined from XRD analysis, the average particle size observed from the SEM images analysis is 25 nm.

Both the qualitative and quantitative status of the components that might be involved in the creation of nanoparticles is provided by EDS analysis. (Fig. 4) validates the generation of silver nanoparticles by displaying the elemental profile of synthesized nanoparticles made with leaf extracts from *S. siamea*. Higher counts at 3 keV are also seen in Fig. 4 as a result of silver nanoparticles. Because of surface plasmon resonance, metallic silver nanocrystals often exhibit an optical absorption peak about 3 keV [35]. The largest proportion of silver was found by elemental analysis of the silver nanoparticles depicted in the picture. According to reports, nanoparticles made with plant extracts are stable in solution for several months after synthesis because they are encased in a thin coating of organic material that caps the particles, which comes from the plant leaf extract [36].

The biomolecules for capping and effective stabilization of the metal nanoparticles produced by *S. siamea* leaf extract were identified using FTIR measurements. Fig. 5 displays the silver nanoparticles' FTIR spectrum. H-bonded alcohols and phenols with O-H stretching have a band at 3541 cm^{-1}. The signal at 2920 cm^{-1} is indicative of carboxylic acids with an O-H stretch. Primary amines with N-H bends are assigned at 1602 cm^{-1}. The aromatic amine group's C-N stretching is represented by the peak at 1388 cm-1, while the alcohols, carboxylic acids, ethers, and esters' C-N stretching is represented by the bands seen at 1186, 1244, and 1024 cm^{-1}.

As a result, proteins and metabolites like flavonoids—which have functional groups made of alcohols, ketones, aldehydes, and carboxylic acids—surrounded the created nanoparticles. The carbonyl groups in proteins and amino acid residues have a stronger ability to bind metal, as demonstrated by FTIR study analysis. This suggests that proteins may use metal nanoparticles (such as silver nanoparticle capping) to prevent agglomeration and stabilize the medium. This shows that biological molecules may have two roles in the aqueous medium: they may produce and stabilize silver nanoparticles [37]. Although the FTIR results show that the *Senna siamea* leaf extract contains proteins and flavonoids, it is possible that other bioorganic compounds exist in solution and contribute to the reduction of silver ions and the stabilization of the resulting nanoparticles that are formed by surface capping [38].

About 25 nm was discovered to be the average crystallite size, which is in excellent agreement with the average particle size recorded in (Table 1). Here, we report for the first time on the creation of silver nanoparticles using *Senna siamea* leaf extract to lower the silver ions in a silver nitrate solution (Lam).

Table 1. Crystallite size, lattice parameter, orientation and FWHM intense Peak green synthesized nanoparticles

2θ	Orientation	Size of the crystallite (nm)	Lattice Parameter (*a*) Å	FWHM of intense peak *β* (radians)
38.46	111	24	4.0894	0.00760
44.63	200	24	4.0880	0.00760
64.77	220	26	4.0871	0.00622
77.66	311	24	4.0861	0.00899
81.76	222	26	4.0911	0.00691

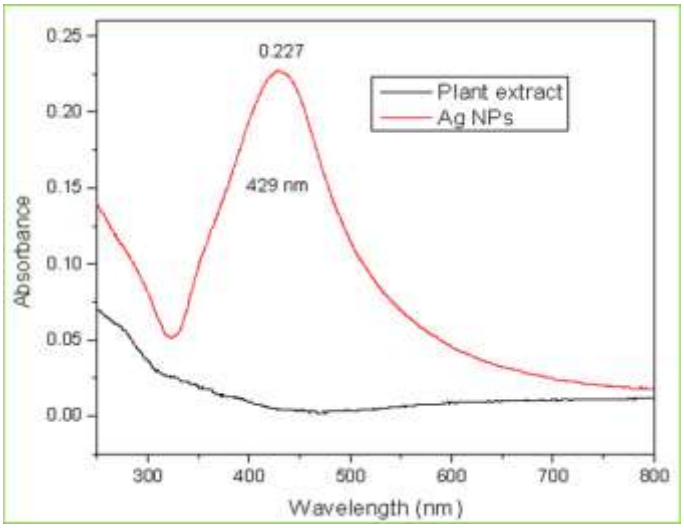


Fig. 1. UV-Visible Spectrophotometer only one broad peak was obtained at 429 nm, that corresponded to plasmon excitation of silver nanoparticles. Black line indicates plant extract and Red line indicates Ag NPs

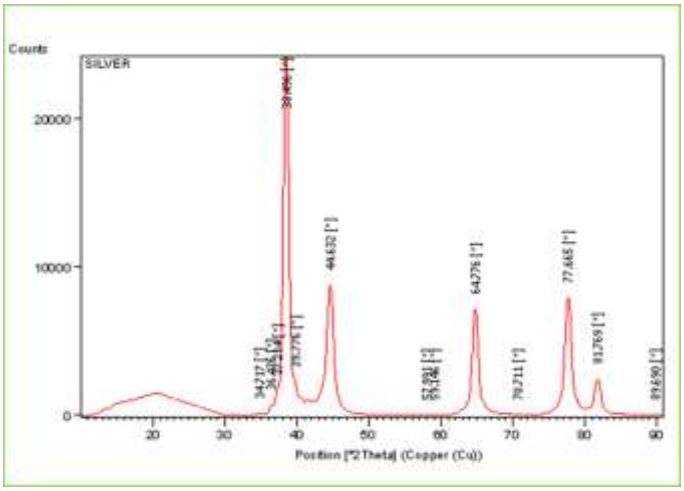


Fig. 2. The XRD patterns of vacuum-dried silver nanoparticles made with *Senna siamea* leaf extract are displayed in the figure. There are several Bragg reflections seen, which may be indexed to the (111), (200), (220), (311), and (222) facets of silver, respectively. The 2Θvalues of these reflections are 38.46^0, 44.63^0, 64.77^0, 77.66^0, and 81.76^0 sets of lattice planes. The XRD pattern therefore amply demonstrates the crystalline character of the silver nanoparticles produced in this current process

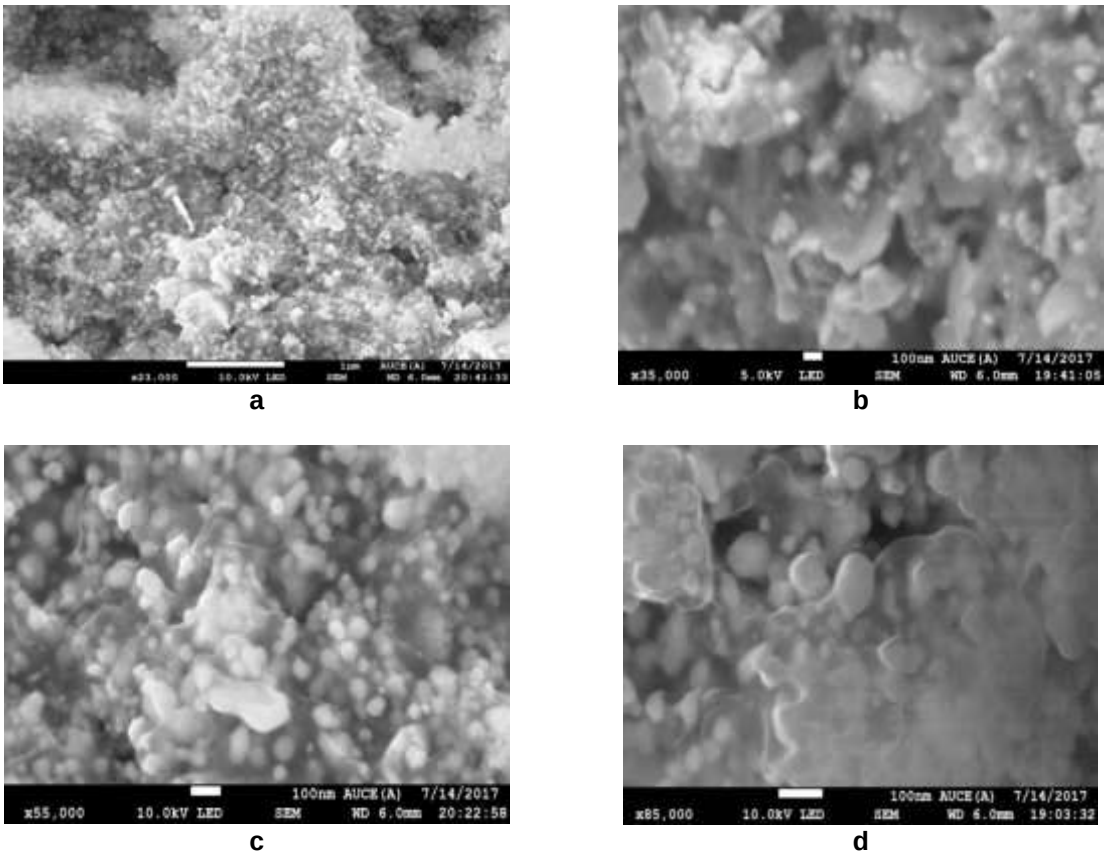


Fig. 3. SEM pictures of silver nanoparticles made using *S. siamea* leaf extract at various magnifications, magnifications of X23000, X35000, X55000, and X85000, in that order. The AgNps were found to have an aggregated form and a roughly spherical shape, as seen by the scanning electron microscopy. This indicates that the powder's particles are hexagonal and cubic in shape

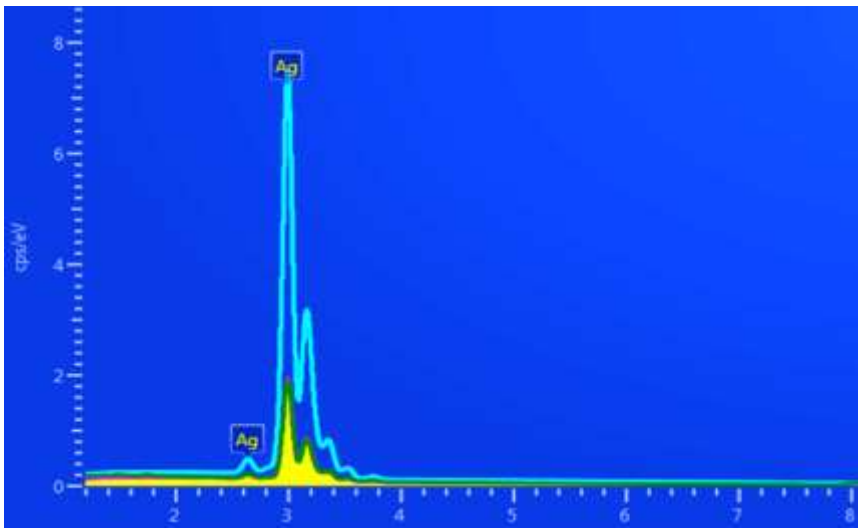
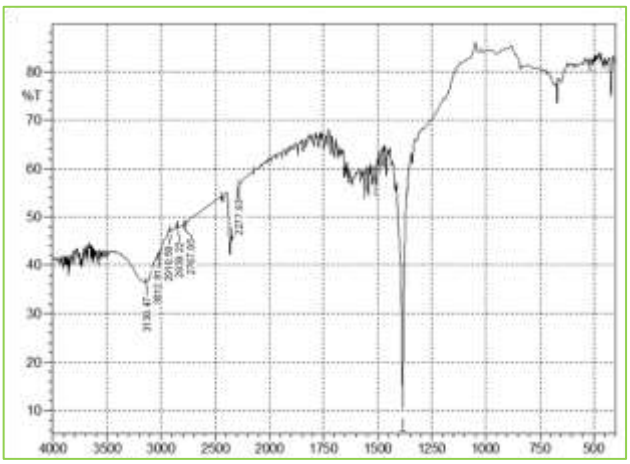
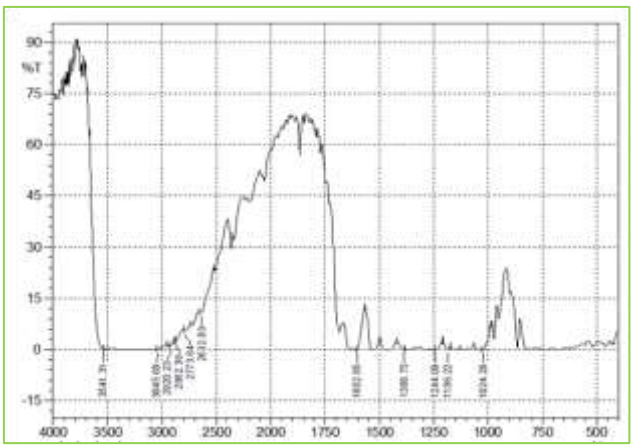


Fig. 4. The silver nanoparticles' energy dispersive X-ray spectra revealed a significant silver signal. Figure demonstrates the creation of silver nanoparticles and displays the elemental profile of synthesized nanoparticles made with leaf extracts from *S. siamea*. Because of the silver nanoparticles, Figure also displays increased counts at 3 keV



a



b

Fig. 5. FTIR spectra of (a) Capped Silver nanoparticles synthesized using *S. siamea* leaf extract. Figure shows the presence of silver with transmission peaks at3541, 3045, 2920, 2632, 1602, 1388 and 1186 cm^{-1} respectivelyand (b)plain *S. siamea* leaf extract with no significant peaks corresponding to silver

4. CONCLUSION

Plant extracts are used in the green chemistry synthetic pathway for the manufacture of silver nanoparticles. The findings demonstrated the production of significant amounts of silver nanoparticles (AgNPs), and UV-visible spectroscopy, SEM, FTIR, and EDS were used to confirm this. The antifouling activity of AgNPs against the larvae of the worldwide biofouling bacterium *Amphibalanus amphitrite* will be investigated further.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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