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Green Synthesis of Silver Nanoparticles Using *Solanum surattense Burm* f. Leaf Extract and Catalytic Activity on Methylene Blue

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RESEARCH ARTICLE

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ABSTRACT

The green synthesis process of silver nanoparticles (Ag NPs) by plant extract is simple, cost-effective, eco-friend and non-toxic. In the present investigation, the silver NPs were prepared from the leaf extract of *Solanum surattense Burm*. f. with different quantity. Ag NPs were attained within 1 h from the aqua solution of 1×10^{-3} M $AgNO_3$ and leaf extract. Green synthesized Nanoparticles (NPs) has been analysed by UV-visible spectrum confirmed the react of silver NPs. XRD indicated that the crystalline structure of the silver NPs, the functional groups are alcohols, alkenes, primary amines, proteins and aliphatic amine compounds of leaf extract which are involved in the reduction of Ag+ ions were identified from the FT-IR spectrum. FE-SEM obtained by surface morphology mostly spherical shape. The TEM analysis informed the particle size is spherical in nature with its size distribution between 16 nm-32 nm. Thus, synthesized Ag NPs showed good free radical scavenging and excellent dye degradation activity, which was analysed by the sunlight irradiation method from *S. surattense* leaf extract.

INTRODUCTION

Current scenario, nanotechnology has gained deep consideration in the due to its extensive application in diverse areas like medicine, catalysis, energy and materials. Individual NPs are building blocks in the nanotechnology field [1]. NPs are having great potential in medicinal and industrial applications due small size (10^{-9} m) with large covered surface area [2]. The preparation of metal nano domains are interesting attention in current research the valuable properties which make them useful for catalysis, sensor technology, and the biological labelling, opto-electronic device [3]. Generally, silver Ag nanoparticles have synthesized and stabilized by physico-chemical methods, however in this method is very complicated and expensive, not suitable for pharmaceutical applications because low yield and it is critically to prepare silver NPs with a well identified size. Therefore, new method needs to alternate the chemical and physical methods [4]. Ag NPs are applicable materials that have been studied widely, can be prepared by biological methods. In this method (biological methods), microbes and plants act as a reducing and capping agent [5]. Bio-synthesized Ag NPs possess particularly opto-electrical as well as biomedical properties and applied in catalysis, bio-sensing image, drug delivery, nano-device fabrication [6]. Many researchers have aimed at their notice to expand new, economical and most helpful drugs from plant sources based on nanotechnology [7]. Previously we have been published bio-synthesized Ag NPs using several plant extracts that the *Melia dubia* [8], *Croton sparsiflorus morong* [9], *Trichodesma indicum* [10], and *Leucas martinicensis* [11].

S. surattense (Solanaceae) is a perennial herb considered as one of the widely useful of traditional medicine around India. The medicinal plant used in pharmaceutical activities such as antibacterial, antifungal, antinociceptive, antioxidant, hypoglycaemic and larvicidal, respectively [12]. Phyto-chemical investigation of the *S. surattense* reported to have a number of alkaloids, flavonoids, glycosides, tannins, gums, resins, essential oils, fatty oils, carbon compounds, and hydrogen, oxygen and nitrogen salts of some chemicals [13]. These biomolecules induce the reduction of silver ions of $AgNO_3$ to Ag NPs. In the current scenario, nanotechnology has been prolonged to the wastewater treatments due to surface area Ag nanoparticles shows an enhanced reactivity [14].

Hence, the present work aims to create eco-friendly production of silver NPs by using *S. surattense* leaf extracts and also studied the photocatalytic degradation of methylene blue and the occurred of silver colloids.

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EXPERIMENTAL DETAILS

Materials

All the compound and reagents used in this synthesis were of AR grade Silver $AgNO_3$ (99.9%) was purchased from Sigma-Aldrich Chemicals. All glassware was washed in con. HNO_3 (30%) and rinsed directly with ultramile pure water previous to use and dried in a hot air oven. The leaf *S. surattense* leaves were collected from botanical garden Annamalai Nagar, Annamalai University, Cuddalore District, Tamilnadu.

Preparation of Leaf Extraction

The plant leaves were washed in flowing tap water and then thoroughly cleaned with doubly distilled water. 20 g of fresh leaves mixed in 100 mL sterile distilled water and boiled at 80°C for 20 min and after decreased temperature at room temperature, this solution was filtered with remove the precipitation and stored 4°C for further preparation.

Synthesis of Silver Nanoperticles

1×10^{-3} M of $AgNO_3$ has to be taken in an Erlen meyer flask containing 100 mL of distilled water. Three different concentration of a leaf extract (5.0 mL, 10.0 mL and 15.0 mL) are added individually then stored in brown bottles separately at atmosphere temperature. After 15 min the silver nanoparticle was formed, it was confirmed by the change of colour solution (yellow to dark brown).

Catalytic Activity

In the catalytic activity of bioproduct silver nanoparticles were studied by degrade of MB dye under the sunlight irradiation. Initially, the dye solution was ready to dissolving 1 mg of MB powder in 50 mL deionized water (keeping 10 mg/l concentration) and then Ag NPs (10 mgs) were added to dye solutions. The combination was stirred well for 50 min in darkness before exposing to sunlight. A control prepared and kept under the same condition for comparing any change in colour of the dye solution.

CHARACTERIZATIONS

The decrease of the silver ions by the supernatant of the analyse plant extracts in the solutions. The arrangement of silver NPs were analysed by UV-visible spectrum to visualize by sampling with the aqueous component (2.0 mL). The UV-vis spectra of these samples were detected on a UV-2450 (Model SHIMADZU UV-1800-resolution 2nm) spectrophotometer operated at wavelengths of 225 nm-800 nm. Distilled water was used to adjust the baseline. X-ray diffraction was performed on an X-ray diffractometer (XPERT-PRO) functioned at 40 kV and 30 mA. The patterns were recorded by CuKα radiation with λ of 1.5406 Å and nickel monochoromator. The scanning was done in the region of 2θ from 35° to 80°. For EDAX analysis, the decreased silver was dried on a C^{12} tape placed on a Cu stub and performed on a (JEOL JSM 6701-F) FFE-SEM equipped with an EDAX attachment. FT-IR spectra were recorded in the 4000 cm^{-1}-400 cm^{-1} region using FT-IR Spectrometer (SHIMADZU-8400 FT-IR). Morphology and size of silver NPs were evaluated using a TEM (TECHNAI10-Phliphs).

Mechanism of Silver NPs Formation

Ajitha, et al. [15] investigated that they in particular, flavonoids are highly reducing agents and have added to the decrease of silver ions to NPs. As flavonoids (poly-phenols) are more powerful reducing agents and also directly scavenge molecular species of active oxygen, these antioxidant activities of flavonoids give forth from their ability to give e^- or H atoms. The plausible mechanism of silver NPs formation may be recommended as the flavonoids are oxidized during the decrease of Ag^+ to Ag NPs. Therefore, reduction and capping processes by the biomolecules or atoms are presence in the leaf extract of *S. surattense* could be authentic for the extended stability **(Figure 1).**

RESULTS AND DISCUSSION

UV–Vis Spectra of Silver NPs

(Figure 2) shows the spectra of absorption of prepared silver particles with different quantity (5.0 mL, 10.0 mL and 15.0 mL) of *S. surattense* leaf extract. The absorption peak at about 464 nm confirms the formation of silver nano particles. As the increasing of leaf extract concentration, the rabidly increase in Plasmon resonance band intensity and blue shift in absorption edge were observed. The blue shift might be due to reduce in particle size caused by the high concentration of the leaf extract in the reaction mixture [16]. Surface Plasmon resonance patterns, characteristics of metal NPs strongly depend on particle size, stabilizing molecules or the surface adsorbed particles and the electric property of the medium [17].

The **Figure 2** shows the mixture of $AgNO_3$ and leaf extract, the initial colour of the reaction mixture changes on yellow to yellowish brown then after 1 hour at room temperature 30°C to change in the dark brown colour. The variation in colour may be occurred to the excitation of the surface Plasmon resonance in the metal NPs caused by the reduction of silver ions [18]. Silver NPs have free e^- give rise to SPR resonance absorption could be the combined vibration of e^- of the metal NPs in oscillate with the light wave [19]. The process is known as a surface Plasmon resonance peak. This resonance is based on the size and shape of the NPs

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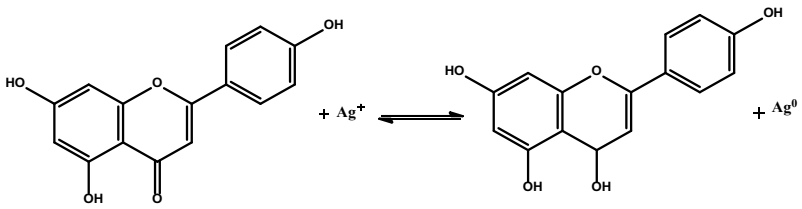

Figure 1: Mechanism of Ag NPs.

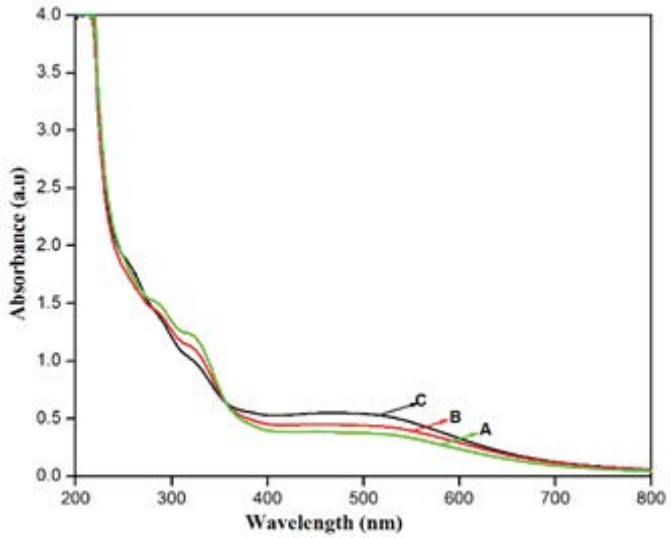

Figure 2: UV-vis spectra of Ag NPs synthesized using aqueous bark extracts of S. *surattense*.

formed. It was the absorption peak intensity of silver NPs increases and broadness of the peaks as decreases with increase with time. The mechanism formation of silver nano particles can be represented by (Scheme 1).

XRD Studies

The synthesized NPs were confirmed as face centered cubic structure of silver by X-ray Diffraction Profile, according to JCPDS card no (87-0719). **(Figure 3)** consists of XRD patterns of silver Particles prepared from 5 mL, 10 mL, 15 mL volumes of leaf extraction. That, it is found all the samples exhibit a diffraction peak occur at 38.14° and other weak peaks at 44.29°, 64.48° and 77.39° associate with the indices of (111), (200), (220) and (311) of silver powder, respectively. Moreover, the organic diffraction peaks related to leaf extract were observed. In all the sets of samples, the preferential orientation is only along (111) plane. With the change of leaf extract volumes in the reaction mixture, the change in peak intensities and the varied in FWHM of diffraction peaks are found which indicates that the rearrangement of crystallinity due to segregation of crystallite at grain boundaries. However, the preferential orientation of NPs remains unchanged.

The average crystallite size was determined using Scherr's formula

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

Where, 'D' is the crystallite size, 'λ' is the X-ray wavelength (1.5406 Å), 'β' is the Full Width at Half Maximum (FWHM) and 'θ' is the Bragg's angle.

The determined mean crystallite sizes of the silver NPs are given in **Table 1.** From the **Table 1**, it is observed mean crystallite size is decreased from 28 nm to 24 nm caused by the Ostwald ripping effect. The results are good in agreement with TEM results.

FT-IR analysis

The leaf extraction is responsible for the reduction of Ag^+ to AgNPs (Ag^o) to indicate the presence of functional groups by using FT-IR spectroscopy [20]. FT-IR reported as a suitable technique to analyse functional groups in synthesized various concentration of the silver NPs. Characteristics peaks of absorption of silver NPs were obtained at 3398 cm^{-1}-3429 cm^{-1}, 2924 cm^{-1}-2935 cm^{-1}, 2343 cm^{-1}-2349 cm^{-1}, 1610 cm^{-1}-1627 cm^{-1}, 1382 cm^{-1}, 1099 cm^{-1}-1085 cm^{-1} and 661 cm^{-1}-466 cm^{-1} **(Figures 4)**. The wide peaks in the region 3398 cm^{-1}-3429 cm^{-1} appeared due to O-H group and also the H-bonded alcohols and phenols [21]. The medium peak in the region 2924 cm^{-1}-2935 cm^{-1} might be due to a C-H group of alkenes. The medium bands at 1610 cm^{-1}-1627 cm^{-1} are due to N-H bending of primary amines [22]. The peak 1382 cm^{-1} is due to -C-N group of proteins [23]. The medium peaks in the region 1099 cm^{-1}-1085 cm^{-1} were assigned to the C-N group of aliphatic amines. From the FT-IR analysis, the occurrence of the functional group of alcohols, alkenes, primary amines, proteins and aliphatic amines in compound of the S. *surattense* leaf extracts. The FT-IR results suggest the proteins involved in capping and stabilization of the prepared Ag NPs.

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Table 1: Calculation the particle size of $AgNO_3$ of *S. surattense* by using Debye Scherer's equation.

Leaf concentration (mL)	2θ (degree)	d spacing (10^{-10} m)	FWHM (β) (radians)	Miller indices JCPDS	Particle size (D) (nm)
0.5	38.146	2.35728	0.31	[111]	28.1
1	38.08	2.03778	0.33	[111]	26.39
1.5	38.233	2.35215	0.35	[111]	24.89

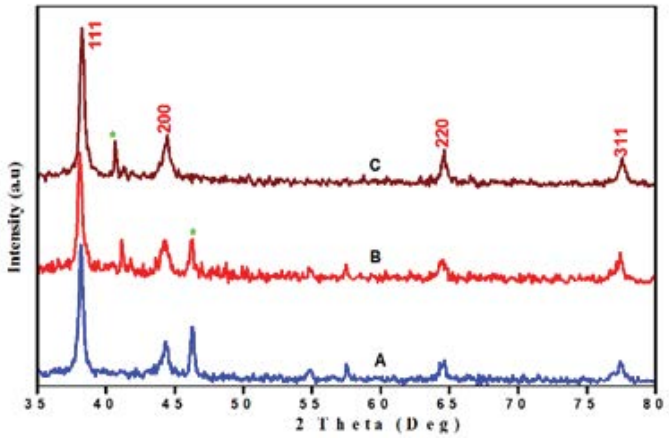

Figure 3: XRD results of Ag NPs.

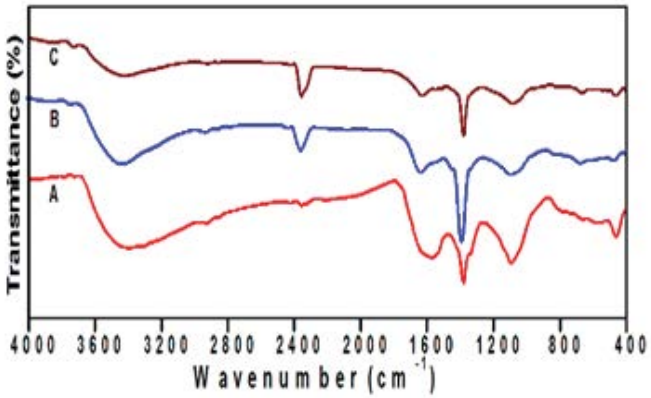

Figure 4: FT-IR spectra of green synthesis Ag NPs.

FE-SEM with EDX

(Figures 5a-5c) shows the SEM images of silver NPs prepared using various quantities of leaf extracts. From the SEM images it is found that the particles are shape of spherically and nano-sized in nature. Moreover, smaller grains are get-together and form large grains. The FE-SEM determines the surface morphology and size of particles. It was mentioned that the particles were mostly spherically in the shape. The element's presence and powder nature of silver NPs in the material were observed by EDAX analysis shown in **(Figure 5d)**. The presence of elemental composition of the silver NPs powder samples is sense using an SEM instrument with EDAX detector. The EDAX spectrum exposes that the strong signal of silver atoms. This result is exactly shows that the silver NPs reduced by *S. surattense* leaf extract.

HR-TEM Analysis

TEM gives that the information spherical shape of the green synthesized silver NPs. The particle size distributions of silver and *S. surattense* leaf extract the quantity of 15.0 mL is observed. The TEM image clearly explained that the mean particles size are mostly uniform size and shape is approximately 23 nm from the TEM diagram is noticed in **(Figures 6)**. This result is good in agreement with XRD and FE-SEM results.

Photocatalytic Activity of Ag NPs

A photocatalytic activity of the biosynthesized silver particles was investigated by the Methylene Blue (MB) degraded under the solar irradiation method. The analysing of absorption peak of MB dye was observed at 664 nm. The dye degradation in shows of bio-synthesized silver nano particles is visualized by rapidly change in deep blue to colourless. From, the degradation of dye was confirmed by the reduced in intensity of absorption peak at 664 nm within 3 h was observed. The control does not change in the intensity of peak was observed during the solar light exposure in time **(Figure 7a).** The percentage of dye degradation was determined by the following relation and its difference with exposure time is shown in **(Figure 7b)**.

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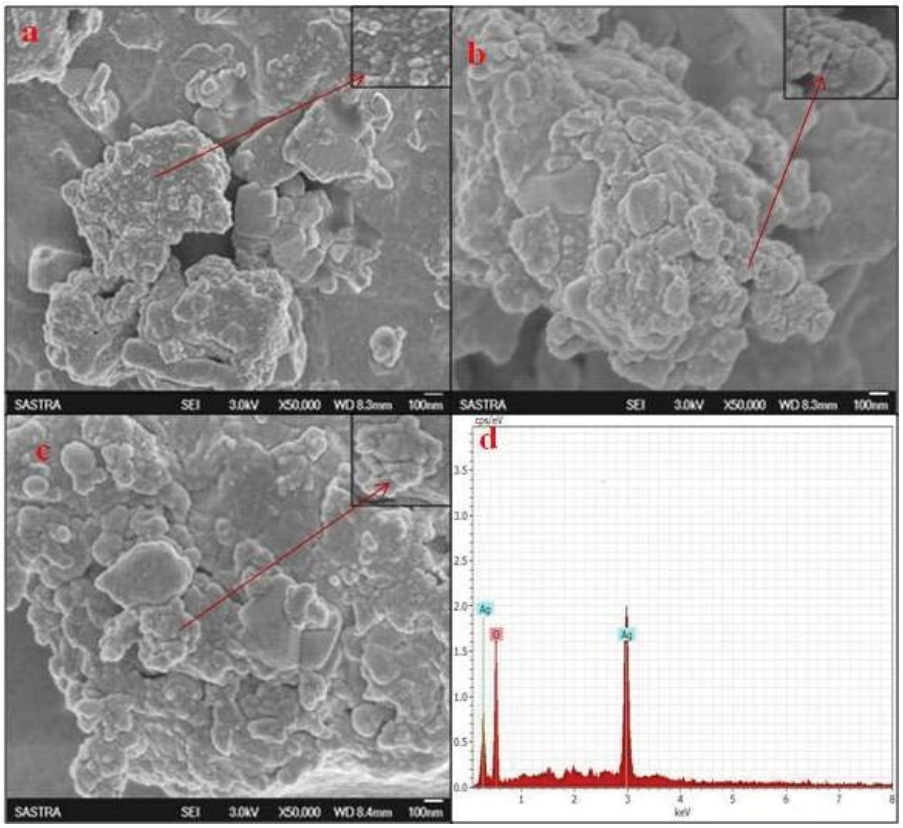

Figure 5: a-c): FE-SEM image; d): EDAX spectrum of green synthesis Ag NPs.

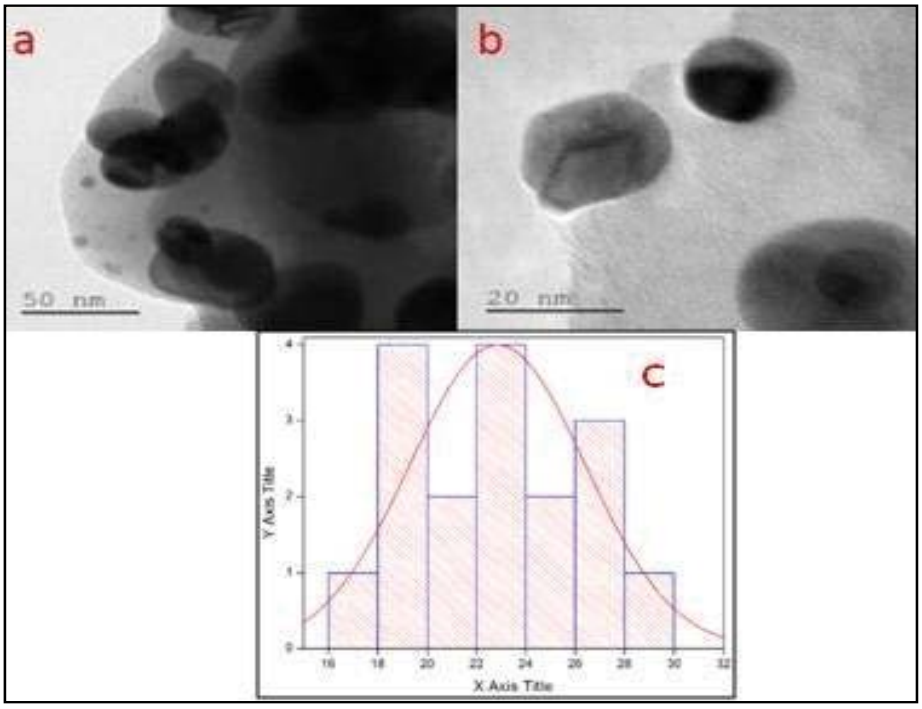

Figure 6: TEM image and histogram of green synthesis Ag NPs.

Dye degradation (%)=$[(C_o-C_t)/C_o] \times 100$

Where 'C_o' is the initial concentration of methylene blue solution and 'C_t' is the final concentration of the dye solution after 't' hours of exposure in solar irradiation. The results depict that the dye degradation of silver nano particles at different time interval of time 30 min-210 min is 31%-71%, respectively. The literature survey reveals that the photocatalytic activity can be highly dependent on the crystallographic morphology and size of the particle [24]. The green synthesized silver nano particles are stable and effective catalysts for the degradation of natural dyes under solar irradiation. The tiny nanoparticles are as evidence from both the XRD and TEM results.

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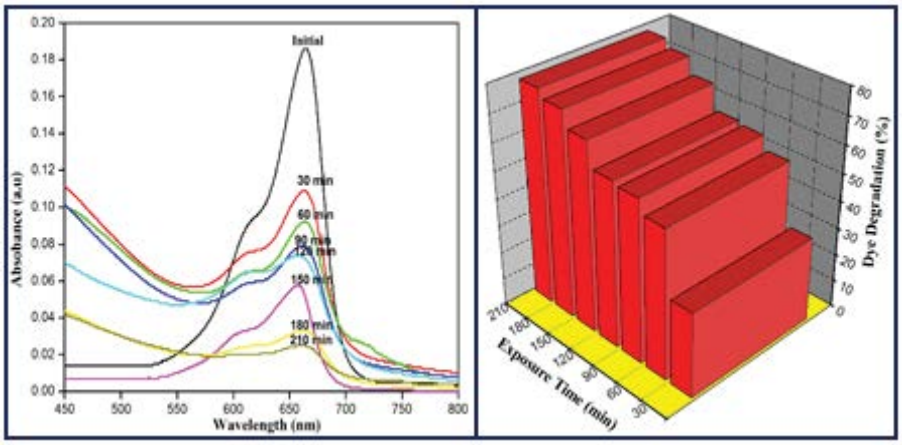

Figure 7: The absorption spectra of aqueous solution of methylene blue tested at different time intervals in the presence of; a): Ag NPs synthesized using *S. surattense;* b): The logarithm of the ration between the original concentration of dye and the concentration after Photocatalytic degradation *versus* corresponding irradiation time (min).

CONCLUSION

The reducing agent for preparing Ag NPs, *S. surattense* was used in green synthesis process. The prepared silver nano particles were analysed for their structural, surface morphological, FT-IR, TEM and catalytic activity of MB was investigated. From the structural studies, it is found that the prepared NPs are having perfect crystalline nature with (111) plane preferential orientation. FE-SEM images reveal is spherical shape in the grains of silver NPs. EDX result confirmed the presence of silver. The FT-IR studies confirm that the presence of protein and amine, it can be used as a decreasing and capping agent during the formation Ag NPs. Photocatalytic activity of the silver nano particle investigated degradation of methylene blue under solar light. The synthesized Ag NPs exhibit suitable photocatalytic activity.

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