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Characterization of Biologically Synthesized Silver Nanoparticles

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Abstract

Silver Nanoparticles (AgNPs) synthesized through biological methods have attracted a lot of attention due to their eco-friendly nature and potential applications in various fields. This paper comprehensively characterizes biologically synthesized silver nanoparticles using wet biofriendly synthesis. The bio-synthesized silver nanoparticles were characterized using Scanning Electron Microscopy with Energy-Dispersive X-ray Spectroscopy (SEM-EDS), Fourier-Transform Infrared Spectroscopy (FTIR), and UV-Visible-NIR Spectrophotometry. The SEM-EDS analysis confirms the morphological and elemental composition of the nanoparticles, while FTIR reveals the functional groups involved in their stabilization. While UV-Visible-NIR spectroscopy provides insights into their optical properties. The combination of these techniques offers valuable information about the structural, chemical, and optical characteristics of biologically synthesized silver nanoparticles, facilitating their potential applications in catalysis, sensors, water treatment, and biomedical fields.

***Key words*:** Silver nanoparticles, Biosynthesis, SEM-EDS, FTIR, UV-Visible-NIR Spectrophotometer

Silver nanoparticles (AgNPs) have garnered immense interest in recent years because of their distinct physicochemical characteristics and potential applications in various fields, including catalysis, sensing, imaging, and drug delivery [11]. These nanoparticles are produced through environmentally friendly methods that utilize natural sources like plants, microbes, and algae, which act as reducing and stabilizing agents in the synthesis process. The use of biological synthesis methods not only ensures sustainability but also leads to AgNPs with excellent biocompatibility and low cytotoxicity, making them highly desirable for biomedical applications. Biological synthesis methods offer sustainable and environmentally benign approaches for the production of nanoparticles, utilizing natural sources such as plants, microbes, and algae as reducing and stabilizing agents [6]. Biologically synthesized AgNPs have demonstrated excellent biocompatibility and low cytotoxicity, making them attractive candidates for biomedical applications [5]. However, to fully exploit the potential of biologically synthesized AgNPs, it is vital to characterize their structural, chemical, and optical properties accurately. Understanding the interactions of AgNPs with biological systems is essential for biomedical applications. Techniques such as cell viability assays, cellular uptake studies, and in vivo imaging can be employed to evaluate the biocompatibility, cellular uptake, and biodistribution of AgNPs. This paper presents a detailed characterization of biologically synthesized silver nanoparticles using a combination of spectroscopic, microscopic, and analytical techniques, including Scanning Electron Microscopy with Energy-Dispersive X-ray Spectroscopy (SEM-EDS), Fourier-Transform Infrared spectroscopy (FTIR), and UV-Visible-NIR spectrophotometry.

MATERIALS AND METHODS

Vetiveria zizanioides

Family	:	Poaceae
Subfamily	:	Panicoideae
Genus	:	Chrysopogon
Species	:	C. zizanoides
Common name	:	Vetiver or Khus

Vetiver root scientifically known as *Chrysopogon zizanioides*, is a perennial indigenous grass from India that is highly valued for its multifaceted uses and benefits. In traditional medicine systems vetiver root is prized for its therapeutic properties and anti-inflammatory, antiseptic, and cooling effects. It is useful for treating skin conditions like acne, eczema, and wounds [12]. Vetiver oil is also used in aromatherapy to alleviate stress, anxiety, and insomnia, promoting relaxation and mental well-being. Vetiver grass is highly effective in soil conservation and erosion control due to its deep and dense root system. The intricate network of vetiver roots. Assisting in the cohesion of soil particles, it lessens erosion by water runoff and wind. It is often planted along slopes, riverbanks, and other vulnerable areas to stabilize soil, prevent landslides, and protect agricultural land [9].

Bio-synthesis of silver nanoparticles

Silver nanoparticles were synthesized using a biological method based on reducing silver ions (Ag^+) by a plant extract. Vetiver roots were collected and washed thoroughly with distilled water to remove any surface contaminants. Vetiver roots were then dried, powdered, and sieved to get an equal-sized extract, which was used as a reducing and stabilizing

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agent in the synthesis process. 10 gm of Vetiver root powder was taken in a clean conical flask 100 ml of distilled water was added to, and heated at 80°C in a water bath for 10 minutes the obtained solution was filtered using Whatman filter paper no.42 and stored in a dark color bottle in refrigerator at 4°C for further use. The Vetiver root extract (VRE) will be designated as such. A solution of Silver Nitrate (1 mM AgNO3) was prepared in aqueous form. Combining 5 ml of VRE with 95 ml of 1mM AgNO3, this mixture underwent heating at 1500 rpm on a magnetic stirrer at 80oC for 30 minutes. The shift in color from light yellow to brown indicated the formation of silver nanoparticles, demonstrating the activation of the inherent surface plasmon resonance characteristic of AgNPs.[1],[2],[3]

Characterization of biosynthesized silver nanoparticles

The biologically synthesized silver nanoparticles were characterized using various spectroscopic, microscopic, and analytical techniques, including SEM-EDS, FTIR, and UV-Visible-NIR spectrophotometry [13].

Scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS)

The morphology, size, and elemental composition of the silver nanoparticles were analyzed using SEM-EDS in the ULTRA ZEISS 55 GEMINI SERIES. The nanoparticle samples were mounted onto aluminum stubs and sputter-coated with gold for 60 seconds to enhance conductivity. The samples were then analyzed under SEM at an accelerating voltage of 20 kV, and EDS analysis was performed to identify the elemental composition of the nanoparticles.

Fourier transform infrared spectroscopy (FTIR)

FTIR analysis was conducted using PERKIN ELMER FTIR SPECTROMETER FRONTIER to identify the functional groups involved in stabilizing silver nanoparticles. The thin layer of the sample was prepared on a 1cm × 1cm glass slide. The slide was coated with the material and subjected to annealing in a muffle furnace for 300°C for 3 hours. The prepared slide is placed in a sample holder and analyzed. The FTIR spectra of the nanoparticle powder were recorded in the range of 400-4000 Cm^{-1} [10]. FTIR (Fourier Transform Infrared) spectroscopy is a versatile analytical technique that offers valuable insights into the organic composition and molecular structure of materials. FTIR spectra are mainly used to identify functional groups and types of bonds (such as C=O, C-H, O-H, N-H, etc.) present in a sample. Each functional group absorbs infrared radiation at characteristic frequencies, which act as the fingerprint of the material, allowing for the identification of specific chemical groups within a molecule.

UV-visible-NIR spectrophotometer

The optical properties of the silver nanoparticles were analyzed using a UV-Visible-NIR spectrophotometer (Ocean optics USB 4000XR) in the 200-1000 nm wavelength range and Ocean optics DT-mini-2 as source meter respectively). The absorption and transmission spectra were recorded in the range of 200-1000 nm to determine the presence of surface plasmon resonance characteristics of AgNPs, the path length was kept to 1cm, and all the samples were characterized for the freshly prepared samples.

RESULTS AND DISCUSSION

Scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS)

SEM and EDX characterization are mainly utilized for examining the external structure of the material and the elemental makeup of a specimen, respectively. Fig-1a shows the SEM images of biosynthesized silver nanoparticles embedded in its plant extracts. We can see spherical nanoparticles distributed evenly in the samples, with sizes varying between 100 to 200nm. Further, the surface is even and uniform with no defects. While EDS uses the electron beam of the SEM to interact with the sample and emits characteristic X-rays. These X-rays are then analyzed to identify the constituents found within the material and quantify their concentrations. Fig-1b shows the EDS graph and Table-1 shows the EDS percentage of the biosynthesized nanoparticles, the analysis confirmed the presence of silver nanoparticles in the samples, with an atomic percentage of 0.94% indicating the successful synthesis of AgNPs. The EDS percentage is not a precise indicator of the material's elemental composition, as it mainly takes on the surface of the material. However, EDS gives a significant insight into the material composition [8].

Table 1 Elements embedded in biosynthesized silver nanoparticles with percentage

Element	Weight %	Atomic %
C K	21.21	30.07
N K	12.98	15.78
O K	36.98	39.36
Si K	22.85	13.85
Ag L	5.97	0.94
Total	100.00	

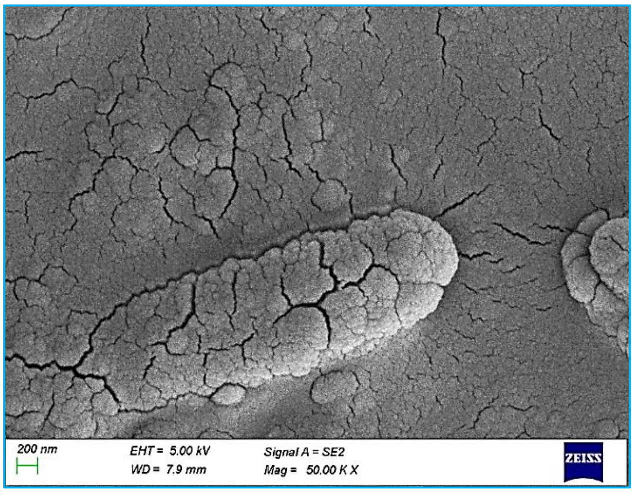

Fig 1a SEM image of biosynthesized silver nanoparticle

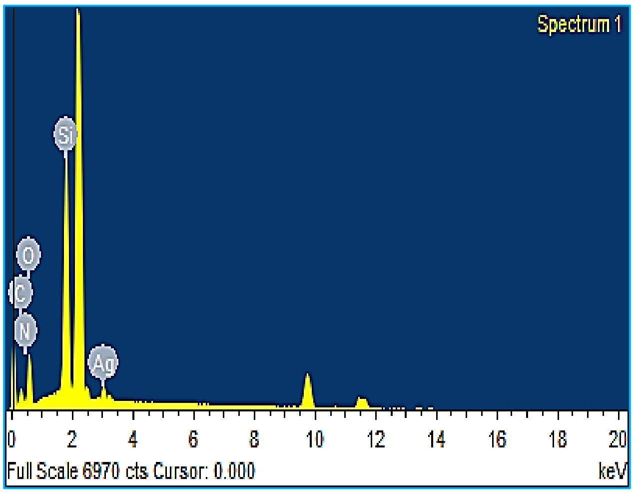

Fig 1b EDS graph of biosynthesized silver nanoparticle

Fourier- Transform infrared spectroscopy (FTIR)

Biosynthesized silver nanoparticles (Fig 2) are subjected to FTIR in the range of 400-4000 Cm^{-1}. The spectra produced by FTIR show 6 characteristic bands at 817, 1029, 1307, 1631, 2380, and 3360 Cm^{-1} [4].

- 817 Cm^{-1}: This band may indicate the presence of metal-oxygen vibrations, suggesting interactions between silver ions and functional groups in the stabilizing agents or plant extracts used in the synthesis process.
- 1029 Cm^{-1}: This band could correspond to C-O stretching vibrations, implying the presence of carbohydrates or polyphenols from the plant extracts, which may act as reducing and stabilizing agents for the silver nanoparticles.
- 1307 Cm^{-1}: This band might represent C-H bending vibrations, potentially originating from organic molecules or surfactants involved in the stabilization of the nanoparticles.
- 1631 Cm^{-1}: This band is characteristic of C=O stretching vibrations, indicating the presence of carbonyl groups from organic compounds in the plant extract or potential surface functionalization of the nanoparticles.
- 2380 Cm^{-1}: This band is quite high and falls outside typical organic functional group regions, suggesting it may be an instrumental artifact or due to gas absorption.
- 3360 Cm^{-1}: This band likely indicates O-H stretching vibrations, suggesting the existence of hydroxyl groups within organic molecules or water molecules absorbed onto the nanoparticles' surface which could contribute to their stability.

In summary, the presence of these FTIR bands indicates the participation of organic compounds derived from the plant extract in the synthesis of silver nanoparticles. These compounds likely contribute to stabilizing the nanoparticles, potentially boosting their antimicrobial effectiveness and broadening their possible uses across different fields like water treatment, etc.

UV-Visible-NIR spectrophotometer

Optical properties of materials correspond to the interaction of light and its influence on absorption, and transmission of the material. This gives the instincts about molecular structure, composition, bandgap energy, and electronic transitions [7]. These properties play a vital role in scientific understanding, the behavior, and the performance of materials in various practical applications. Fig-3a shows the absorbance and Fig-3b shows the transmittance of the bio-synthesized silver nanoparticles, the absorbance spectra show a strong absorbance peak at around 440nm, with a wide range of absorbance from 200 to 650nm. The transmittance spectra show two distinct transmittance edges clearly showing the formation of nanoparticles, with transmission edges at 320nm and 600nm respectively. The band gap energy of the materials was calculated using absorption edge wavelength using the formula:

$$E_g = \frac{hc}{\lambda}$$

The E_g is band gap energy of the material, "h" represents Planck's constant (6.626×10−34 m²Kg/s), "c" denotes the speed of light in vacuum (3×108 m/s), and "λ" stands for the wavelength of the light. (in m). The energy band gap was found to be 4.4eV corresponding to wavelength of 280nm. The energy difference of the silver nanoparticles band gap can be precise control over the size, shape, and aggregation state by fine-tuning the synthesis time, and pH of the reaction. Further, silver nanoparticles demonstrate a strong phenomenon of localized surface plasmon resonance (LSPR) and are distinguished by vibrant and adjustable absorption bands within the visible spectrum to near-infrared spectra, making them highly attractive for various advanced applications.

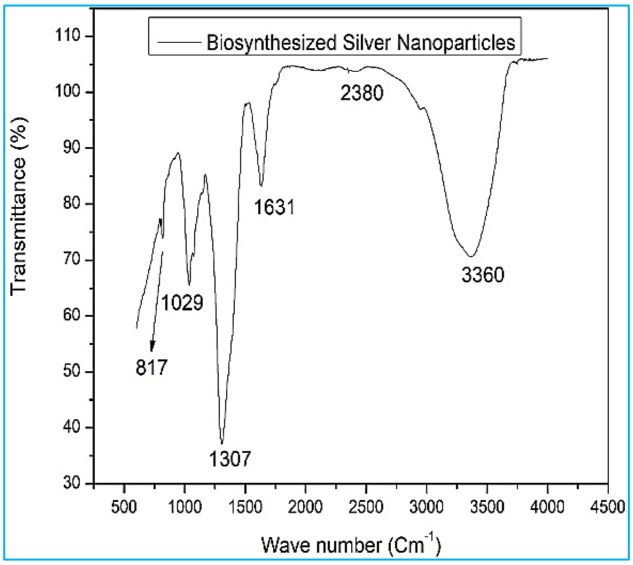

Fig 2 FTIR spectra of biosynthesized silver nanoparticle

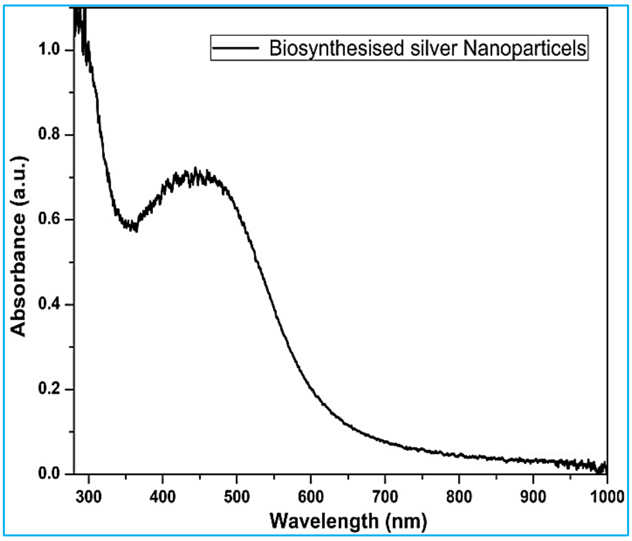

Fig 3a Absorbance spectra of biosynthesized silver nanoparticles

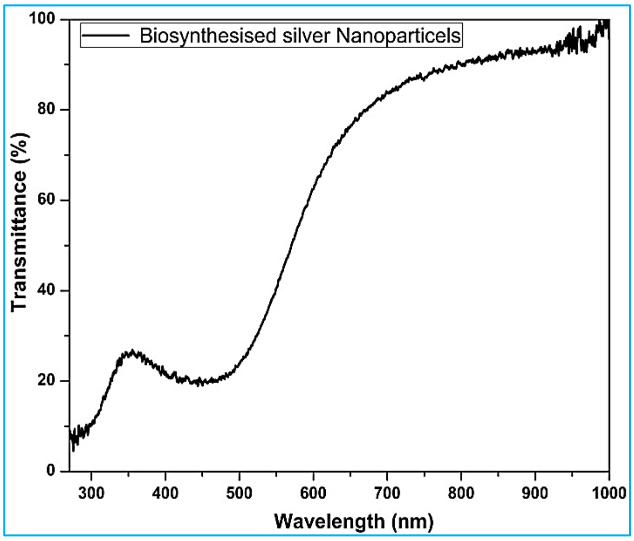

Fig 3b Transmittance spectra of biosynthesized silver nanoparticles

CONCLUSION

In conclusion, this study comprehensively characterizes biologically synthesized silver nanoparticles using SEM-EDS, FTIR, and UV-Visible-NIR spectrophotometry. SEM-EDS analysis confirmed the morphology and elemental composition of the nanoparticles, while Fourier-transform infrared spectroscopy (FTIR) revealed the involvement of functional groups in their stabilization. While UV-Visible-NIR spectroscopy provided insights into their optical properties. The combination of these techniques offers valuable information about the structural, chemical, and optical characteristics of biologically synthesized silver nanoparticles, facilitating their potential applications in various fields.

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