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Biogenic synthesis of multi-applicative silver nanoparticles by using *Ziziphus Jujuba* leaf extract

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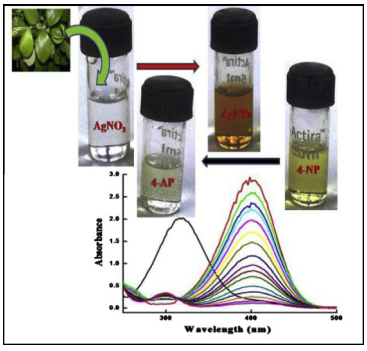
HIGHLIGHTS

- A novel synthesis of AgNPs at room temperature using *Ziziphus Jujuba* leaf extract.
- A green, rapid, one step, cost effective, environmentally friendly synthesis.
- Biogenically synthesized AgNPs were found to be stable for more than six months.
- Reduction of 4-nitrophenol and Methylene Blue for environmental protection. Also the synthesized AgNPs shows good antimicrobial activity against *Escherichia coli*.

GRAPHICAL ABSTRACT



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ABSTRACT

Herein, we are reporting for the first time one step biogenic synthesis of silver nanoparticles (AgNPs) at room temperature by using *Ziziphus Jujuba* leaf extract as a reducing and stabilizing agent. The process of nanoparticles preparation is green, rapid, environmentally benign and cost effective. The synthesized AgNPs were characterized by means of UV–Vis., XRD, FT-IR, TEM, DLS and Zeta potential. The absorption band centered at λ_{max} 434 nm in UV–Vis. reflects surface plasmon resonance (SPR) of AgNPs. XRD analysis revealed, that biosynthesized AgNPs are crystalline in nature with the face centered cubic structure. FT-IR analysis indicates that nanoparticles were capped with the leaf extract. TEM images shows the synthesized nanoparticles are having different shapes with 20–30 nm size. The data obtained from DLS that support the hydrodynamic size of 28 nm. Zeta potential of −26.4 mV indicates that the nanoparticles were highly stable in colloidal state. The effect of pH, quantity of leaf extract and concentrations of $AgNO_3$ were also studied to attend control over the particle size and stability. The synthesized AgNPs shows highly efficient catalytic activity towards the reduction of anthropogenic pollutant 4-nitrophenol (4-NP) and Methylene Blue (MB) for environmental protection. Synthesized AgNPs also exhibited good antimicrobial activity against *Escherichia coli*.

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Introduction

The demands of nanomaterials are increasing day by day due to their distinctive physicochemical properties as compared to bulk counterparts [1]. In particular noble metal nanoparticles have wide applications in various fields such as medicine, electronics, energy and catalysis [2–4]. The synthetic route for the preparation of nanostructure materials are more important in the current research due to their physical properties which can be tailored for a specific application by controlling their size and morphology [5,6]. Generally metal nanoparticles have been prepared by

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physical and chemical methods [7]. However, due to their negative impact, there is an urgent need to replace the chemical method with clean, non toxic and environmentally acceptable biological method.

From the literature survey, plenty of reports are available on the synthesis of silver nanoparticles by bio-reduction method based on bacteria, fungi, microorganisms and plant extract [8–12]. The use of plant extract for the synthesis of nanomaterials could be more advantageous as it does not involve elaborative process and recoil from maintenance of microbial cell culture [13]. From the literature survey, it is found that the rate of metal ion reduction by plant extract is much more faster than the microorganism [14]. The biomolecules in plant extract like proteins, phenols and flavonoids plays crucial role for the reduction of metal ions [15].

Silver nanoparticles have gained much popularity on account of their broad spectrum of antimicrobial and surface plasmon resonance (SPR) effect [16–20]. AgNPs have been synthesized by variety of plants extract that includes Aloevera [21], Cassia auriculata, Ocimum tenuif lorum, Mentha piperita [22]. A very few reports are available on the use of biogenically synthesized AgNPs without supporting on other materials for the reduction reactions. To the best of our knowledge, we are reporting for the first time, one pot environmentally benign and rapid synthesis of AgNPs using easily available *Ziziphus Jujuba* leaf extract as a green reducing and stabilizing agent. The synthesized AgNPs were thoroughly characterized by UV–Vis., XRD, FT-IR, TEM, DLS and Zeta potential. Moreover, the synthesized AgNPs were tested for the reduction of 4-nitrophenol to 4-aminophenol (4-AP) and Methylene Blue. Further AgNPs were also tested for antimicrobial activity against *Escherichia coli* (*E. coli*).

Materials and methods

Z. Jujuba leaves were collected from Kolhapur India. Silver nitrate was obtained from Sigma Aldrich chemicals Pvt. Ltd., Mumbai. NaOH, H_2SO_4, $NaBH_4$, 4-nitrophenol and Methylene Blue were purchased from Spectrochem Pvt. Ltd., Mumbai (India). All the chemicals were of analytical grade and used as supplied. All solutions were prepared in millipore water obtained from millipore water system (Millipore Corp. Bangalore, India). All glassware used for preparation of AgNPs were properly washed with distilled water and dried in oven.

Synthesis of AgNPs

Fresh leaves of *Z. Jujuba* were collected and washed several times with distilled water then shade dried to remove residual moisture. 10 g dried leaves were crushed, boiled in 200 mL of distilled water for 10 min then leaf extract was filtered through Whatman No. 1 filter paper and centrifuged at 1500 rpm for 5 min to remove heavy biomaterials. Further, for the formation of AgNPs 1.5 mL of leaf extract was added into 100 mL (0.001 M) aqueous solution of $AgNO_3$ with constant stirring at room temperature.

Effect of quantity of leaf extract on AgNPs synthesis

In order to optimize the quantity of leaf extract for the synthesis of AgNPs the leaf extract was varied from 0.5 to 2.5 mL in 100 mL of $AgNO_3$ solution. The formation of AgNPs were tested by using UV–Vis. spectrophotometer.

Effect of concentration of $AgNO_3$ on synthesis of AgNPs

The concentration of precursor is also significant parameter for the synthesis of nanoparticles. Therefore, the concentration of $AgNO_3$ solution was optimized by varying the concentration of $AgNO_3$ from 0.0001 to 0.01 M.

Effect of pH on synthesis of AgNPs

pH is a significant factor for biosynthesis of nanoparticles. The effect of pH on the synthesis of AgNPs was studied in the range of 4–9. To control the pH, H_2SO_4 and NaOH (0.01 M) solutions were used. The other factors like volume of leaf extract, concentration of $AgNO_3$ and temperature were kept constant. The effect of pH on the synthesis of AgNPs was monitored by UV–Vis. spectroscopy.

Characterization of AgNPs

The formation of AgNPs was monitored by UV–Vis. NIR spectrophotometer (Shimadzu, UV-3600). XRD analysis describes the crystallinity and an average size of synthesized material. FT-IR (Cary 630, Agilent Technology) spectral analysis in the range of 4000–650 cm^{-1} has been carried out to identify the possible biomolecules present in the *Z. Jujuba* leaf extract which are responsible for the formation and stability of AgNPs. The shape and size of the materials were obtained by using transmission electron microscopy (TEM). TEM images were scanned with JEOL JEM 2100 TEM equipped with high resolution Gatan CCD camera. The particle size distribution and stability of nanoparticles were evaluated by DLS and Zeta potential measurements using Malvern Zetasizer (nano ZS-90) equipped with 4 mW, 633 nm He–Ne Laser (U. K.) at 25 °C under a fixed angle of 90° in disposable polystyrene cuvettes.

Catalytic activity of AgNPs

The catalytic activity of synthesized AgNPs was tested for the reduction of 4-NP and MB. The reduction reactions were monitored by UV–Vis. spectrophotometer.

Catalytic activity of AgNPs for reduction of 4-NP

In order to check the catalytic activity of synthesized AgNPs for the reduction of 4-NP in a 3.5 mL quartz cuvette 1.5 mL of water, 0.5 mL of 4-NP (0.5 mM) and 1 mL $NaBH_4$ (0.02 M) were taken. In the same solution 15 μL colloidal solution of AgNPs were added and time dependant absorption spectra were recorded for the reduction reaction at room temperature.

Catalytic activity of AgNPs for reduction of MB

The catalytic activity of AgNPs for the reduction of MB was investigated by comparing the absorbance values of three different reaction mixtures which is filled in three test tubes. In the first test tube, 1 mL MB (5 ppm) and 2 mL water were taken and the reaction was monitored by using UV–Vis. spectrophotometer. In the second test tube, 1 mL MB (5 ppm) and 0.2 mL *Z. Jujuba* leaf extract were mixed into 1.8 mL water and the absorbance was monitored after 30 min. In the third test tube, 1 mL MB (5 ppm) and 0.2 mL *Z. Jujuba* leaf extract were mixed into 1.8 mL AgNPs and absorbance was monitored after 30, 45, 60 and 75 min. After that absorbance values of first and second reaction mixture were compared with the absorbance values of third one.

Antimicrobial activity

The antimicrobial activity of AgNPs against *E. coli* was studied by agar well diffusion method. Stock culture was maintained at 4 °C on agar slant of nutrient media. Before experiment, pure culture was sub cultured onto nutrient agar slant and it was incubated overnight at 37 °C. Later nutrient agar plate was prepared and punctured for wells and located for 50 μL of AgNPs. After 24 h of

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