

Original Research Article

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Nano Dimensional (1-20nm) Silver Nanoparticles: Stem Extract of *Capparis decidua* (FORSK.) EDGEW Mediated Synthesis and its Characterization-A Lab to Land Approach

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ABSTRACT

A renewable innate source aqueous *Capparis decidua* stem extract was used to investigate an eco-friendly approach for the synthesis of silver nanoparticles (AgNPs) from silver nitrate solution. Biosynthesized silver nanoparticles were characterized by UV-visible Spectroscopy, Transmission Electron Microscopy (TEM) and Fourier Transform Infra-Red Spectroscopy (FTIR). The UV-visible spectroscopy showed the maximum absorbance *i.e.* 0.94 at 460 nm. The stability of nanoparticles after four months depicted almost no shift in the absorption intensity and the absorption maxima which indicated that the particle size is same. The broadening of peak specified that the particles were polydispersed. TEM analysis conformed the formation of crystalline silver nanoparticles of size range 1.0-19 nm. Very small size range of silver nanoparticles makes it a good antimicrobial agent. Functional groups like amines, amides, alkynes, alkenes, bromoalkanes etc. identified by FTIR analysis investigated the responsive functional groups which stabilises the synthesized silver nanoparticles. The absorbance bands in bioreduction are observed in the region of 400-4000cm⁻¹ by FTIR.

Keywords

Capparis decidua,
Silver nanoparticles,
TEM, FTIR,
Polydispersity,
Absorption maxima.

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Introduction

Today nano metal particles especially silver have drawn attention of scientist because of their extensive application in the development of new technologies in the areas of electronics, material sciences and medicine at the nano scale. Silver nanoparticles are nanoparticles of silver which are in the range of 1 and 100 nm in size. Silver nanoparticles have unique properties which help in molecular diagnostics, in therapies, as well as in devices that are used in several medical procedures. The major methods used for silver nanoparticle synthesis are the physical and chemical methods. Considering the drawbacks of physio-chemical methods, cost-

effective and energy efficient new alternative for AgNP synthesis using microorganisms (Sharma *et al.*, 2009), plant extracts (Song *et al.*, 2009) and natural polymers (Huang *et al.*, 2004) as reducing and capping agents are emerging very fast. The association of nanotechnology and green chemistry will unfold the range of biologically and cytologically compatible metallic nanoparticles which is a strong benevolent to lab to land approach. In physical synthesised silver nanoparticles by laser ablation method the properties of particles synthesised depends upon the wavelength and duration of the laser, the ablation time duration and the effective

liquid medium which may or may not contain the surfactant (Mafune *et al.*, 2000; Kabashin *et al.*, 2003). The chemical synthesis approach is inevitably associated with the use of hazardous chemicals such as reductants, stabilizers and organic solvents, or has special requirements for the employed techniques such as high energy radiation, microwave irradiation and inert gas condensation (Shin *et al.*, 2004; Mittal *et al.*, 2004). Global warming and climate change has persuaded a worldwide awareness to reduce the toxic and hazardous waste materials as a result of this the green synthesis has gained progress in the fields of science and industry (Ahmad *et al.*, 2010). In recent years, the biological approaches using microorganisms and plant extracts have become valuable alternatives to chemical synthesis. The physical and chemical processes often involve high temperatures/pressure for the reaction and the use of hazardous chemicals due to which the biological methods of synthesising metal nanoparticles is gaining importance. Plant extracts are considered cost-effective, environment friendly and efficient alternative for the large-scale synthesis of nanoparticles. Here the present work represents a stupendous effort to develop the eco-friendly, nontoxic and cost-effective approaches for the preparation of AgNPs without the application of toxic chemicals and special equipments.

The use of plant and plant extract in nanoparticle synthesis is considered advantageous over microbial based system because it reduces the elaborate process of maintaining cell cultures. In comparison to microorganisms, the application of plant extracts for the synthesis of AgNPs is more advantageous in terms of resource availability, security, reaction rate and convenience, and feasibility of large scale synthesis (Mane Gavde *et al.*, 2015, Jalaluddin *et al.*, 2016). The particle size, growth can also be controlled by altering synthesis conditions like pH, reductant

concentration, temperature, mixing ratio of the reactants etc. The plant based synthesis can be carried out either extracellularly or intracellularly. Intracellular synthesis takes place inside the plant whereas the extracellular synthesis occurs *in vitro*. The findings reported that extracellular synthesis using plant extracts has been considered better as compared to intracellular (Makarov *et al.*, 2014) because it abolish the extraction and purification methods. Green synthesis have been reported in *Magnifera indica* (Sandeep *et al.*, 2017), *Corynebacterium* species (Gowramma *et al.*, 2015).

Till date, lot of papers has been published in this area which describes the mechanism and role of active biomolecules in synthesis. These studies suggested that presence of phytochemicals in plant extracts are the key component in reduction and stabilization of silver ions (Jegadeeswaran *et al.*, 2012) The phytochemicals which are responsible for reduction are terpenoids, flavonoids, ketones, aldehydes, amides, and carboxylic acids. The water soluble metabolites such as flavones, organic acids, and quinones are solely responsible for the bioreduction ions. Some researchers have reported that a keto-enol transition of anthraquinone is responsible for formation of AgNPs. It has been also observed that mesophytes contain three types of benzoquinones: cyperquinone, dietchequinone, and remirin which might be responsible for reduction of ions and formation of AgNPs (Nafeesa khaton *et al.*, 2017).

Capparis decidua (Family, Capparidaceae) is a shrub or small tree commonly known as 'ker' is an important medicinal plant. It is a rich source of beta-carotene (Talili *et al.*, 2009) and mineral elements (Ozcan *et al.*, 2008). The leaves of *C. decidua* possess phenolic compounds with multiple biological activities (Ramezani *et al.*, 2008). Aerial parts (flower, fruit, stem and seed) contain n-

triacontane, n-triacontanol, n-pentacosane, 6-(1-hydroxy-non-3-enyl) tetrahydropyran-2-one, 2-carboxy-1-dimethylpyrrolidine, β -sitosterol, β -carotene, Glucosinolates, Ascorbic acid, proteins, carbohydrates, Calcium, Potassium, Phosphorous, Zinc, Iron and Manganese (Rathee *et al.*, 2010). The quantity of β -sitosterol in the stem is found to be 0.044% by TLC densitometric method using HPTLC (Rathee *et al.*, 2010).

Materials and Methods

Preparation of the stem extract

The explant was collected from village Samargopalpur (Rohtak, Haryana) India in December 2014. Stem extract of *Capparis decidua* was selected for the biosynthesis of AgNPs because of its ease of availability and medicinal properties. For biosynthesis Fresh and healthy part of stem were collected locally and rinsed thoroughly with tap water followed by doubled distilled water to remove all dust and unwanted visible particles, after which they were dried at room temperature to remove the water from the surface of stem, then cut into small pieces. Next, 20 g of these finely incised stem parts crushed in 100ml distilled water and boiled at 80°C for 20min.

After cooling at room temperature, the mixture was centrifuged at 20,000 rpm for 30 min and filtered. The filtrates were then stored at 4–8 °C and used as reducing and stabilizing agents in the synthesis of AgNPs. The sterile conditions were maintained throughout the experiment (Medda *et al.*, 2014).

AgNPs synthesis

Aqueous solution of 1mM silver nitrate (AgNO₃) was prepared in a 250ml Erlenmeyer flask and used for the synthesis of AgNPs. Briefly, 12ml of *Capparis decidua*

stem extract was added into 88ml of aqueous solution of 1mM silver nitrate and incubated in the dark overnight at room temperature. The primary step to detect complete reduction of AgNO₃ to Ag⁺ ions was confirmed by the change in colour from colorless to colloidal brownish yellow. The colloidal mixture was then sealed and stored properly for future use. The formation of AgNPs was further confirmed by spectrophotometric analysis. The silver nitrate (AgNO₃) was purchased from Sigma-Aldrich, India.

UV-Vis spectra analysis

Preliminary characterization of the AgNPs was carried out using UV–Visible spectroscopy. The reduction of silver ions to the nanoparticle form was monitored by measuring the UV–Visible spectra of solutions after diluting the sample with Millipore water 20 times. The spectra of AgNPs solution was monitored by a UV-Vis spectrophotometer SHIMADZU UV-Vis Spectrophotometer 2450 and a UV-Vis spectrograph of the colloidal solution of silver nanoparticles was recorded as a function of time by using quartz cuvette with water as reference and scanning the spectra between 200-800 nm at the resolution of 0.1nm.

Fourier transformation infrared (FTIR) spectroscopy

FTIR spectroscopy was used to investigate the functional groups in the stem extract and their possible involvement in the synthesis of AgNPs. The modified functional groups in the green synthesized nanoparticles were determined with various concentrations of AgNPs. Buffer subtracted transmission spectra were recorded in the wave number range of 375–4000 cm⁻¹ using a ALPHA Platinum ATR (attenuated total reflectance) FTIR spectrometer (Bruker, Germany) from the Department of Genetics, M.D.U Rohtak.

The binding properties of AgNPs investigated by FTIR analysis and the difference between the respective binding agents are verified.

Transmission electron microscopy (TEM)

TEM grids were prepared by sonicating the AgNPs sample solution for 20 min and placing a few drops on the 300 mesh carbon-coated copper grid and dried for the complete evaporation of water under a lamp and operated at an accelerating voltage of 100 kV using EM2000Fx-II, transmission electron microscope, which is a (<Hitachi H-7500>, resolution 0.204 nm, lattice image: 0.36 nm point to point, magnification: 6, 00,000x) with AMT XR-16 16mp high resolution CCDcamera, facility was availed from Sophisticated Analysis Instrument Facility, Panjab University, Chandigarh to characterize the sample after usual alignment procedures.

Results and Discussion

Silver nanoparticles were synthesized successfully from the stem extract of *Capparis decidua* with aqueous silver nitrate solution. The presence of nanoparticles in the medium was confirmed by the change in color from colorless to brown or deep yellow shown in (Fig. 1). The color intensity increased with a period of incubation due to the reduction in silver nanoparticles and excitation of surface plasmon vibrations (S.Li *et al.*, 2007) and provides a convenient spectroscopic proof of their formation (N. Saifuddin *et al.*, 2009).

UV–Vis spectrophotometer analysis

The silver nanoparticles were characterized by UV–Vis spectroscopy, one of the most widely used techniques for structural characterization of silver nanoparticles (Sun *et al.*, 2001). Biological method of silver nanoparticles synthesis exhibit strong

absorption of electromagnetic waves in the visible range due to their optical resonant property, called occurs due to its collective oscillation of conduction electrons, combined with the incident light (Kreibig and Vollmer, 1995). Figure 2 shows the UV–Vis absorption spectra at 460 nm with 0.94 absorbance. This peak is due to Surface Plasmon Resonance (SPR) property of silver nanoparticles. Nanoparticles have optical properties that are sensitive to size, shape, concentration, agglomeration state, and refractive index near the nanoparticle surface, which makes UV/Vis/ IR spectroscopy a valuable tool for identifying, characterizing, and studying these materials (Shakeel Ahmad *et al.*, 2016) Nanoparticles made from certain metals, such as gold and silver, strongly interact with specific wavelengths of light and the unique optical properties of these materials is the foundation for the field of plasmonics. The prepared aqueous solution of AgNPs showed a strong absorption band between 410 and 460 nm, which is a typical absorption band of spherical Ag nanoparticles due to their surface plasmon and widening of the peak indicated that the particles were polydispersed (Fig. 2) Similar results were reported in *Cassia italica* (Sermakkani *et al.*, 2012; Mulvney 1996; Shibila ans Johnson, 2015; Sai Lakshmi *et al.*, 2015).

TEM analysis of silver nanoparticles

The well-known technique for imaging solid materials at atomic resolution is TEM. The technique was employed to visualize the size and shape of Ag nanoparticles. From our results, it is observed that most of the Ag nanoparticles were spherical in shape. Figure 3 clearly shows the TEM image of individual silver nanoparticles with the particle size estimated as in between the range of 1- 20 nm. The stem extract synthesized nanoparticles with spherical shape has also been reported in *Cinnamom zeylanicum* with

size range of 31-40nm (Joyita Banerjee and Narendhirakannan, 2011), *Shorea tumbuggaia* and *Boswellia ovalifoliolata* (Harekrishna Bar *et al.*, 2009). In the present work as per Transmission Electron Microscopy results the nanoparticles were found not in direct contact and even no aggregates were seen which is an indication of the stabilization of the nanoparticles by a capping agent (due to

presence of proteins and secondary metabolites in the stem extract). Proteins have stronger ability to bind metal indicating that the proteins could be a covering layer around the AgNPs preventing agglomeration, hence, behaves as a capping agent for AgNPs (Sermakkani *et al.*, 2012). The smaller range of size of silver nanoparticles of *Capparis decidua* makes it a better microcidal.

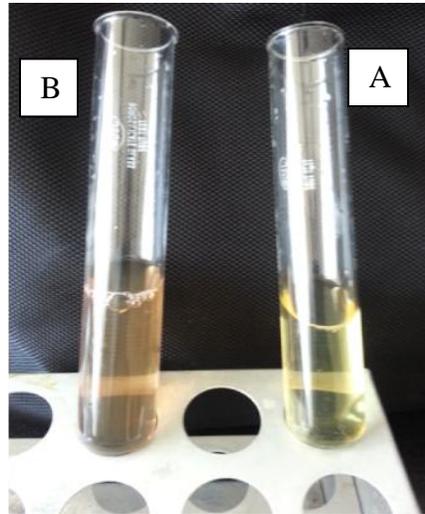


Fig.1 *Capparis decidua* (a) stem extract (b) silver nanoparticle synthesized

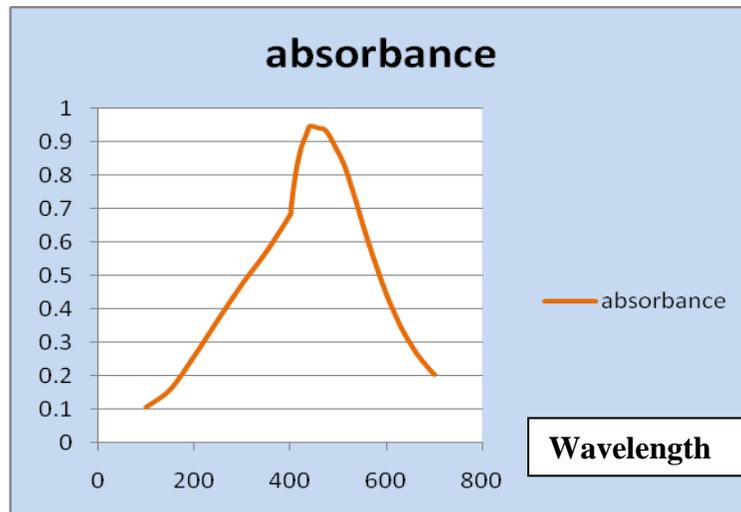


Fig.2 UV-Visible absorption spectrum of Biosynthesized silver nanoparticles

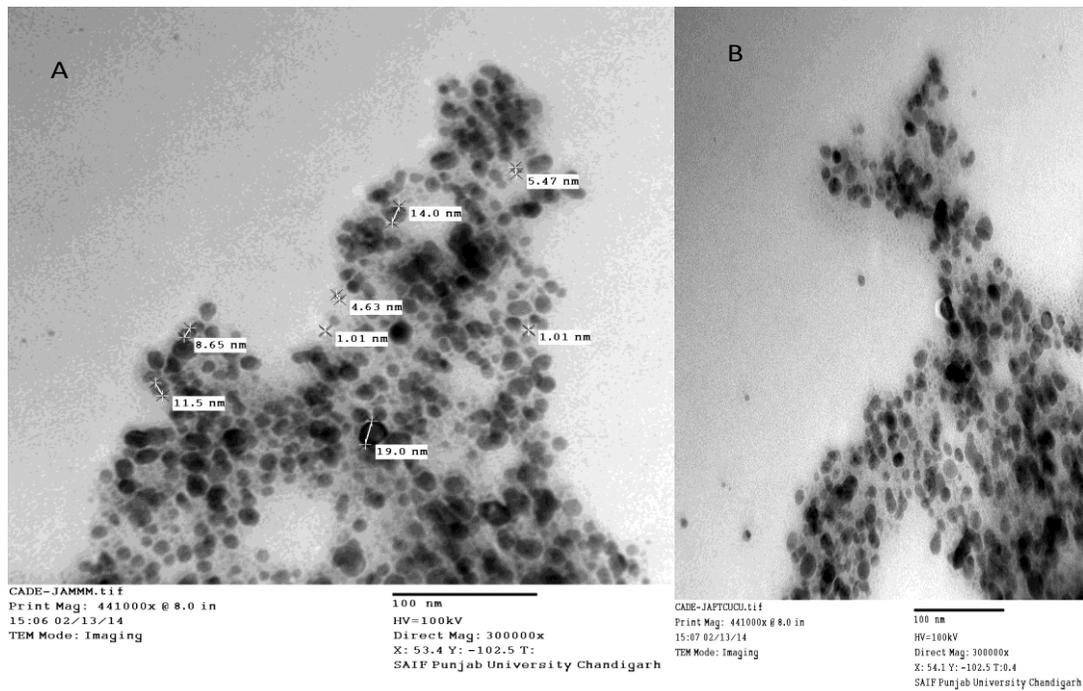


Fig.3 TEM images of silver nanoparticles at different magnification levels

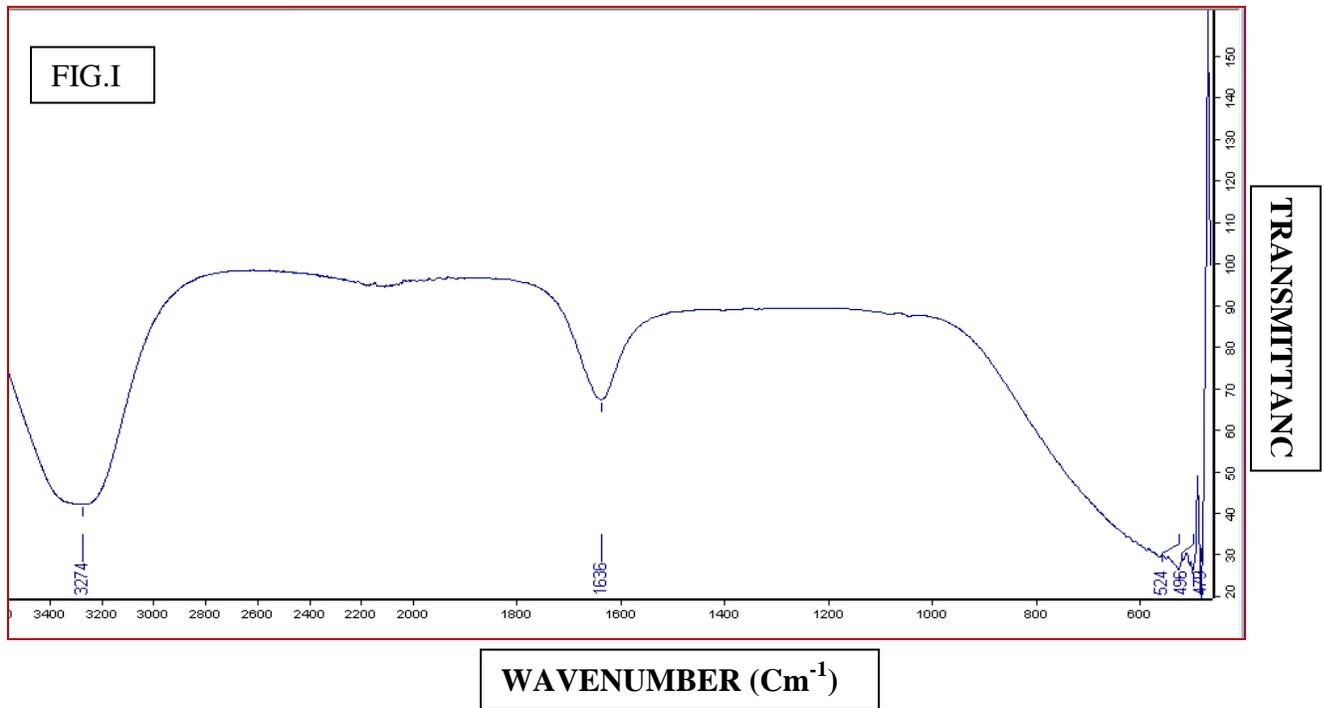


Fig.4 FTIR spectra of stem extract of *C. decidua*

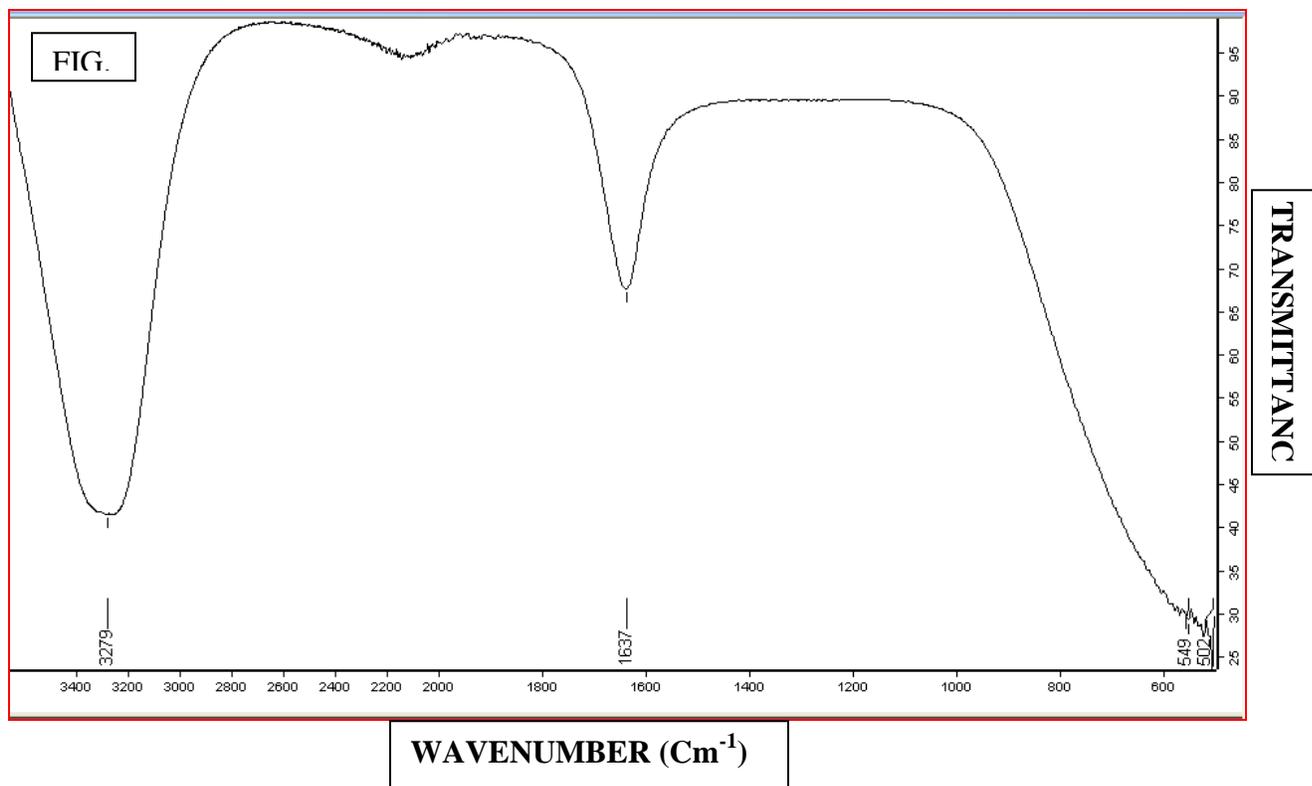


Fig.5 FTIR spectra of stem extract synthesized silver nanoparticles of *C. decidua*

FTIR analysis

FTIR absorption spectra of water dissolved Stem extract and the stem extract synthesized AgNPs (Figs. 4 and 5). FTIR can be used to analyze samples up to ~11 millimetres in diameter and either measure in bulk or the top ~1 micrometer layer. FTIR measurements were carried out to identify the possible biomolecules responsible for capping and efficient stabilization of the metal nanoparticles synthesized by *Capparis decidua* stem extract. The absorbance bands analysis in bioreduction are observed in the region of 470-4000 cm are 3274, 1636 524,496 and 470 cm. The peak observed at 3274 represents N-H stretch (amines and amides) as the range of 3400-3250 cm^{-1} is amide region which is further associated with terminal Alkynes considered generally as stronger and sharper. The results are relevant to the earlier literature where binding as well

as stabilization occurs by free amide groups present in proteinaceous substance used for synthesis (Rajesh *et al.*, 2009; Sermakkani *et al.*, 2012). The peak at found at 1636 *i.e.* between the range of 1614-1640 is C-C in alkene rings and C=C stretch of aromatic rings, 524 cm^{-1} lies in the range of 690-515 cm^{-1} is C-Br stretch which is characteristic of alkyl halides. Whereas 470-496 cm^{-1} range may also be assigned to the first overtone mode of the methyl torsion vibration (Marimuthu *et al.*, 2011) From the above observation it is assumed that the stabilisation is achieved by the identified compounds present in the plant extract. FTIR analysis was used to detect the characteristic peaks and their functional groups. The broadening of peaks indicated that the particles are polydispersed. The capping was confirmed by the existence of bands at 3279, 1637, 549 and 502 cm^{-1} (Ahlawat *et al.*, 2015, Jyoti *et al.*, 2016).

Here we report a simplistic 'green' route for the synthesis of AgNPs using aqueous stem extract of *Capparis decidua*, and the characterization of the nanoparticles via UV-Vis spectroscopy, TEM analysis. Native Synthesized nanoparticles were showing UV-Vis absorption spectra at 460 nm with 0.94. The TEM analysis indicates the presence of 1-20 nm size of silver Nanoparticles, which strongly support it to become a better microcidal agent. The synthesized silver nanoparticles were spherical in shape. The present study is eco-friendly, need cheaper cultivation requirements, higher growth rates on laboratory scales and use of biological agent in the synthesis which enhances lab to land approach. The stabilization of the nanoparticle is believed to occur by the presence of phytochemicals present in the plant extract and in recent years, the progress of efficient green chemistry approaches for the fabrication of commercially viable noble metallic nanoparticles has become a major focus of researchers. Moreover, this process could be easily scaled up for the industrial applications to increase the yield of the nanopartilces significantly, which undoubtedly would establish its commercial viability in medicine.

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Conflict of Interest

The Authors have no conflict of interest.

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