



## Parthenium Mediated Synthesis of Gold Nanoparticles and its Characterization

Siva Kumar Ramamurthy<sup>\*1</sup>, Chenchugari Sridhar<sup>2</sup>

<sup>1\*</sup> Research scholar of Rayalaseema University Kurnool, Andhra Pradesh, India – 518007.

<sup>2</sup> Professor & Director (Academics), Sri Padmavathi School of Pharmacy, Tiruchanoor, Andhra Pradesh, India – 517503.

\*Corresponding author's E-mail: [sivapharma2@gmail.com](mailto:sivapharma2@gmail.com)

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### ABSTRACT

The prime objective of this study is to biosynthesize Gold nanoparticles by using *parthenium hysterophorus* plant extract as a reducing agent and its characterization by spectroscopic techniques. A novel method was developed to prepare gold nanoparticles by using gold trichloride as a precursor and biosynthesis of gold nanoparticles was mediated by *parthenium hysterophorus* plant extract without the aid of external energy (high pressure and temperature). This new method involves simple techniques such as centrifugation, filtration and stirring. Gold nanoparticles formation was confirmed by analytical techniques such as UV-Visible spectroscopy, powder X-ray diffraction (XRD), Fourier Transforms Infrared Spectroscopy (FTIR) and by scanning electron microscopy (SEM) analysis. The XRD measurement showed that gold nanoparticles possess a typical face center cubic structure and the crystallite size of the synthesized gold nanoparticles was found to be 10.8 nm calculated by scherrer's formula. The UV-Visible spectrum of gold nanoparticles shown well-defined excitation band at 534 nm which is the characteristic of gold nanoparticles surface plasmon resonance (SPR) and confirms the formation of gold nanoparticles with red color. The SEM images show agglomeration of gold nanoparticles clusters. The characteristic bands (3327.21, 1625.99 and 1377.17<sup>-cm</sup>) observed in the FTIR spectrum further confirmed synthesized nanoparticles are gold. An eco-friendly method was established to prepare gold nanoparticles with *parthenium hysterophorus* plant extract which is a novel approach without the aid of external energy. This method can be used in pharmaceutical industry for the synthesis of an antimicrobial agent / Therapeutic agents.

**Keywords:** Gold, nanoparticles, *Parthenium hysterophorus*, UV-Visible spectroscopy, SEM, XRD, FTIR.

### INTRODUCTION

Gold has attracted much attention due to Gold nanoparticles (AuNPs) are considered nontoxic to human cells and presents higher stability when in contact with biological fluids.<sup>1</sup> They also have remarkable potential application in the field of medicine like biological activities such as antimicrobial, antioxidant, etc.

Many studies also demonstrated the high antibacterial activity of gold nanoparticles connected to antibiotics such as ampicillin, vancomycin, cefaclor, and the antibacterial enzyme lysozyme against Gram-positive and Gram-negative bacteria.<sup>2</sup>

Over the years, there has been a marked improvement in the ability to synthesize gold nanoparticles. However, conventional methods normally involve the use of toxic compounds that are not appropriate for long-term environmental sustainability. There is a growing interest to prepare different type of nanoparticles by environmentally friendly methods that do not use toxic materials in the synthesis procedures.<sup>3-8</sup> Synthesis of metal oxide nanoparticles using the medicinal plant extract is quite novel, which is effective at an affordable cost,<sup>9-13</sup> without any external energy (high pressure, energy, temperature).

The medicinal plant *parthenium hysterophorus* (Feverfew) is traditionally used for vast pharmacological applications (such as treatment of fevers, migraine, headache, infertility, etc.). Among Greek and early

European herbalists, the *parthenium* herb has a long history of use in traditional and folk medicine. *Parthenium hysterophorus* plant extract and gold both have antimicrobial properties. Based on the above facts, *Parthenium hysterophorus* plant was selected with gold metal particle for green synthesis of gold nanoparticles. The objective of the study was to establish an easy method for biosynthesis of gold nanoparticles by using *parthenium hysterophorus* plant extract and its characterization by spectroscopic techniques.

Rationale for the study is its pharmaceutical use as antimicrobial agent. Easy method for synthesis without the aid of external energy such as high pressure and temperature. This study is a novel approach that describes the easy synthesis of gold nanoparticles without heat treatment, using *parthenium hysterophorus* plant extract. These gold nanoparticles were characterized by spectroscopic techniques for the confirmation of the formation of gold nanoparticles. This research study provides an established method for biosynthesis of gold nanoparticles which can be used as an antimicrobial agent in the pharmaceutical industry.

### MATERIALS AND METHODS

#### *Parthenium hysterophorus* plant material

Flowers and leaves of *parthenium hysterophorus* plant were collected from the forest of Tirumala, Andhra Pradesh, India. *Parthenium hysterophorus* plant of family



*Asteraceae* was identified by the Department of Botany, Sri Venkateswara University, Tirupati with voucher number 1216. The plant was identified based on the leaves, lobed with fine soft hair, flowers on the top are small creamy colored with black colored seed. Based on the features of the plant it was confirmed as *parthenium hysterophorus*.

#### Preparation of *parthenium hysterophorus* plant extract

After the identification of the plant, the leaves and flowers were separated from the plant. The leaves and flowers were dried under dark and shady conditions, without exposing the material to sunlight. After drying, leaves and flowers were powdered in a mechanical grinder and the fine powder was collected by passing through sieve no 40. This powder is stored in a cool and dry place until its use. Plant powder was extracted in a number of solvents such as methanol, hexane, anhydrous sodium sulfate, acetone, chloroform, diethyl ether. Of all the solvents used, acetone is considered as the best solvent for the extraction of the compound from the leaves and flowers of *parthenium hysterophorus* plant.

50 g of powdered *parthenium hysterophorus* plant material was weighed and carefully transferred into the round-bottomed flask of Soxhlet extractor. Then 250 ml of acetone was added, and the plant material was soaked in acetone for 24 h at room temperature. Then the acetone extract of the plant was filtered using Whatman no 1 filter paper. This supernatant is taken in a separate beaker.

Then the extract was evaporated under reduced pressure to obtain a residue. The residue was adsorbed on silica gel and subjected to column chromatography eluted with hexane and a mixture containing increasing amounts of ethyl acetate. The fraction eluted at 2% of ethyl acetate in hexane was collected separately concentrated and rechromatographed using silica gel column to obtain pure extract. This pure plant extract was used only for further analysis.

#### Biosynthesis of gold nanoparticles with *parthenium hysterophorus* plant extract:

A quantity of 1g of *parthenium hysterophorus* plant extract was dissolved in 100 ml of de-ionized water and centrifuged for 15 min and filtered. Gold chloride trihydrate 0.75g (0,1 M) was used as the precursor for the preparation of gold nanoparticles. 40 ml of the extract of *parthenium hysterophorus* was added dropwise in gold precursor while stirring using a magnetic stirrer. In order to adjust the pH = 12 of the solution, sodium hydroxide (NaOH, 1 M) was added drop-wise while stirring. A red colored precipitate of gold was obtained, which is washed 2-3 times with de-ionized water, filtered and dried to obtain the gold nanoparticles.

## RESULTS AND DISCUSSION

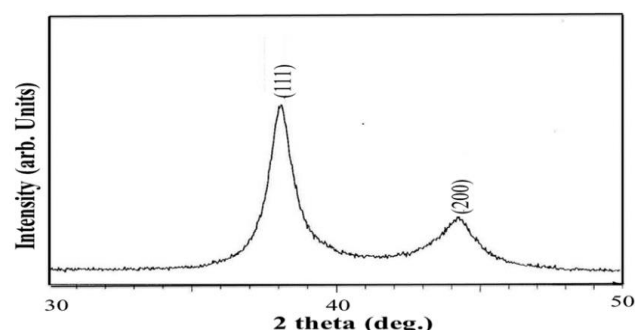
### Characterization of gold nanoparticles:

#### a. Powder X-ray diffraction:

XRD was taken to examine the crystal structure and phase purity of synthesized gold nanoparticles using the extract of *parthenium hysterophorus* plant without annealing. Fig. 1 shows the corresponding XRD pattern. The dominant peaks are corresponded to standard Bragg reflections (111) and (200) of face center cubic (FCC) lattice. The intense diffraction at 38.1 peak shows that the preferred growth orientation of gold was fixed in (111) direction. This XRD pattern is typical of pure gold nanocrystals [standard joint committee on powder diffraction standards (JCPDS) card no. 04-0784]. Further, the pattern shows a line broadening, which indicates the crystallite size reduced. The crystallite size of the material was calculated using the Scherrer's formula:

$$d = Kl/b \cos q,$$

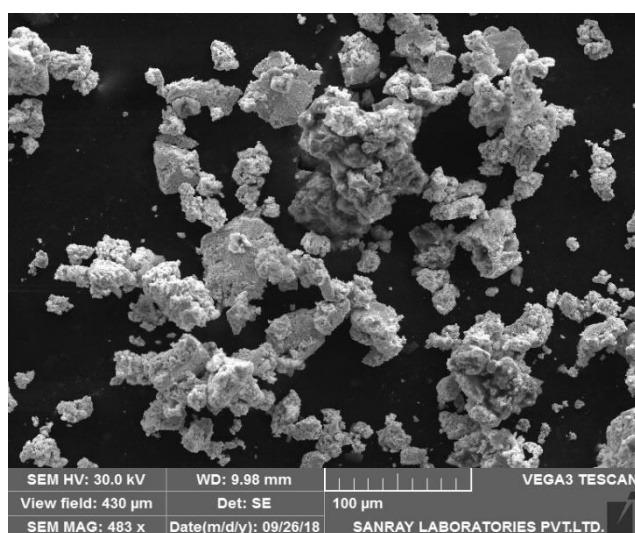
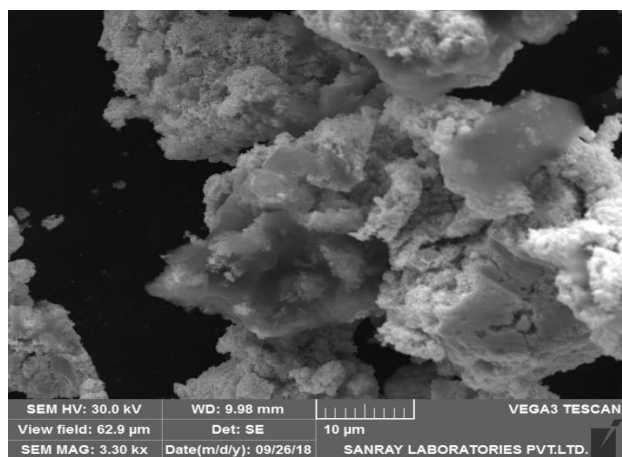
Where  $d$  is the crystallite size,  $K$  is the dimension less shape factor (0.94),  $l$  is the X-ray wavelength,  $b$  is the full width half maxima (FWHM) and  $q$  is the Bragg's angle. The crystallite size of the synthesized gold nanoparticles was found to be 10.8 nm.



**Figure 1:** Room temperature powder XRD pattern of as-prepared gold nanoparticles using *parthenium* extract.

#### b. Scanning electron microscopy:

The SEM images show agglomeration of gold particles (fig. 2(a)). The magnified image is shown in fig. 2(b), it appears to be some of the particles are triangular and hexagonal in shape. Fig. 2 represents the morphology of the as-synthesized gold nanoparticles prepared by using *parthenium hysterophorus* plant extract. Typical SEM images of the gold nanostructures at two different magnifications are shown in fig. 2(a) and (b). It is clear from the lower magnification image that the as-synthesized gold nanoparticles are clusters in a large-scale area and have approximately uniform morphology. Fig. 2(b) shows the higher magnification image of such spherical particles surrounded by amorphous *parthenium hysterophorus* plant extract.

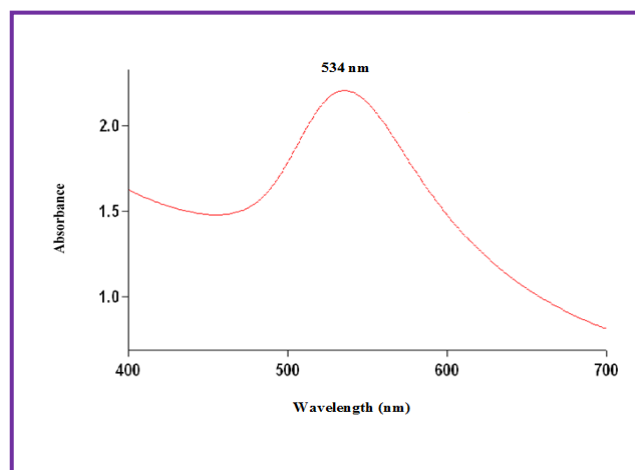


**Figure 2:** SEM images of as-prepared gold nanoparticles using *parthenium* extract at different magnitudes (a) 10  $\mu\text{m}$  and (b) 100  $\mu\text{m}$ .

### c. UV-Visible Spectroscopy

UV-visible absorption spectrum as showed in fig. 3, is carried out to evaluate the potential optical properties of the as-prepared gold nanoparticles using *parthenium hysterophorus* plant extract. For the UV-visible absorption measurement, the as-prepared gold nanoparticles using *parthenium hysterophorus* plant extract sample is ultrasonically dispersed in absolute ethanol before the examination, using absolute ethanol as the reference. The spectrum was corrected for the solvent contribution.

The absorption spectrum of gold nanoparticles using *parthenium hysterophorus* plant extract shows well-defined excitation band at 534 nm which is in good agreement with the characteristic gold nanoparticles surface plasmon resonance (SPR). The SPR property of gold nanoparticles is evident with a characteristic absorption band in between 500 to 600 nm in the UV-Visible spectrum. This absorption band depends on the size and shape of the gold nanoparticles and the dielectric constant of the medium. Hence the present study results confirm the formation of gold nanoparticles.

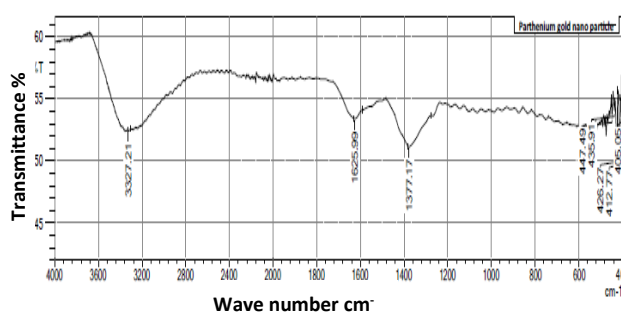


**Figure 3:** Absorption of as-prepared gold nanoparticles using *parthenium* extract as a function of wavelength.

### d. Fourier Transform Infrared Spectroscopy

Fourier Transforms Infrared Spectroscopy (FTIR) was used to identify the possible biomolecules responsible for the reduction of the gold ions and capping of the bio-reduced gold nanoparticles synthesized by *parthenium* plant extract.

The FT-IR spectrum below indicates characteristic three bands at about 3327.21, 1625.99 and 1377.17  $\text{cm}^{-1}$ . The band at 3327.21  $\text{cm}^{-1}$  corresponds to N-H stretching or O-H stretching vibration. The peak 1377.17  $\text{cm}^{-1}$  corresponds to C-N stretching of aromatic amine group. The band at 1625.99  $\text{cm}^{-1}$  corresponds to C=O stretching vibrations due to the carbonyl group in the proteins of amide I group of proteins. The amide I band (between 1600 and 1700  $\text{cm}^{-1}$ ) is mainly associated with the C=O stretching vibration (70-85%) and is directly related to the backbone conformation. Gold nanoparticle might get bonded with protein through free amine group or carboxylate group. This infers that the synthesized gold nanoparticles are surrounded with protein and metabolite such as terpenoid that has functional groups of ketone, alcohol and carboxylic acids.



**Figure 4:** FT-IR spectrum of synthesized gold nanoparticles using *parthenium* extract.

### DISCUSSION

In present time, nanoparticle synthesis approaches include three methods such as Physical methods, Chemical methods and Biological methods.<sup>14,15</sup> Chemical methods

include the reduction of chemicals,<sup>16</sup> electrochemical procedures,<sup>17</sup> and reduction of photochemicals.<sup>18</sup>

Physical and chemical methods are being used extensively for production of metal and metal oxide nanoparticles. However, this production requires the use of very reactive and toxic reducing agents such as sodium borohydride and hydrazine hydrate, which cause undesired detrimental impacts on the environment, plant and animal life it supports.

Nanoparticle synthesis by using plant extracts is eco-friendly, involves simple reactions or minimal conditions such as low temperature and extraction process.<sup>19</sup> Hence Nanoparticle synthesis by using plant extracts is attracting much attention. Major benefits in the plant mediated synthesis of nanoparticles are plant secondary metabolites have pharmacological activity, opportunity for scale up, devoid of hazardous chemicals and their pharmaceutical applications.<sup>20</sup> In addition, synthesis requirements like high temperature, pressure, energy are insignificant.

The green method of synthesizing metal nanoparticles involves platinum (Pt), gold (Au), silver (Ag), copper (Cu), and zinc (Zn), among whom Au exhibits a unique and tunable surface plasmon resonance (SPR).<sup>21</sup> Gold nanoparticles possess different size, shape, and aggregation ability. Previous reports on gold nanoparticle synthesis comprise the use of *Cassia auriculata*,<sup>22</sup> *Medicago sativa*,<sup>23</sup> *Aloe vera*,<sup>24</sup> *Pelargonium graveolens*,<sup>25</sup> *Tamarindo's indica*,<sup>26</sup> *Coriandrum sativum*<sup>27</sup> and *Cymbopogon citratus*.<sup>28</sup> Gold nanoparticles show high biocompatibility and have a wide range of medical applications,<sup>29</sup> drug delivery,<sup>30</sup> gene delivery,<sup>31</sup> and photo-thermal therapy.<sup>32</sup> Hence *Parthenium* plant was selected to mediate the synthesis of gold nanoparticles.

XRD is used to examine the crystal structure and phase purity of synthesized gold nanoparticles. In the present study the XRD pattern of synthesized gold nanoparticles using the extract of *parthenium hysterophorous* plant showed standard Bragg reflections (111) and (200) of face center cubic (FCC) lattice of gold. These results are in good agreement with the previous reports on green synthesis of gold nanoparticles that indicates similar Bragg's reflection for gold.<sup>33,34</sup> This XRD pattern is typical of pure gold nanocrystals with JCPDS card no. 04-0784.

Gupta et al., reported that<sup>35</sup> four different intense peaks at  $2\theta$  angle: 38.22, 44.42, 64.71, and 77.62 with Bragg reflections corresponding to (111), (200), (220), and (311) in biomass- associated gold nanoparticles. Alternatively, only a single prominent peak was observed at  $2\theta$  angle: 38.22 with a Bragg reflection corresponding to (111) in extracellular gold nanoparticles. Our present findings are consistent with earlier studies that used biological methods to synthesize gold nanoparticles using plant extracts.<sup>36-38</sup>

The UV-visible spectroscopy is carried out to evaluate the potential optical properties of the as-prepared gold nanoparticles using *parthenium hysterophorous* plant

extract. The basic property of gold nanoparticle is that it has characteristic wine-red color in solution. In the present study during synthesis of *parthenium* plant mediated synthesis of gold nanoparticles, red color change was observed, which confirms the formation of gold nanoparticles. Wani et. al.,<sup>39</sup> in his study stated that gold nanoparticles are known to exhibit at maximum in the range of 200 to 800 nm. This color formation belongs to the surface Plasmon vibration of the metal nanoparticles.

The red color is due to the SPR phenomenon of gold nanoparticles. SPR occurs when the incoming visible light strikes with the electron oscillating on the surface of the nanoparticle then at a certain wavelength, a resonance takes place where the frequency of the light matches with the frequency of the oscillation of the electron.<sup>40,41</sup> Hence SPR phenomenon is due to change in metal oxidation state where in Au<sup>+</sup> was reduced to Au<sup>0</sup> and is mediated by the biomolecules present in *Parthenium* plant extract. The gold nanoparticles absorption band at 534 nm observed in the present study is characteristic SPR of gold nanoparticles and is in good agreement with previous reports.<sup>42, 43, 44</sup>

The FT-IR spectrum in the present study indicates characteristic three bands at about 3327.21, 1625.99 and 1377.17<sup>cm</sup><sup>-1</sup>. The band at 3327.21 <sup>cm</sup><sup>-1</sup> corresponds to N-H stretching or O-H stretching vibration. The peak 1377.17 <sup>cm</sup><sup>-1</sup> corresponds to C-N stretching of aromatic amine group. The band at 1625.99 <sup>cm</sup><sup>-1</sup> corresponds to C=O stretching vibrations due to the carbonyl group in the proteins of amide I group of proteins.

Similar observations were reported in the previous study of S. Vijayakumar et al.,<sup>21</sup>. The intense broad absorption peak at 3451 <sup>cm</sup><sup>-1</sup> represents the O-H stretching vibrations of phenols and carboxylic acids. The peak located at 1636 <sup>cm</sup><sup>-1</sup> was assigned to the C=O stretching in carboxyl or C=N bending in the amide group. The band observed at 1387<sup>cm</sup><sup>-1</sup> was assigned to C-N stretching or the O-H bending and its shift to 1034<sup>cm</sup><sup>-1</sup> implicated the role of these groups in the interaction with chloroauric acid. Results in this study are consistent with the literature data.<sup>21, 45, 46</sup>

## CONCLUSION

A novel eco-friendly method was established to synthesize Gold nanoparticles without the aid of external energy. Formation of these gold nanoparticles was confirmed by spectroscopic techniques. Gold nanoparticles is a proven antimicrobial agent, biocompatible and nontoxic to human cells. Hence the established method can be further scaled up for gold nanoparticle synthesis for pharmaceutical use.

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