



## SYNTHESIS OF SILVER NANOPARTICLES

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### ABSTRACT

Widely Silver nanoparticles have been investigated because they exhibit unusual optical, electronic, and chemical properties, depending on their size and shape, thus opening many possibilities with respect to technological applications. The silver nanoparticle is one of the inorganic nano materials which is a good antimicrobial agents. The researcher found that the bactericidal nano materials have opened a new epoch in pharmaceutical industries. Silver nanoparticles are the metal of choice as they hold the promise to kill microbe's effectively and effect on both extracellularly as well intracellularly, the researchers by using different methods, manufactured silver nanoparticles with spherical, octahedral, tetrahedral, hexagonal, cubic, wire, coaxial cable, triangular prism, disc, triangular mark, belt, and shell shapes.

**Keywords:** silver nanoparticles, surface plasmon, chemicals reduction.

### 1. INTRODUCTION

There are different methods to synthesize silver nanoparticles, such as conventional temperature assisted process, controlled reaction at elevated temperatures, and microwave assisted process K. J. Sreeram *et al.*, [1] Chemical, physical synthesis methods and Bioinspired synthesis. Chemical and physical methods lead to the presence of some toxic chemical species adsorbed on the surface that may have adverse effects in medical applications. Bioinspired synthesis of nanoparticles provides advancement over chemical and physical methods. Microbes generally have a harder time developing resistance to silver than they do to antibiotics. V. Parashar *et al.*, [2] metallic nanoparticles shows unique properties such as excellent conductivity, chemical stability, and catalytic activity, etc. which are dependent on the particle size, size distribution and shape. Among all metals, silver has the highest electrical and thermal conductivity S. L-C Hsu, and R-T Wu [3]. The intrinsic properties of metal nanoparticles are mainly determined by their size, shape, composition, crystallinity, and structure. In principle, any one of these parameters could be controlled to fine-tune the properties of metal nanoparticles. D. Kim *et al.*, [4].

Green synthesis of silver NPs using , Parthenium leaf Vyom Parashar *et al.*, [2], citrus limon, latex of *Jatropha curcas*, starch, banana peel extract, leaf extract of *Rosa rugosa*, seed extract of *Jatropha curcas*, sucrose and maltose, *Hibiscus rosa sinensis*, cochlospermum gossypium honey and *D. carotas*, have been reported M. Umadevi [5].

### 2. METHODS FOR PRODUCE NANO SILVER

V. Parashar. *et al.*, [2] used fresh leaves of *Parthenium hysterophorus* L. weighing 25 g were thoroughly washed thrice in distilled water for 15 min, dried, cut into fine pieces and were boiled in a 500 ml Erlenmeyer flask with 100 ml of sterile distilled water up to 5 min and were filtered, Silver nitrate ( $\text{AgNO}_3$ ) 50 ml of fresh leaf extract was added into the aqueous solution of 1 mM Silver nitrate. Average size of the particles synthesized was 50 nm with size range 30 to 80 nm with

irregular shape. Due to our interest to get much smaller particles, above solution was centrifuged at a rate of 1200 rpm up to 15 minutes and investigated that particles present in the supernatant were nearly homogenous with average size of 7 nm silver ion complex (1mM) and leaf extract it was observed that precursors in the ratio of 1:1 gave best results.

S. L-C Hsu, and R - T Wu [3], dissolved  $\text{AgNO}_3$  in de - ionized water in a beaker to this solution, a protecting agent [poly (N - vinyl - 2 - pyrrolidone or thiosalicylic acid or triethylamine) was added. After being stirred, HCHO solution was then added to the solution. Subsequently, a promoter (triethylamine or pyridine) was added drop wise. The color of the solution turned from clear to black. After being stirred for 200 min at room temperature, the precipitates were washed several times with ethanol, followed by centrifugation (6000 rpm, 10 min), to remove unbound TEA. The particles were then dried at room temperature under vacuum for 24 h. The silver nanoparticles suspensions were prepared from the dried silver nanoparticles by re-dispersing them into alphaterpineol to prepare the nanoparticles in order to reduce the sintering temperature. As shown in Figure-2(a), when we used triethylamine as the reaction promoter and TSA as the protecting agent, the silver nanoparticles were successfully reduced from the  $\text{AgNO}_3$  precursor. During the preparation of Ag nanoparticle suspensions, the individual particles had a tendency to form large agglomerates through the van der Waals force or Coulomb's force. In order to prevent the agglomeration of small particles, we added TEA to the suspensions as the stabilizer. For TEA - protected silver nanoparticles, the agglomeration of small particles increased with the increasing TEA concentrations. Because the reduction reaction of  $\text{AgNO}_3$  by formaldehyde is slow without the addition of basic catalysts. A higher pH is favored for higher reducing power. In order to avoid the use of inorganic bases as the reaction promoter, which usually contains other unwanted metal ions, we chose organic bases, triethylamine or pyridine as the reaction promoter. These bases are easy to be washed out after reaction and will not contaminate the resulting silver nanoparticles.



Silver nitrate (>99%) and trisodium citrate dihydrate (99.0%) were purchased from Aldrich. All descriptions of water below refer to Nano pure deionized water (electrical resistance >18.4 MΩ-cm), produced from a Barnstead Nano pure water system. Silver nanoparticles were prepared by citrate reduction of silver nitrate. AgNO<sub>3</sub> (17.0 mg) was dissolved in 100 mL water in a 250 mL tri-neck flask. The solution was heated to boiling with a hemisphere heating mantle under vigorous magnetic stirring. After boiling for 2 minutes, an aqueous solution of sodium citrate (35 mM, 10 mL) was rapidly added to the flask. The solution gradually turned yellow within a few minutes, indicating the formation of Ag nanoparticles. The solution was kept boiling for an additional 6 minutes. After that, the heating mantle was removed, and the solution was allowed to cool. TEM imaging of the nanoparticles was carried out with a Tecnai F30 STEM/TEM microscope, which is equipped with a thermal Schottky field emission gun and operates at 300 kV. Images are acquired with a 4096x4096 pixel CCD (Gatan; pixel resolution for images in Figure-1 (zoom-in images) and Figure-3 is 0.5 nm). The as-prepared Ag colloids typically consist of a majority (~90%) of nearly spherical nanoparticles with an average diameter ~50 nm (standard deviation ~ 20%) and a fraction (5~10%) of nano rods with various aspect ratios. A 1% solution of starch was prepared. To this starch solution, silver nitrate (5 mM) was added. The resultant solution was stirred for 30 min. The transparent solution was taken for different methods of preparation of silver.

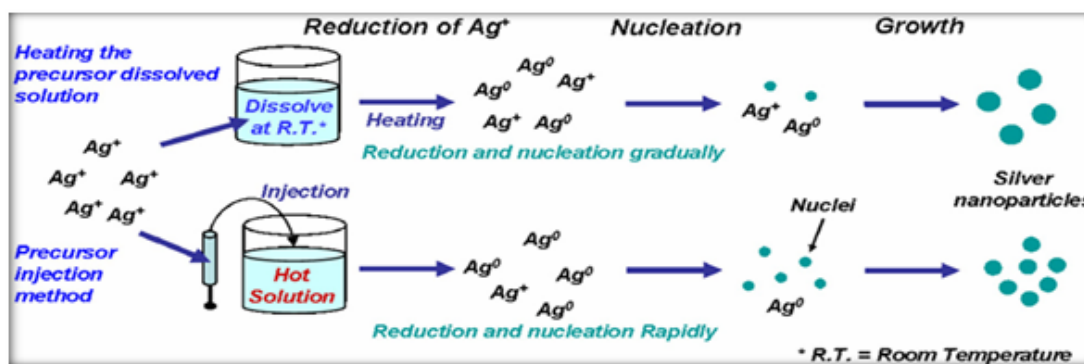
Synthesis of silver particles by heating the precursor solution Silver nitrate (99.9%, Aldrich), used as a precursor of Ag, was dissolved in ethylene glycol (EG, 99.9%, Aldrich) together with polyvinylpyrrolidone (PVP, Mw = 10 000, Aldrich), which protects the synthesized silver nanoparticles from agglomeration. This solution was stirred vigorously in a reactor fitted with a reflux condenser, followed by heating the precursor solution at temperatures of 100-150°C, at a constant heating rate of 1-7.5°C min<sup>-1</sup>. The reaction was maintained for 30 min at each reaction temperature. After the reaction was

completed, the solution was cooled to room temperature, and the silver particles were separated from the liquid by centrifugation and then repeatedly washed with ethanol. The resulting particles were dried at room temperature.

Synthesis of silver particles by injecting precursor solution all the chemicals used in the precursor injection method are identical to those described above, except for the injection of silver nitrate aqueous solution. First, PVP was completely dissolved in ethylene glycol and this solution was heated to the reaction temperature. Deionized water containing 40 wt% silver nitrate was injected into a hot PVP-ethylene glycol solution maintained at the reaction temperature. The particle recovery procedures are also identical to those of for the previous method D. Kim, S [4]. Fresh *D. carota* (carrot) was washed several times with de-ionized water. Then 170 g of *D. carota* was cut finely and crushed in a mixer grinder with 100 ml distilled water. The mixture was filtered to get the extract. 4 ml of *D. carota* extract was added to a vigorously stirred 30 ml aqueous solution of AgNO<sub>3</sub> (5×10<sup>-4</sup> M) and stirring continued for 1 min at room temperature.

The solution changed from light orange to dark red color which indicates the formation of silver nanoparticle. This procedure was repeated for 5 ml of *D. carota* extract with 30 ml aqueous solution of AgNO<sub>3</sub> (5×10<sup>-4</sup> M). It is found that reduction takes place rapidly with increasing addition of the extract. The absorption spectra of prepared silver nanoparticles were carried out in different days to find out the time variation (1<sup>st</sup>, 3<sup>rd</sup>, 6<sup>th</sup>, 9<sup>th</sup> and 12<sup>th</sup> day). The prepared silver NPs were dried at 200 °C. The dried powders were taken for the characterizations such as x-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR).

- There is no need to use high pressure.
- Low cost effective.
- Low energy.
- Low temperature.
- There is no toxic chemical.



**Figure-1.** Schematic illustration of the two synthesis method solution and injection of the precursor s: heating the precursor dissolved.



### 3. LITERATURE REVIEW

Vyom Parashar *et al.*, [2], they extract Parthenium leaf to synthesis silver nanoparticles from  $\text{AgNO}_3$ . the analysis of the product particles shows that they are ~50 nm in range and assembled in very irregular shape of variable morphology. S. L - C Hsu, and R - T Wu, [3] they use different organic compounds as the protecting agent and organic bases as the reaction promoter to prepare contamination-free suspensions of silver nanoparticles from  $\text{AgNO}_3$  with different particle sizes for ink jet printing application that's why they the final product is only pure silver nanoparticles without other metal ion impurities, so the silver nanoparticles product is suitable to use in IC devices. Using PVP, TSA and TEA as the protecting agents, we successfully prepared stable silver nanoparticles suspensions.

The average diameters of the nanoparticles were 15 nm, 7.99 nm, and 2.74 nm, when the protect agents were PVP, TSA and TEA, respectively. The resulting silver nanoparticles showed high crystallinity. The silver nanoparticle can be used to fabricate flexible electronics by ink-jet printing, because they have relatively low sintering temperatures. K.J SREERAM *et al.*, [1], they used Electron microscopy, photon correlation spectroscopy and surface plasmon resonance have been employed to characterize the silver nanoparticles synthesized, and the mixed starch solution (Starch acts as a reductant as well as a capping material to protect the nanoparticles surfaces and prevents the particles from aggregation), silver nitrate at an uncontrolled heating temperature around 80°C and controlled heating from 45-75°C. Compared to conventional methods, microwave assisted synthesis was faster and provided particles with an average particle size of 12 nm. Further, the starch functions as template, preventing the aggregation of silver nanoparticles.

D. Kim *et al.*, [4] used polyol process (heating the precursor solution to the reaction temperature or injecting an aqueous silver salt solution into hot ethylene glycol) to synthesized Spherical silver nanoparticles with various sizes and standard deviations. As in Figure-1 and they found that to induce rapid nucleation in a short period of time with a smaller size and a narrower size distribution, the injection of the precursor solution into a hot solution is an effective means.

M. Umadevi *et al.*, [5], used various concentrations of *D. carota* extract to prod used Biosynthesis of silver nanoparticles (NPs) at average size 20 nm with Spherical shape also present ascorbic acid in *D. carota* extract which is used as reducing agent.

The use of silver nanoparticles as antibacterial agent is relatively new. Because of their high reactivity due to the large surface to volume ratio, nanoparticles play a crucial role in inhibiting bacterial growth in aqueous and solid media. Silver containing materials can be employed to eliminate microorganisms on textile fabrics [6, 7] or they can be used for water treatment [8]. Contrary to bactericide effects of ionic silver, the antimicrobial activity of colloid silver particles are influenced by the

dimensions of the particles the smaller the particles, the greater antimicrobial effect [9]. Therefore, in developing routes of synthesis, an emphasis was made to control the size of silver nanoparticles. Silver nanoparticles have been produced using different methods: electrochemical method [10-12], thermal decomposition [13], laser ablation [14], microwave irradiation [15] and son chemical synthesis [16]. The simplest and the most commonly used bulk - solution synthetic method for metal nanoparticles is the chemical reduction of metal salts [17, 18]. In fact, production of nano sized metal silver particles with different morphologies and sizes [19] using chemical reduction of silver salts has been reported [20]. This synthetic method involves reduction of an ionic salt in an appropriate medium in the presence of surfactant using various reducing agents [21]. The dispersions of silver nanoparticles display intense colors due to the plasmon resonance absorption. The surface of a metal is like plasma, having free electrons in the conduction band and positively charged nuclei. Surface Plasmon resonance is a collective excitation of the electrons in the conduction band; near the surface of the nanoparticles. Electrons are limited to specific vibrations modes by the particle's size and shape. Therefore, metallic nanoparticles have characteristic optical absorption spectrums in the UV - V is region [22]. Antimicrobial susceptibility testing methods are divided into types based on the principle applied in each system. They include: Diffusion (Kirby - Bauer and Stokes), Dilution (Minimum Inhibitory Concentration) and Diffusion and Dilution (E - Test method). Antimicrobial susceptibility testing in the clinical laboratory is most often performed using the disc diffusion method. The Kirby - Bauer and Stokes' methods are usually used for antimicrobial susceptibility testing, with the Kirby-Bauer method being recommended by the National Committee for Clinical Laboratory Standards (NCCLS) (NCCLS, 03). The Kirby - Bauer method was originally standardized by Bauer *et al.*, (the so called Kirby - Bauer method). This method is well documented and standard zones of inhibition have been determined for susceptible and resistant values [23, 24]. The antibacterial characteristics of silver nanoparticles produced have been demonstrating by directly exposing bacteria to colloid silver particles solution [25].

The reduction of materials' dimension has pronounced effects on the physical properties that may be significantly different from the corresponding bulk material. Some of the physical properties exhibited by nano materials are due to (i) large surface atom, (ii) large surface energy, (iii) spatial confinement, and (iv) reduced imperfections [26]. A lot of interest has been created by the term "green nanotechnology". In a broad sense, this term includes a wide range of possible applications, from nanotechnology enabled, environmentally friendly manufacturing processes that reduce waste products (ultimately leading to atomically precise molecular manufacturing with zero waste); the use of nano materials as catalysts for greater efficiency in current manufacturing processes by minimizing or eliminating the use of toxic



materials (green chemistry principles); [27] the use of nano materials and nano devices to reduce pollution (e.g. water and air filters); and the use of nanomaterials for more efficient alternative energy production (e.g. solar and fuel cells). Use of plants in synthesis of nanoparticles is quite novel leading to truly green chemistry which provide advancement over chemical and physical method as it is cost effective and environment friendly easily scaled up for large scale synthesis and in this method there is no need to use high pressure, energy, temperature and toxic chemicals. Now days we are using bacteria, fungi for the synthesis of nanoparticles but use of leaf extract reduce the cost as well as we do not require any special culture preparation and isolation techniques [28]. Here we report synthesis of silver nanoparticles, reducing  $\text{Ag}^+$  ions present in the aqueous solution of silver nitrate by the help of onion extract. Through elaborate screening process involving number of plants, we observed that onion (*Allium cepa*) was potential candidate for synthesis of silver nanoparticles. We also study the antibacterial property of silver nanoparticles toward *E. coli*. Although, several previous reporter have studied the antibacterial activity of chemically synthesized silver nanoparticles but here we study the biologically (using onion extract) synthesized silver nanoparticles.

Biosynthetic methods have been investigated as an alternative to chemical and physical ones. Biologically synthesized silver nanoparticles could have many applications, as in spectrally selected coatings for solar energy absorption, as intercalation material for electrical batteries, as optical receptors, as catalysts in chemical reactions, as antimicrobials, and in bio - labeling [29] and other applications, such as spectrally selective coatings for solar energy absorption and intercalation material for electrical batteries [30], as optical receptors [31], catalysts in chemical reactions, bio labelling [32], etc. However, it is only recently that microorganisms have been explored as potential bio factory for synthesis of metallic nanoparticles such as cadmium sulphide, gold and silver [33]. To achieve the objective of developing simple and eco - friendly technology researchers in this field have turned to biological systems. Holmes *et al.*, have shown that the bacteria, *Klebsiella aerogenes* when exposed to cadmium ions resulted in intracellular formation of CdS particles in the range of 20-200 nm [34]. Sastry *et al.*, [33] have reported that fungus *Verticillium sp.* and, *Fusarium oxysporum* when exposed to gold and silver ions, reduced the metal ion fairly rapidly and formed respective metallic nanoparticles. Klaus *et al.*, have observed that the AG259, isolated from a silver mine, when placed in a concentrated solution of silver nitrate produced silver nanoparticles of well-defined size and distinct morphology within the periplasmic space of the bacteria [35]. Anovel biological method for synthesis of silver nanoparticles using *Verticillium* was proposed by Mukherjee and coworkers [36]; the two-step mechanism was suggested. The first step involves trapping of the  $\text{Ag}^+$  ions at the surface of the fungal cells. In the second step, the enzymes present in the cell, reduce silver ions. So, our aim is to biologically

synthesize silver nanoparticles using fungus *Trichoderma harzianum* and characterized by means of UV - vis spectroscopy and Transmission electron microscopy (TEM). Synthesis of silver nanoparticles by production of biomass to prepare the biomass for biosynthesis, the fungus obtained were grown aerobically in liquid broth containing malt extract powder, glucose, yeast extract, peptone. The culture flasks were incubated on room temperature at 27°C. The biomass was harvested after 120 hours of growth by sieving through a plastic sieve followed by extensive washing with sterile double-distilled water to remove any medium components from the biomass. Typically 15 g of biomass (wet weight) were brought into contact with 100 mL sterile double-distilled water for 48 hours at 27°C in an Erlenmeyer flask and agitated 150rpm. After incubation the cell filtrate was filtered by what man filter paper No.1 (Oakland, California, USA). After filtration the observed pH of cell filtrate was 7.2. Into these 100 mL of cell filtrate, a carefully weighed quantity of silver nitrate was added to the Erlenmeyer flask to yield an overall  $\text{Ag}^+$  ion concentration of 10 M, and the reaction was carried out under dark conditions [37].

Synthesis of silver nanoparticles using silver nitrate, 15 Gms of biomass was re - suspended in about 150mL milli Q water and incubated for 72 hours at 290C. Subsequently, the biomass was filtered using a what mann filter paper No1. The pH of filtrate was found to be 5.84. To the 150mL of filtrate, silver nitrate was added to give an overall concentration of 1mM (0.017g/100mL). The reaction was carried out in dark. The control with only the cell filtrate and without silver ion was run along with the test sample. Time dependent formation of silver nanoparticles was measured by using UV - visible spectrophotometer at 24, 48 and 72 hours and the absorbance was measured at wave lengths ranging from 200 to 600nm [38].

Synthesis of silver nanoparticles from  $\text{AgNO}_3$  solution (1 mM) was prepared by dissolution of 0.16 g in 1L water (Mill-Q). *L. camara* fruit was washed with water, crushed and filtered through 0.45  $\mu$  filter. The filtrate was further passed through 0.25  $\mu$  filter. The fruit extract of *L. camara* (10 mL) was added with 90 mL of 1mM  $\text{AgNO}_3$  solution at room temperature. The mixture was allowed to stand for approximately 1 hour until a yellowish brown colour solution was observed [39].

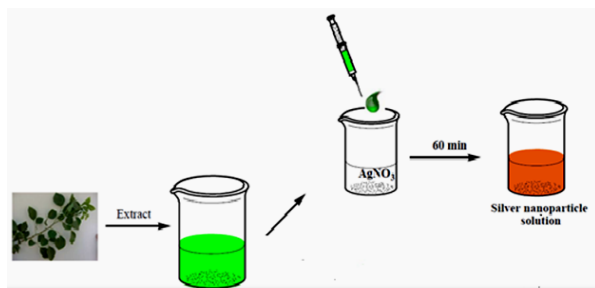
Green synthesis of silver NPs using citrus limon [40], latex of *Jatropha curcas* [41], starch [42], banana peel extract [43], leaf extract of *Rosa rugosa* [44], seed extract of *Jatropha curcas* [45], sucrose and maltose [46], hibiscus *Rosa sinensis* [47], *Cochlospermum gossypium* [48] and honey [49] have been reported. Green synthesis include on many methods such as Polysaccharide method, Tollens method, Irradiation method.

Reetz and Helbig, [50] were the first to describe in detail an electrochemical technique for the synthesis of nanoparticles, in which a metal sheet was anodic ally dissolved and the intermediate metal salt formed was reduced at the cathode, giving rise to metallic particles



stabilised by tetraalkylammonium salts. This work was successfully adopted for the electrochemical synthesis of the silver nanoparticles in acetonitrile containing tetrabutyl ammonium salts by Rodríguez - Sánchez *et al.*, [51]. Using a similar approach, silver nanoparticles were obtained by potentiostatic or galvanostatic polarisation of silver in ethanol solution by Starowicz *et al.*, [52]. In the work of Yin *et al.*, [53], Poly (N - vinyl -2 - pyrrolidone) (PVP) was essential in the process of electrochemical synthesis of silver particles. The main advantages of the electrochemical methods lie in the high purity of particles and the possibility of the nanoparticle size control by adjusting the current density without a need for expensive equipment or vacuum. The key to the success of electrochemical methods is the right choice of the chemical agents and the process conditions. However, as pointed out earlier (Rodríguez - Sánchez *et al.*, [51], the method has its limitations, as the deposition of silver on the cathode during the electrochemical process diminishes the effective surface available for particle production. As the entire cathode surface gets covered with the silver electrodeposits, the particle production comes to a halt altogether.

Synthesis of silver nanoparticles [54], the bioreduction of Ag (I) into the Ag (0), 5ml of leaf extract was added to 50 ml of 10<sup>-3</sup> M AgNO<sub>3</sub> solution. After the addition of leaf extracts both the solutions were kept in the incubator at 37°C (Figure-2).



**Figure-2.** Schematic representation for synthesis of silver nanoparticles.

#### 4. USES OF SILVER NANOPARTICLES

- a) In the coating industry, silver nanoparticles are employed for spectrally selective coating for solar energy [3]
- b) Pharmaceutical industries, and medicine [4]
- c) Ink jet printing application
- d) Biological labelling
- e) Nanodevice fabrication
- f) Surface Enhanced Raman Scattering (SERS)
- g) Photography
- h) Catalysis
- i) Biosensing
- j) Imaging
- k) Drug delivery
- l) Photonics
- m) Optoelectronics

#### 5. CONCLUSIONS

There are various methods for synthesis Silver nanoparticles with different shapes like spherical, octahedral, tetrahedral, hexagonal, cubic, wire, coaxial cable, triangular prism, disc, triangular mark, belt, and shell shapes which effect on the uses of these particles.

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