

# Methods for Obtaining Silver Nanoparticles - a Review

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**Abstract** - Silver has been part of active medical use for treatment and disinfection from ages, even in the present times. Particularly relevant today is the question of the effect of silver nanoparticles on bacteria and viruses, including against COVID-19. The study reviews distinct methods for producing silver nanoparticles. The comparison and preferences of different methods based on their classification and features are reviewed. The main goal of this study is to reveal the top-notch method and their benefits and prospects of the possibilities of the method - laser ablation, as a cost-effective and environment friendly technology for generating silver nanoparticles in an aqueous environment. The method of Pulsed Laser Ablation in Liquid for obtaining nanoparticles is explained in detail. The paper is divided into several main parts, starting with a briefing on silver and its nanoparticles and is followed by various methods for their production.

**Keywords** - Silver Nanoparticles, Laser Ablation, Material Processing, Nanoparticles, Pulsed Laser Ablation in Liquid.

## 1. SILVER NANOPARTICLES

Silver (Ag) is the remarkably vital antimicrobial agent usable before the introduction of antibiotics. [1] Silver Nanoparticles (Ag NPs) are nanoparticles (NPs) of Ag produced via nanotechnology of size varying between 1 – 100 nm in any dimension. [2] Ag NPs have been the research theme from the past several decades because of size, shape, antimicrobial effects, chemical stability, catalytic activity high conductivity, and other extraordinary characteristics. Several techniques for obtaining Ag NPs have been priorly researched such as laser ablation, gamma irradiation, electron irradiation, chemical reduction, photochemical methods, microwave processing, and synthetic biological methods. [3]

Nanotechnology is an essential field of present-day research dealing in design, synthesis, and administrating particle structures ranging, approximately, 1-100 nm. NPs have several applications in fields of medication, cosmetics, Eatables, substantial well-being, optics, biomedical sciences, chemical & space industries, electronics and many more. Synthesised Ag NPs are used for various pharmaceutical applications. Few are mentioned below: [3] [4]

- The remedy of ulcerative colitis & acne
- Remedy of dermatitis
- Inhibition of HIV-1 replication
- Molecular imaging of cancer cells
- Disclosure of viral structures (SERS & Ag nanorods)
- Antimicrobial effects against infectious organisms
- Varnishing of surgical mesh for pelvic reconstruction
- Varnishing of breathing mask patent (used in COVID-19 pandemic)

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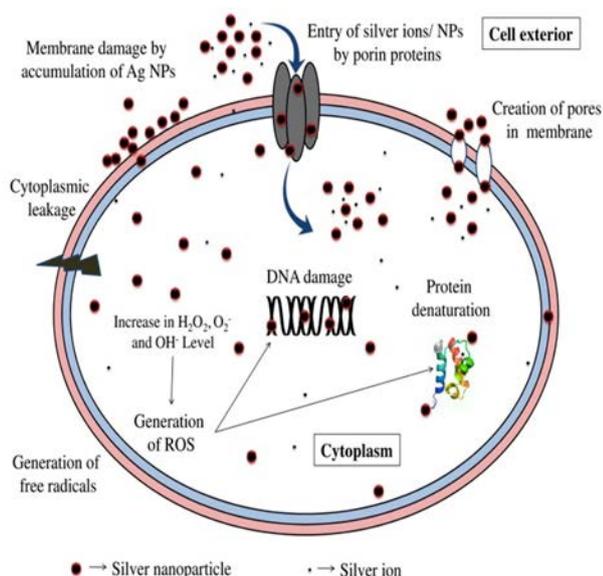


Figure 1: Bio-mechanism of antibacterial action of silver nanoparticles and silver ions [5]

Depending on reducing sources and mechanism, there are three prime methods to formulate Ag NPs. Physical processes usually operate with laser impulse energy to reduce Ag from volume to atoms and ions. Whereas, chemical and biological methods utilise reducing synthetic and organic agents, respectively, to reduce Ag ions from their harbinger into metallic Ag atoms. [6]

## 2. FACTS ABOUT SILVER AND AG NPs

Over the centuries, because of alteration in nomenclature, the complexity has remained among scientists, that the use of Ag, commercially, is accessible for over 100 years and are used as wound remedy, conductive/antistatic composites, catalysts, as a biocide, and many more is undeniable. The use of nanoscale Ag has a protracted, distinct, and complexed past, that is why recently, an exceptional study of AgNPs nomenclature has been conducted approximately over the past 120 years, which did not use “nano” classification. [7]

### A. FACTS [7] [1]

- In 702–705, Gabor listed the demand for silver nitrate being used as a medical agent, for the first time.
- In 980 A.D. the Persian physicist-Avicenna registered silver filings as a blood purifier, which countered the heart palpitations and offensive breath.
- In 1520, Paracelsus introduced a treatment, which is in use until present, used silver internally and applied silver nitrate as a caustic for the treatment of wounds.
- In 1614, Angelo Sala gave silver nitrate internally as a counterirritant, as a purgative, and for the treatment of brain infections.

- In 1889, M. C. Lea reported the fusion of a citrate-stabilized silver colloid and the obtained size for the particles was between 7 - 9 nm.
- At present, every year, an estimate of about 320 tons of nano-silver is manufactured and used universally.

## 3. METHODS FOR PROCESSING AG NPs

The synthesis of Ag NPs has been examined extensively and, over the years, several procedures have been offered for the processing. [8] Under two important techniques, NP Production is classified (figure 2):

- **Top-down Approach:** - In this, an external force applies to a solid bulk that guides it to breaking it into nanostructures.
- **Bottom-up Approach:** - It begins at the atomic scale & produces the NPs, till the required size & shape is obtained. [9]

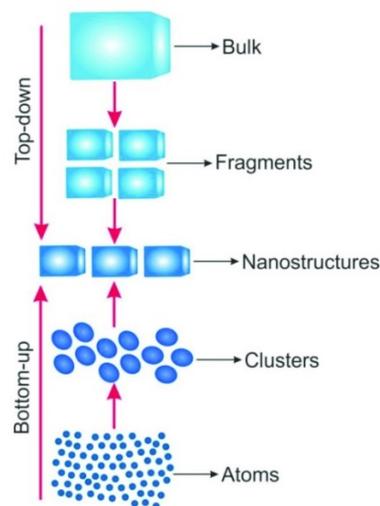


Figure 2: Schematic representation of ‘top-down’ and ‘bottom-up’ approaches for the synthesis of nanoscale materials. [10]

Different methods to synthesise Ag NPs; such as Physical, Chemical, and Biological Methods are explained below:

### A. PHYSICAL METHODS

Having non-synthetic liquids in the groomed thin films and the NPs distribution having consistency are some of the advantages of physically synthesised NPs as compared to that of chemical methods. [3] These can be categorised into different types of processes such as [8]

- **Arc Discharge** - The original element is vaporized via an arc discharge between electrodes pursued by condensation, nucleation, and production of NPs. [11]
- **High-Pressure Magnetron Sputtering** - It is a bottom-up mechanism & dwells in condensation of a super-saturated vapour in a flow of cold inert gas and this vapour atmosphere is procured by various methods as

thermal evaporation, laser ablation, magnetron plasma sputtering, etc. [12]

- **Laser Ablation** - The process separated the layers from solid bulk materials by utilising a laser beam for utmost accuracy. The laser beam irradiates the top layer, concluding that it has been disclosed to laser beam radioactivity. [13]

### B. CHEMICAL METHODS

The synthetic procedure became famous via several works by John Turkevich in the 1950s, a professor of chemistry in Princeton and a pioneer in the field of catalysis. The synthetic reduction by organic and inorganic reducing agents is the most appropriate and suggested method for generating the Ag NPs. Various reducing agents such as sodium citrate, ascorbate, elemental hydrogen, polyol process, are commonly used for silver ions (Ag<sup>+</sup>) reduction in all kind of solutions, whether it is liquid or non-liquid. These agents dwindle Ag<sup>+</sup> and manage the formation of silver (Ag<sup>0</sup>). [14] Protective agents are a priority to be used to stabilise the breaking-up NPs during metal NPs preparation and secure them from being consumed or bind onto NPs surfaces, escaping their aggregation. [15] The presence of surface-active agents like acids, amines or alcohol, comprising functionalities for cooperation with particle skin can stabilise the growth of particle, and conserve particles from sedimentation, accumulation, or losing surface properties. [3] The chemical methods can be differentiated into various types also: [16] [8]

- **Photochemical** – It involves Ultraviolet irradiation of the solid forerunner solution in an electron-contributing reagent. With this method, NPs are produced under solid-state status & at low temperatures. [17]
- **Microemulsion** – It is thermodynamically balanced colloidal diffusion of water in oil or vice-versa, balanced by a surfactant. Ultralow interfacial tension, large interfacial area, and being thermodynamically stable and monodispersed NPs, are the advantages of this method. [18]
- **Hydrothermal** - It is a solution reaction-based approach and can produce NPs in a room- to a very high- temperature. [19]
- **Chemical Reduction** - A prevailing synthesis method of Ag NPs, by organic & inorganic reducing agents such as sodium citrate, ascorbate, sodium borohydride (NaBH<sub>4</sub>), elemental hydrogen, N-dimethylformamide (DMF), & poly (ethylene glycol)-block copolymers are used for Ag<sup>+</sup> ions reduction. [3]
- **Electrochemical Mechanism** - A traditional method, utilizing a two-electrode system for the electrochemical synthesis. It has advantages over other bottom-up approaches (purity of NPs). [20]

### C. BIOLOGICAL METHODS

The concept of Green synthesis comprises polysaccharides, organic and irradiation mechanisms, which have preferences over traditional methods requiring synthetic agents associated with substantial harms. [21] It has been gaining interest because this process uses organic agents such as bacteria, fungi, yeast, and plant, making it a simple, cost-effective, and eco-friendly mechanism. It has a regular chemical reduction, with the reducing agent as natural agents. [8] Ag NPs can be natural synthesised (a.k.a. green synthesis) with the help of:

- **Bacteria** – It has great abilities to diminish heavy metal ions & is the most prescribed for manufacturing NP. E.g.: Pseudomonas stutzeri and Pseudomonas aeruginosa. [22]
- **Plants** - Plant crude extract consists of metabolites, which are highly important for the reduction of ionic into bulk metallic NPs production. E.g.: Acalypha indica, Alternanthera sessilis. [23]
- **Fungi** - These are interesting reducing agents for the biosynthesis of Ag NPs as they offer high tolerance to metals and are handled easily. The extraction of extracellular proteins from fungi contributes to the stability of the NPs. E.g.: Fusarium oxysporum. [24]
- **Algae** - These are uni- or multi- cellular organisms residing in the distinct surrounding. Synthesis at low temperature with higher energy efficiency, less toxicity, & the risk to the environment, are some of its advantages. E.g.: Chlorella vulgaris, Spirulina platensis. [25]

## 4. COMPARISON

From all the above-mentioned methods, the mechanisms for generating Ag NPs, Physical Methods can be preferred as a top-notch method after tabular comparison with other mechanisms, see table 1.

TABLE 1: COMPARISON BETWEEN VARIOUS MECHANISMS FOR THE SYNTHESIS OF SILVER NANOPARTICLES [8]

METHOD →	PHYSICAL	CHEMICAL	BIO
FEATURES ↓			
Type of Method	Green	Non-Green	Green
Environment Friendly	Yes	No	Yes
Hazardous Liquids	None	Yes	None
Time consumed	Timesaving	Long time	Long time
Synthesised using	Laser Ablation	Reducing agents	Bacteria, Fungi, etc
Resulted NPs	Pure and Uncontaminated	Chemically Uncontaminated	Organically Uncontaminated

## 5. TOP-NOTCH PREFERRED METHODS

Pulsed Laser Ablation in Liquid (PLAL) under physical methods has the top-notch priority for synthesising Ag NPs of metallic bulk in liquid because it is a green method and consumes the lowest time & finances as compared to

traditional mechanisms. [26] [27] It is also introduced as a bottom-up process because of nucleation, advancement, & gathering of clusters. With the interaction of ultrashort laser pulses ( $10^{-13}$  -  $10^{-8}$  s), the generation of nanostructures from the solid bulk material substrate, is defined as laser ablation. Materials can be ablated in a vacuum, gas, or liquid mediums. [9] [28] [29] [40]

Moreover, the Ag NPs produced by laser ablation have rare properties as they are pure and uncontaminated metal colloids. The obtained efficiency and the characteristics of Ag NPs generated are based on several specifications, including the laser's wavelength ( $\lambda$ ), pulse duration and ablation time operating on the marker. [30] [31] This method is of high-quality and highly preferred because it produces metal colloids without any synthetic or organic assistance. Crucial specifications such as laser wavelength ( $\lambda$ ), pulse duration, & the interaction of individual NP with a laser beam inside the liquid, influences the characteristics of NPs obtained in the liquid solution. NPs size distribution in aqueous solution is controlled either by spatial profiling of laser beam intensity or fine balancing of  $\lambda$ . [32] Some of the important parameters are briefed below:

- a) **Pulse Duration:** PLAL is used for the generation of NP with pulse duration varying from Femto- to nano-seconds. NPs produced by PLAL, originate due to sputtering of the molten layer by the recoil pressure of the liquid around the targeted element. The temperature "T" of the laser spot is expressed by the Simple heat balancing equation, i.e.:

$$T \approx \frac{A_j}{c_p h} \quad (1)$$

Here, **A** - absorptivity of the targeted material (**A** = 1 - **R**) [**R** - reflectivity coefficient]

**c** - heat capacity of the targeted material,

**$\rho$**  - density of the target material,

**h** - heat diffusion length inside the targeted material,

The heat diffusion length "h" dependent on the heat diffusivity of the targeted element, expressed as:

$$h \propto \sqrt{at_p} \quad (2)$$

wherein,  $a = \frac{k}{c}$ , [k - heat conduction coefficient of the target, & " $t_p$ " - laser pulse duration].

Larger the  $t_p$ , thicker is the layer of the solid bulk which is fiery by absorbed laser energy. Also, for heating & evaporation of a liquid adjacent to the laser spot, an amount of energy is consumed which is as small as to be neglected in comparison to the one absorbed because of lower thermal conductivity of liquids. [32]

- b) **Laser Wavelength:** Any available wavelength is relevant when PLAL is considered. Undoubtedly, produced NPs can absorb laser radiation. Most of the

NPs absorb in UV range because of which the use of excimer UV lasers is least preferred. [32]

- c) **Repetition Rate:** With each laser beam pulse (absorbed laser energy can melt the material), NPs are thrown out from the solid target. Therefore, the production of NPs is higher, provided the repetition rate of laser pulses is higher too. [32]

In the PLAL method, laser radiation is focused onto a solid target immersed in a liquid. It is assumed that the liquid is transparent at the laser wavelength, otherwise, the focalization of the beam would be problematic due to absorption of laser radiation in it. The simplest way is working with the free surface of the liquid, which allows avoiding additional reflection at the interface "covering glass/air". However, the use of volatile liquids, such as acetone, ethanol, etc., requires covering the liquid with a window that is transparent at the laser wavelength. [32]

The process of laser ablation works when - nanostructures of noble metal are produced by pulsed laser ablation in liquid, see figure 1. [33] In the process, a metallic Ag target is placed in distilled water under a pulsed laser and is synthesised by the laser ablation. Various lasers can be used in the process such as Solid, Gas or chemical lasers of several wavelengths varying from picosecond to femtosecond. Laser ablated NPs have unique properties and are non-reproducible by any existing methods. [34]

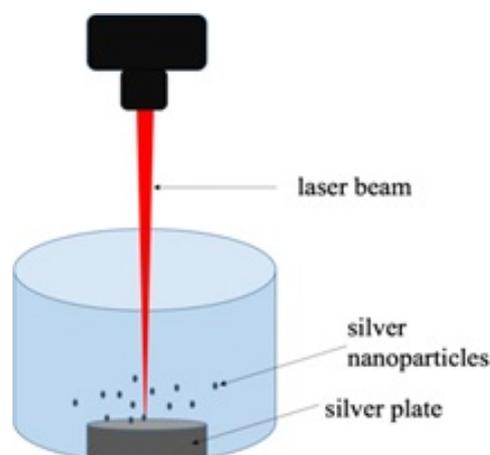


Figure 3: Systematic showing the working of the laser ablation process for producing silver nanoparticles [6]

To generate Ag NPs out of a metallic silver target through laser ablation, high-power pulsed lasers are required. Eventually, the ablation process relies on the physical features of the element used. The ablation rate is affected by several laser specifications including wavelength ( $\lambda$ ), fluence, pulse duration, repetition rate, the light absorption efficiency of the marked material, transmission, and chemical composition of the liquid. These laser parameters can be used to tune several NP features, such as size, shape, surface properties, aggregation state, solubility, structure, and chemical composition, see table 2. [8]

TABLE 2: COMPARISON OF DIFFERENT PARAMETERS FOR PREPARING SILVER NANOPARTICLES VIA LASER ABLATION. [35] [36] [37]

Wavelength (λ)	532 nm	800 nm	1064 nm
Laser	Nd: YAG (ns)	Ti: Sapphire (fs)	Nd: YAG (ns)
Mean Diameter	2-3 nm	400 nm	2-5 nm
Absorbance	0.28	> 0.05	< 0.1
Quantity	170 ml (pure water)	5 ml (deionised water)	25 ml (pure water)

When compared, the formation efficiency and the size of colloidal pieces, was greater than those generated by a nanosecond laser. The nanosecond pulses have higher formation efficiency as of the femtosecond pulses. And the produced colloids by nanosecond pulses are more scattered than that of femtosecond pulses. Moreover, it was discovered that the ablation performance for femtosecond ablation in distilled water was diminishing than that in the air. In contrast, in the case of nanosecond pulses, the ablation energy was similar in both liquid and air. It was noted that self-absorption efficiency of Ag is superior at lower wavelengths than that of higher wavelengths, which concludes that the ablation energy with shorter-wavelength laser is lessened than that of a longer-wavelength laser. [35] [38] [41] [42]

#### SUMMARY

As Ag NPs possess unique properties, they are usable in various fields of science and technology, such as medicine, ecology, food industry, pharmacy, biotechnology, microelectronics, physics, and others. This is because Ag NPs are chemically stable and antimicrobial agents have a significant protective effect against microbial pathogens. Also, Ag NPs are not toxic to the human body in low concentrations. This makes them an integral part of the composition of various products with antimicrobial action.

These characteristics necessitated the active search and development of new technologies to produce Ag NPs with different shapes and sizes. Various teams from leading research laboratories study and improve the mechanisms of Ag NPs extraction, using chemical / synthetic reduction, laser ablation, electron beam exposure, concentrated microwave exposure, and many more.

Bio methods for NP synthesis have new possibilities for using organic and stabilizing agents. The proposal to use bio-organisms to extract NPs can contribute to the ecological purity of the environment. The biological activity of Ag NPs depends on factors including surface chemistry and morphology, size, shape, coating agent, NP agglomeration and dissolution rate, particle reactivity in solution, and ion release efficiency.

The flexibility of the chemical methods of Ag NPs and the rapid incorporation of Ag NPs into individual

environments have motivated researchers to continue to study the mechanistic aspects of the antimicrobial, antiviral and anti-inflammatory effects of these NPs. This is achieved mainly through studies leading to a change in the form, size of Ag NPs by changing the reaction conditions, as well as the reagents and stabilizers used.

The laser ablation method to generate pure and uncontaminated metal NPs with unique properties, without the use of chemicals - organic or inorganic agents, has an exceptional environmental effect. In this method, laser parameters such as wavelength (λ) and laser intensity turn out to be the most characteristic responsible for NP production, for example, green lasers show higher formation efficiency than infrared lasers, and nanosecond pulses of the laser cause a strong thermal effect.

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