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COMPARATIVE CHARACTERISTICS OF Ag NANOPARTICLES SYNTHESIZED BY DIFFERENT APPROACHES

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Abstract

Nanoparticles have been the subject of researchers due to their unique properties. The importance of nanoparticles in technological fields is related to their adaptable properties. They are synthesized by reducing metal ions to uncharged nanoparticles using reducing agents. However, in recent years there have been several initiatives to develop green technology that uses natural resources instead of hazardous chemicals to produce nanoparticles. In green synthesis, biological methods are used for the synthesis of nanoparticles because biological methods are environmentally friendly, safe, economical, uncomplicated and highly productive. So, in this review we discuss different approaches in nanoparticles' synthesis and comparative characteristics of nanosilver particles, synthesized by physical, chemical, and biological methods.

Keywords: nanotechnology, synthesis methods, green synthesis, silver nanoparticles.[1]

1.İntroduction

In the last few decades, more attention has been paid to the synthesis of metallic nanoparticles due to their unique optical and electrical properties. The surface plasmon resonance exhibited by metallic nanoparticles is one of their most important properties, and these optical properties make them unique. Metal nanoparticles have proven to be very efficient and useful in electronics, photonics and medicine. The properties of metal nanoparticles vary according to their size, shape and morphology.

Many researchers have shown great interest in the synthesis of silver nanoparticles due to their enhanced antimicrobial activity and use as anticancer agents. In its pure form, silver has the highest electrical and thermal conductivity of all metals and the lowest contact resistance. There are studies and reports about negative effects of nano-silver on humans and the environment [1]. However, a green approach offers a toxic chemical-free and environmentally friendly synthesis of AgNPs.

Today, we live in the era of nanoscience, which plays an important role in all aspects of life. Silver nanoparticles (AgNPs) are widely studied compared to other noble metal nanoparticles due to their optical, antimicrobial, anticancer, antioxidant properties, as well as their low cost. Different methods can be used to synthesize AgNPs. The most commonly used route is the chemical reduction of Ag^{+} ions from an aqueous solution of silver nitrate by using various reducing agents, such as ascorbic acid, hydrazine, ammonium formate, dimethylformamide, and sodium borohydride. Although the chemical reduction method is fast, it is toxic and has a negative impact on the environment. Taking into account that green synthesis is

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environmentally friendly, nontoxic, and compatible with green chemistry protocols, we chose to focus on it, because it opens up a wide range of modern nanoscience research. Metallic NPs can be synthesized biologically using various plants and their extracts which are easily available in huge quantities. The plants and their extracts are safe to handle, less toxic and eco-friendly [2].

Recently, nanomaterials have emerged as a period of development in various research fields due to their exceptional physical, chemical, and electronic properties. These properties are important for various scientific fields such as catalysis, electronics, targeted drug delivery, and water research. In general, the term "nanoparticles" is used to describe particles ranging in size from 1 to 100 nanometers, although in the field of biotechnology this is usually expanded to include particles up to 500 nanometers in size [3]. it is a parameter that makes it unique from other materials and can create certain deep physic-chemical properties. Typically, the prepared nanoparticles are metallic in nature and exhibit an effect known as "Surface Plasmon Resonance" which plays an important role in the quantum mechanical effects of light in the UV-Visible regions and leads to unique optronic/optoelectronic properties. The size or shape of a nanoparticle affects its interparticle interactions as well as absorption properties [4]. Due to their exceptional properties, nanoparticles are widely used in various biomedical applications too [5, 6].

Numerous developments in nanoscale science have produced many nanoscale materials to improve related research, and thus a variety of valuable nanoscale materials have been produced on a commercial scale. It is believed that in the future, nanoscale materials and related products will be helpful in everyday life. It is equally important that it has the ability to interact with various biological molecules inside of the cells and on their surface. The ability to enter cells allows these molecules to control various physicochemical and biochemical processes of the cell.

The method of synthesis of nano-sized materials is an important chemical process. Currently, both chemical and physical methods are used to prepare nanoscale materials, but these may not be optimal choices due to high cost and potential environmental pollution. Consequently, alternatives to these existing methods that are environmentally friendly (green synthesis) during the entire production process must be developed for the synthesis of nanoscale materials, which are of interest to researchers worldwide. Conventional synthesis procedures (both physical and chemical) are usually performed under extremely harsh conditions. In contrast, biological procedures are generally carried out at ambient temperature and pressure, resulting in simplicity, energy efficiency, and reduced toxicity or harm to both humans and the environment [7].

Given these advantages, a variety of biological resources, including bacteria, fungi, yeast, plants, and algae, are used to synthesize intracellular nanoscale materials. Although nanoparticles are synthesized by biological methods (biosynthesis), their synthesis mechanism remains elusive. However, these methods are well established and commonly used for the synthesis of nanoscale materials because they are more cost-effective and environmentally friendly than traditional methods [8]. Biological methods harness the power of renewable resources such as plants and microorganisms to act as reducing agents to stabilize nanomaterials, eliminating the need for chemical additives. Synthesized nanoparticles are comprehensively characterized using various analytical techniques to confirm their composition and physicochemical properties. After synthesis, various spectroscopic and microscopic methods play an important role in characterizing the synthesized nanoparticles. For example, UV-Vis spectrophotometry is used to evaluate their optical properties, while FTIR spectroscopy helps to identify functional groups present on the surface of nanoparticles. Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) are used for detailed analysis of their size, shape and structure. In addition, Dynamic Light Scattering (DLS) and Zeta potential measurements are used to determine the size and surface charge of the nanoparticles. These techniques provide valuable insights into the properties and behavior of synthesized nanoparticles.

2. Synthesis of nanoparticles

Regarding synthesis of nanoparticles, we generally can say that two approaches can be used to synthesize nanoparticles [9]: “top-down” approach - here, nanoparticles are produced using physical methods such as grinding or abrading the material [10, 11] and "bottom-up" approach - nanoparticles are formed from the "building blocks" of atoms or molecules, resulting in more complex compounds (Fig.1). Using this approach, three alternatives are identified.

- Chemical synthesis: A method of forming molecules or particles by reacting substances used as raw materials.
- Self-assembly: A technique in which atoms or molecules arrange themselves through physical and/or chemical interactions.

- Positional assembly: Atoms, molecules, and aggregates are deliberately manipulated and placed individually. However, this method is extremely laborious and unsuitable for industrial applications.

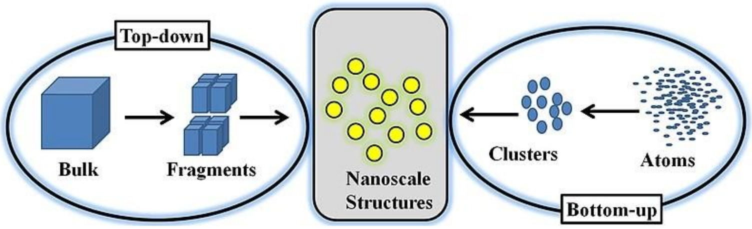

Figure 1. Illustration of the top-down and bottom-up approach for making nanoparticles. [Vicente Neto / CC BY 4.0]

A bottom-up approach is preferred because no special equipment is required and the time to obtain nanoparticles is shorter. Green synthesis is gaining relevance in the production of nanoparticles within the "bottom-up" approach [12]. The use of microorganisms such as plant species, algae, bacteria or fungi is one of the most used resources for this procedure. Various compounds derived from plants or microorganisms, including polyphenols, alkaloids, carbohydrates, proteins, play an important role in the synthesis of nanoparticles [13, 14]. It should be noted that not only the biological resources (plants, algae, or microorganisms) used to carry out the synthesis, other factors such as metal ion concentration, pH, reaction time, and temperature also affect the shape and size of nanoparticles [13, 15].

In general, the green synthesis of nanoparticles is a stepwise process [13].

Initial stage: In addition to the precursor salt, which is a source of metal ions, obtaining a reaction medium containing an aqueous extract of one or more parts of the plant species or a culture medium for the growth of microorganisms.

Activation stage: Chemical reduction of metal ions and formation of nucleation centers occur where nanoparticles appear and grow.

Growth phase: Small nanoparticles spontaneously grow into larger particles to form aggregates, which are affected by factors such as temperature, concentration and type of compounds, pH and reaction time.

Termination phase: The final shape of the nanoparticles is determined and the compounds involved in the reaction help to stabilize and enhance their properties.

3. Synthesis of silver nanoparticles

Taking into account the wide ranges of the applications of Ag nanoparticles, let's discuss different methods of its synthesis (Fig. 2).

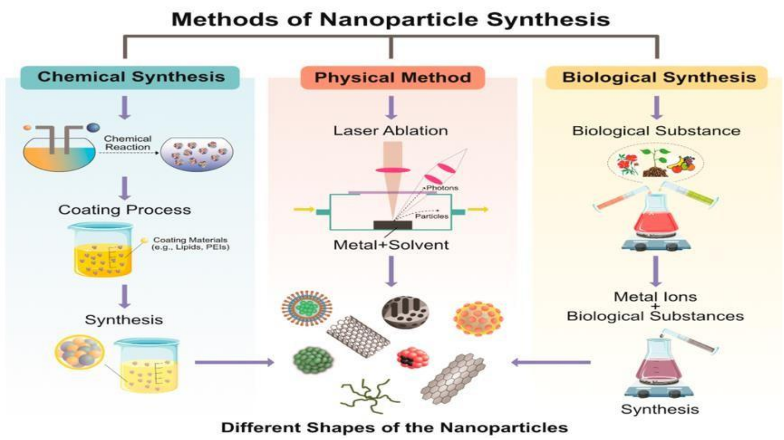

Figure 2. Schematic view of the commonly used nanoparticle synthesis methods [Jeyaraj et al., 2019]

Physical methods. Evaporation-condensation and laser ablation are the most important physical approaches. The absence of solvent contamination and uniformity of nanoparticle distribution in the prepared thin films is an advantage of physical synthesis methods compared to chemical processes. The physical synthesis of silver nanoparticles using a tube furnace at atmospheric pressure has some disadvantages, such as the tube furnace takes up a lot of space, consumes a large amount of energy by raising the ambient temperature around the source material, and takes a long time to achieve. Moreover, a typical tube furnace requires energy consumption of more than several kilowatts and a preheating time of several tens of minutes to reach a stable operating temperature [16, 17].

Silver nanoparticles can be synthesized by laser ablation of metallic bulk materials in solution [18, 19, 20, 21, 22]. The ablation efficiency and characteristics of the produced nano-silver particles depend on many parameters, including the wavelength of the laser hitting the metal target, the duration of the laser pulses (in the femto-, pico- and nanosecond mode), and the laser fluence.

An important advantage of the laser ablation technique compared to other methods for the production of metal colloids is the absence of chemical reagents in the solutions. Therefore, pure and uncontaminated metal colloids can be prepared by this technique for further applications [23]. Silver nanospheroids (20–50 nm) were prepared by laser ablation in water with femtosecond laser pulses at 800 nm [24]. The formation efficiency and sizes of colloidal particles were compared with colloidal particles prepared by nanosecond laser pulses. As a result, the formation efficiency for femtosecond pulses was significantly lower than that for nanosecond pulses. Colloids prepared with femtosecond pulses are less size dispersed than colloids prepared with nanosecond pulses. Furthermore, it was found that the ablation efficiency for femtosecond ablation in water is lower than that in air, while the ablation efficiency in the case of nanosecond pulses is similar in both water and air.

Tien and co-workers [25] used an arc discharge method to prepare a suspension of silver nanoparticles in deionized water without any added surfactants. In this synthesis, silver wires (Gradmann, 99.99%, diameter 1 mm) were immersed in deionized water and used as electrodes. Siegel and co-workers [26] demonstrated the synthesis of silver nanoparticles by direct metal spraying in a liquid medium. A method combining the physical precipitation of a metal into propane-1,2,3-triol (glycerol) provides an interesting alternative to time-consuming, wet-based chemical synthesis methods. The silver nanoparticles have a round shape with a mean diameter of about 3.5 nm and a standard deviation of 2.4 nm.

Chemical methods. The most common approach for the synthesis of silver nanoparticles is chemical reduction with organic and inorganic reducing agents. Generally, various reducing agents such as sodium citrate, ascorbate, sodium borohydride ($NaBH_4$), elemental hydrogen, polyol process, Tollens reagent, N,N-dimethylformamide (DMF) and poly(ethylene glycol)-block copolymers are used for reduction. These reducing agents reduce Ag^+ to form metallic silver (Ag^0), followed by aggregation into oligomeric groups. These clusters eventually lead to the formation of metallic colloidal silver particles [27, 28, 29]. The presence of surfactants containing functionalities (eg, thiols, amines, acids, and alcohols) to interact with particle surfaces can stabilize particle growth and protect particles from sedimentation, aggregation, or loss of surface properties.

Polymer compounds such as poly(vinyl alcohol), poly(vinylpyrrolidone), poly(ethylene glycol), poly(methacrylic acid) and polymethyl methacrylate have been reported to be effective protective agents to stabilize nanoparticles. Kim and co-workers [30] reported the synthesis of spherical silver nanoparticles with controlled size and high monodispersity using a polyol process and a modified precursor injection technique. In the precursor injection method, the injection speed and reaction temperature were the important factors for the production of small uniform size silver nanoparticles.

Silver NPs with a size of 17 ± 2 nm were obtained at an injection rate of 2.5 mL/s and a reaction temperature of 100 °C. Injecting the precursor solution into the hot solution is an effective means to induce rapid nucleation in a short period of time, enabling the preparation of silver nanoparticles with smaller size and narrower size distribution. Zhang and coworkers [31] used hyperbranched poly(methylene bisacrylamide aminoethyl piperazine) with terminal dimethylamine groups (HPAMAM-$N(CH_3)_2$) to produce silver colloids. The amide moieties, piperazine rings, tertiary amine groups, and hyperbranched structure in HPAMAM-$N(CH_3)_2$ are important for its effective stabilizing and reducing ability. Chen and co-workers [32] showed the formation of monodisperse silver nanoparticles using a simple oleylamine-liquid paraffin system. It has been reported that the formation process of these nanoparticles can be divided into three stages: growth, incubation and Oatwald ripening stages. The higher paraffin boiling point of 300°C provides a wider range of reaction temperatures and allows for effective control of silver nanoparticle size by changing the heating temperature alone without changing the solvent. Moreover, the size of colloidal silver nanoparticles can be adjusted not only by changing the heating temperature or ripening time, but also by adjusting the ratio of oleylamine to silver precursor.

Green synthesis. As discussed in the introduction, the synthesis of nanoscale materials using “green” processes is relatively cost-effective and environmentally friendly because non-toxic chemicals are used throughout the process. Therefore, the use of stabilizers and reducing agents of biological origin, such as microbial organisms, fauna, and various other resources, is a sustainable way to produce nanoscale materials. Despite being cost-effective and environmentally friendly, the main factors stimulating the green synthesis of nanoscale materials, the "stability" of the produced material, have attracted the attention of researchers worldwide. Although the methods involved in green synthesis are relatively diverse, the living organisms involved in this process usually simply react with various salts (metals) and reduce them to nano-sized materials that can be used for various purposes only according to their respective characteristics. Both microbial and plant-mediated approaches are used to synthesize nanoscale materials. Microbe-mediated constructions involve their inherently complex biochemical mechanisms, which lead to well-defined nanoparticles of different chemical compositions, shapes, and sizes. However, scaling up can sometimes be difficult with microbial preparations. This deficiency can be easily overcome by using plant-based extracts and consequently production rates can be increased. Plant extracts are more efficient than microbes in terms of production rate. They reduce metal ions faster than microbial organisms and produce nanoscale materials that are very stable [33]. Plants contain various compounds (ie, alkaloids, flavonoids, phenol, tannin, alcohol) that have the ability to reduce metal ions.

Plant-based synthesis of nanoparticles, on the other hand, is faster, safer, and lighter; works at relatively low temperatures and requires only environmentally safe components. Plant-derived nanoparticles have attracted more attention due to the increased interest in environmentally friendly products [34]. In addition, the synthesis of nanoparticles using plants offers other advantages such as the use of safer solvents, reduced use of hazardous reagents, milder reaction conditions making them suitable for use in medical, surgical and pharmaceutical applications. In addition, the physical requirements for their synthesis, including pressure, energy, temperature, and constituent materials, are insignificant.

Synthesis of nanoparticles using plants is very economical and thus can be used as an economical and cost-effective alternative for large-scale production of nanoparticles [35]. *Camellia sinensis* (green tea) extract was used as a reducing and stabilizing agent for the biosynthesis of silver nanoparticles [36]. Phenolic acid-type biomolecules (e.g., caffeine and theophylline) present in *Diospyros kaki* L extract appeared to be responsible for the formation and stabilization of silver NPs. Black tea leaf extract has also been used in the production of silver NPs [37]. The nanoparticles were stable and had different shapes such as spheres, trapezoids, prisms, and rods. Polyphenols and flavonoids seemed to be responsible for the biosynthesis of these nanoparticles.

Plant extracts from clover (*Medicago sativa*), lemongrass (*Cymbopogon flexuosus*) and geranium (*Pelargonium graveolens*) served as green reagents in the synthesis of silver nanoparticles. Moreover, ultrastable silver nanoparticles (16–40 nm) with high density were rapidly synthesized by reducing silver ions with *Datura metel* leaf extract [38].

Song and colleagues elucidated that leaf extracts of *Pinus desiflora*, *Diospyros kaki*, *Ginko biloba*, *Magnolia kobus*, and *Platanus orientalis* extracellularly synthesized stable silver nanoparticles with an average particle size ranging from 15 to 500 nm. In *M. kobus* and *D. kaki* leaf extract, the synthesis rate and final conversion to silver NPs were faster when the reaction temperature was increased. However, the average particle size produced by D. kaki leaf extract decreased from 50 nm to 16 nm when the temperature increased from 25°C to 95°C (39). The researchers also showed that only 11 min was required for more than 90% conversion at a reaction temperature of 95°C using M. kobus leaf extract [29]. It was further demonstrated that leaf extracts from the aquatic medicinal plant Nelumbo nucifera were able to reduce silver ions and form silver NPs (45 nm) of different shapes [40].

Capsicum annuum can also be used for the green synthesis of silver nanoparticles [16]. Proteins with amino groups reduce silver ions and play an important role during synthesis. After interacting with silver ions, the secondary structure of the protein was found to be changed, the crystalline phase of the nanoparticles changed from polycrystalline to single crystal and their size increased with increasing reaction time. A recognition-reduction-limited nucleation and growth model was proposed to explain the possible formation mechanism of silver nanoparticles in Capsicum annuum L. extract [41].

Spherical silver nanoparticles (40–50 nm) were produced by Euphorbia hirta [42] leaf extract. These nanoparticles have a potential and effective antibacterial property against Bacillus cereus and Staphylococcus aureus.

Acalypha indica (*Euphorbiaceae*) leaf extracts produced silver nanoparticles (20–30 nm) within 30 min [43]. These nanoparticles showed excellent antimicrobial activity against waterborne pathogens, E. coli and V. cholerae (minimum inhibitory concentration (MIC) = 10 μg/ml).

Furthermore, silver nanoparticles (57 nm) were obtained when 10 mL of Moringa oleifera leaf extract was mixed with 90 mL of 1 mM aqueous $AgNO_3$ and heated at 60–80 °C for 20 min. These nanoparticles have shown significant antimicrobial activity against pathogenic microorganisms including Staphylococcus aureus, Candida tropicalis, Klebsiella pneumoniae, and Candida krusei [44].

Cotton fibers loaded with biosynthesized silver nanoparticles (~20 nm) using natural extracts of Eucalyptus citriodora and Ficus bengalensis have been reported to have excellent antibacterial activity against E. coli. These fibers had potential for use in burn/wound dressings, as well as in the production of antibacterial textiles and finishing materials [45]. Garcinia mangostana leaf extract can be used as a reducing agent to biosynthesize silver nanoparticles (35 nm).

These nanoparticles had highly effective antimicrobial activity against E. coli and S. aureus [46]. It was reported that Ocimum sanctum leaf extract could reduce silver ions to crystalline silver nanoparticles (4-30 nm) within 8 minutes of reaction time. O.sanctum leaves contain ascorbic acid, which may play an important role in the conversion of silver ions into metallic silver NPs. These nanoparticles showed strong antimicrobial activity against E. coli and S. aureus [47].

Green synthesis of silver nanoparticles with Cacumen platycladi extract was investigated [48]. Reducing sugar and flavonoids in the extract appeared to be responsible for the reduction of silver ions, and their reducing capacity increased at 90°C, leading to the formation of silver nanoparticles (18.4 ± 4.6 nm) with a narrow size distribution.

4. Conclusion

Thus, the wide application of nanotechnologies determines the urgency of conducting research in the direction of improving the ways of synthesis of NPs and obtaining more ecologically safe metal NPs. We believe that the use of green technologies for this purpose will serve to minimize the potential negative impact of nanotechnologies on biodiversity and the ecological situation.

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