

Review

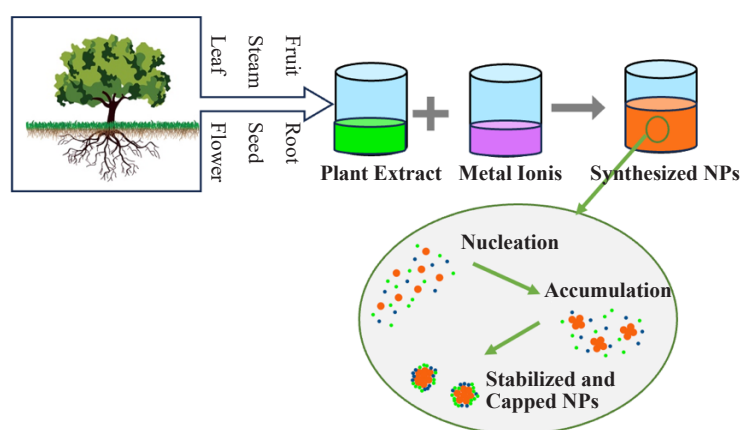
Sustainable Synthesis of Silver Nanoparticles Using Plant-Based Waste Biomass for the Removal of Cationic Dyes-A Review

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Graphical Abstract:



Abstract: Silver nanoparticles (AgNPs) have been the subject of researchers due to their unique properties such as optical, antimicrobial, and electrical properties depending on size and shape. This review focuses on their green synthesis of nanoparticles, metal nanoparticles and mainly AgNPs. Green chemistry synthesis method has attracted great attention as an alternative method in recent years because it is more energy efficient, safer and less toxic. At the same time, this study gives a general idea about the preparation of AgNPs by green synthesis method using lignocellulosic biomass. One of the main uses of lignocellulosic wastes in a circular economy is to introduce renewable resources into the market and on the other hand, to promote the valorization of waste. Moreover, lignocellulosic raw materials expand the sustainability possibilities of industrial processes as a result of their low cost and low environmental impact. On the other hand, dyes, which are wastes from different industries such as textile, plastic, food and paper, are the main cause of water pollution. This study provides a detailed investigation of the removal performance of AgNPs obtained using different lignocellulosic biomass for environmentally harmful cationic dyes.

Keywords: silver nanoparticle, nanotechnology, green synthesis, cationic dye

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1. Introduction

Owing to population growth and globalization, technology has become essential for everyone. It appears that technology is developing quickly in the direction of raising people's standards of living as time goes on. Research on nanotechnology, which is a multidisciplinary field, has accelerated.¹⁻³ Nowadays, with the influence of developing nanotechnology, the search for new materials with unique properties has accelerated. Nanoparticles are very popular because they have very small sizes and a large surface/volume ratio compared to similar chemical structures, and they are more advantageous in terms of various biological, physical and chemical properties such as mechanical, electrical, optical, thermal conductivity and melting temperature. Nanoparticles have applications in many sectors (medicine, food, cosmetics, textile, agriculture, water treatment, etc.).⁴⁻⁵ As an example of applications in these sectors; in a study conducted by Li et al., a sensor developed for dopamine determination was prepared using water-soluble sulfonated graphene.⁶ Jemimah and Arulpani tested the antimicrobial properties of zinc oxide nanoparticles synthesized by *Lactobacillus plantarum* by coating them on diaper fabric.⁷ It was observed that the nanoparticles, which had an antimicrobial effect on some pathogens that cause skin and urinary tract infections, were most effective on *Klebsiella* sp. It was determined that the structure and antimicrobial activity of the nanoparticles did not change after they were coated on the diaper. As another sector, nanosilica is a nanomaterial widely used in concrete applications. In the study of Li et al., a new concrete with high performance was created using nanosilica and nanolime particles. As a result of the research, it was seen that nanosilica acts as an efficient filling material that reduces porous areas and improves the hydration process of cement.⁸ Nowadays, nanoparticle applications are also used in drinking water and wastewater treatment. Nanomaterials such as nanomembranes, carbon nanotubes, nanoclays and alumina fibers are used in water treatment applications.⁹⁻¹¹ In the food industry; nanomaterials are used in packaging technology to extend the shelf life and protect the quality and reliability of food until consumption. Development of nanosensors for the control of targeted gas and moisture concentration in the packaging, coating the packaging material with antimicrobial AgNPs to protect the microbial reliability of the product until consumption, and encapsulation of food ingredients to preserve sensory properties by preserving taste and odor components are approaches whose commercial potential is being investigated for the food industry.¹² The rapid development of the field of nanotechnology in wastewater treatment has focused the use of nanoparticles on the removal of dyes, as dyes pose a major environmental threat.¹³ The presence of dyestuffs in water bodies originates from many industries, such as food, pharmaceuticals, cosmetics, paint, plastic, paper and textile.¹⁴ These synthetic dyes have mutagenic or carcinogenic effects on human health and also affect aquatic flora and fauna.¹⁵ Dye wastewater can be resistant to physical, chemical and biological processes. In addition, it is very difficult to break down non-toxic products and remove them from water due to their aromatic structural stability. For this reason, reduction/degradation, removal and decolorization of cationic dyes are gaining scientific importance.¹⁴⁻¹⁷ In dye removal studies, AgNPs synthesized biologically from *Raphanus sativus*, *Morinda tinctoria*, *Imperata cylindrica*, *Trigonella foenum-graecum* ve *Cynodon dactylon* (L.) Pers were used in the removal of Methylene Blue (MB) dye and their removal efficiency was found to be 10 min. It was found to be between 75% and 100% in experiments carried out between 1 and 5 days.^{16,18-20}

However, it is an unavoidable fact that the production phases will need to be closely examined for environmental safety as the use of nanoparticles grows. As is well known, the synthesis of nanoparticles, which have a complicated structure, is expensive and may be hazardous to the environment, is typically accomplished by chemical and physical methods. To find a solution to this problem, scientists have developed the green synthesis method, which has been frequently used in recent years, and presented their remarkable results in reports.^{2,4} The green synthesis method involves the synthesis of nanoparticles using biological organisms such as bacteria, algae, fungi and plants. According to studies in the literature, scientists have succeeded in synthesizing many nanoparticles such as ZnO, Ag, Au, Ti, Zn, and CuO with the green synthesis method.⁴⁻⁵ The fact that AgNP has many properties (electrical, catalytic, sensor, antimicrobial, nanofertilizer, optical, etc.) has caused scientists to show intense interest in this particle.^{14,21} AgNPs have been used since ancient times. Among various biological methods, AgNPs synthesis from lignocellulosic biomass sources has been found to be an easy and accomplished choice due to the availability of plant sources and easy handling procedures.

It is estimated that approximately 200 billion tonnes of plant-derived waste is produced in the world, and 90% of it

is classified as lignocellulosic residue.²² Lignocellulosic biomass resources are generally; agricultural residues, specialty energy crops, municipal solid waste, forestry residues, food processing and other industrial wastes. Lignocellulosic biomass compositions vary depending on plant species, crops, origins, and management. Lignocelluloses generally contain 40-50% cellulose, 25-30% hemicellulose, 15-20% lignin and pectin, and small amounts of nitrogen compounds and inorganic compounds.²³⁻²⁴ The issue of utilizing these wastes, which have very low added value, and transforming them into new products has become very important today. Some of the lignocellulosic biomass is traditionally destroyed by direct burning in the fields or used in the feed industry. While this practice damages the natural structure of the soil, it can also cause air pollution. In this context, the necessity of processing lignocellulosic biomass and converting it into high value-added products and bringing them into the economies of countries emerges. With the use of lignocellulosic wastes, waste production is reduced and synthetic chemicals can be replaced by natural compounds. Thus, a more sustainable production is achieved. It reduces the carbon footprint of the entire cycle by reducing dependence on petrochemicals.

The most prevalent biopolymer in lignocellulosic wastes is cellulose. Cellulose is the main structural component in cell walls that gives mechanical strength and chemical stability to lignocellulosic biomass. Cellulose is a homopolysaccharide with a linear chain structure in the chemical structure of $(C_6H_{10}O_5)_n$ in the cell wall of lignocellulosic biomass.²⁵ Cellulosic polymers are found in the cell wall in the form of microfibrils, creating the fibrous structure that increases the mechanical strength of lignocellulosic biomass. Polymeric chains consisting of cellulose monomers are connected to each other by hydrogen bonds. In this way, they form a crystalline structure with cellulose chains parallel to each other. It basically consists of hemicellulose uronic acids, hexose (mannose, glucose, galactose) and pentose (xylose and arabinose) sugars.²⁴ Hemicelluloses are divided into three main subgroups. These are mannans, xylans and xyloglucans.²⁶ The third major component of lignocellulose biomass is lignin, which is generated by plants' secondary metabolism.²⁷ Lignin is a phenolic polymer and provides mechanical resistance and strength to the stems of plants. The basic units that make up the lignin molecule are connected to each other by ester bonds, but they also contain carbon-carbon bonds. Additionally, functional side groups such as hydroxyl and carbonyl can be seen in the structure. While the solubility of lignin polymers in alkaline environments is high, its solubility in aqueous media is low. As a result, lignocellulosic plant wastes are accepted as an alternative and important resource because they are renewable, environmentally friendly, abundant, and cheap.²⁸

Pollutant removal is a very important issue worldwide and researchers have reported many reviews on this topic.²⁹⁻³⁵ In this review, unlike other review reports, we present information on the preparation of AgNPs from lignocellulosic materials via a green method and the use of these nanoparticles (NPs) in cationic dye removal. Scientists and engineers in the fields of advanced materials and process control in water treatment may also find this review useful for the development of new materials.

Production processes based on green nanotechnology take place under green conditions without the intervention of toxic chemicals. Therefore, economic viability, environmental sustainability and social adaptation, as well as availability of local resources are of great concern in the production of nanomaterials. To keep nanotechnology prices affordable for consumers, industries must strike a delicate balance between environmentally responsible green processes and their sustainability. In light of all this, this study provides information about the synthesis of AgNPs using lignocellulosic biomass with the green synthesis method and the progress in the use of nanoparticles in the removal of cationic dyes from water.

2. Silver nanoparticles

It is the element whose Latin name is 'argentum' and is symbolized by Ag in group 1B in the periodic table. It is a transition element that is physically white in color, soft and has a metallic luster. It has two stable isotopes, Ag107 and Ag109. These isotopes are found in nature at rates of 51.84% and 48.61%, respectively.³⁶ AgNPs are structures containing 20-15,000 silver atoms. These nanoparticles can be in shapes such as ellipse, spherical, prism or rod. AgNPs have properties such as better conductivity, more catalytic activity and higher stability than nanoparticles synthesized from other metals.³⁷ Additionally, AgNPs are among the most synthesized nanoparticles. AgNPs appear in different colors depending on their size and shape. For example, the color of a 40 nm sized spherical AgNPs are blue, 100 nm

sized and spherical shaped yellow, and 100 nm sized prism shaped red color.³⁸ Silver is an element with high electrical and thermal conductivity. Due to its catalytic properties, it is also used as a catalyst in oxidation reactions. Ag^0 , Ag^{1+} , Ag^{2+} and Ag^{3+} are the four distinct oxidation states of silver in terms of chemistry.³⁹ Though it is a chemically inert element, it can react to generate soluble silver salts when combined with intense sulfuric acid or nitric acid.⁴⁰ Water does not dissolve metallic silver, but it does dissolve its metallic salts, such as silver chloride (AgCl) and silver nitrate (AgNO_3).⁴¹ It is also an effective antibacterial. Yeast, bacteria, fungi, and plants are examples of microorganisms that can be used in the biological synthesis of AgNP. The reduction and stabilization of silver ions by a mixture of biomolecules (such as proteins, amino acids, polysaccharides, terpenes, alkaloids, phenolics, saponins, and vitamins) already present in plant extracts is the easiest and least expensive way to produce AgNPs.⁴²

AgNPs find their place in many different application areas due to their unique properties and their use is becoming widespread in many areas such as food, agriculture, health, energy, industry, biotechnology, cosmetics, textile, environment, defense, electronics and space research.^{5,14-20} In particular, AgNPs obtained by green synthesis are highly preferred in medical applications and water treatment.⁴³ In order to examine AgNPs, it is first necessary to explain nanoparticles.

2.1 Nanoparticle

The word Nano derives from the Greek word ‘Nanos’, meaning ‘dwarf’. Nano is expressed as a unit per billion ($1 \text{ nm} = 10^{-9}$) nanometers in the measurement system. The subject of nanotechnology was first mentioned by Richard Feynman in his speech “There is Plenty of Room at the Bottom” at the annual meeting of the American Physical Society (APS) in 1959, and this speech became the inspiration for nanotechnology.⁴⁴ In 1974, the term “Nanotechnology” was created by Norio Taniguchi from Tokyo University of Science to describe the production of nanometer-sized materials.^{4,45} The aim of this technology is to create atomic or very small nano-sized structures with a size of 10^{-9} meters by physical, chemical, mechanical and thermal means. Thanks to nanotechnology, lighter, more durable, cleaner, higher strength, smarter and very cheap production is possible. Because of the special qualities linked to the size distribution and shape of NPs, nanotechnology has become more significant in recent years in a wide range of in vitro and in vivo applications.⁴⁶

NPs are the basic building blocks of nanotechnology. Materials with sizes ranging from 1 to 100 nanometers and different shapes such as triangle, circular, rod, spherical and star are called nanoparticles.⁴ They can be divided into different classes according to their properties, shape or size. Nanoparticles generally consist of carbon, metal, metal oxides or organic substances.⁴⁷ Different groups include metal NPs (pure metals (gold, platinum, silver, titanium, zinc, iron, etc.) or their compounds (oxides, hydroxides, sulfides, phosphates, chlorides, etc.)), ceramic NPs, and polymeric NPs. NPs are not simple molecules. They generally consist of three layers: (i) a surface layer that can be functionalized with various small molecules, metal ions, surfactants and polymers. (ii) The shell layer, which is a material chemically different from the core in every aspect. (iii) The core, which is the central part of the NPs, is often referred to as the NP itself.⁴⁸

Nanoparticles show unique physical, chemical and biological properties compared to similar particles at larger scales due to their high surface areas and nano sizes.⁹ These properties include differences in properties such as color change, thermal behavior, conductivity, material durability, resolution and optics. Its optical properties are reported to be size dependent, giving different colors due to absorption in the visible region. Decreasing from the macro dimension to the nano dimension often leads to an improvement in magnetic behavior. For example, there are magnetic materials such as various sensors and transformers. The two main applications that benefit from magnetic properties are medical applications and high-density information storage. Metal nanoparticles can be used as ferrofluids for biofuels.⁴⁹ Depending on the nanoparticle’s aspect ratio, chemistry, dispersion and interphase interactions with the polymer matrix, an increase in mechanical properties can be observed. Specific surface area is not only related to properties such as smoothness of nanoparticles, stability of surface area and support material interface, but also mainly relates to catalytic activity and other similar properties. The most suitable example for this situation is precious metal-containing catalysts with large surface area and superior catalytic activity. These unique properties are due to the greater surface area of nanoparticles relative to volume, their high reactivity and stability in chemical reactions.¹ Nanoparticle materials have many application advantages compared to other materials. For example, in the construction sector, nanomaterials are known to improve the strength and impermeability of concrete by strengthening the interfacial transition zone between

the impermeability of mortar and concrete.⁵⁰ Nanosilica is a nanomaterial with properties such as smaller particle sizes, high chemical purity, good dispersion, large surface energy, and stronger surface adhesion in concrete applications.⁵¹ Nanosilica also prevents the possibility of deterioration by reducing the porosity of concrete.⁵² Najjigivi et al. in a study conducted by them, it was observed that nanosilica reacted with lime during the cement hydration process and then increased the mechanical strength and durability of concrete.⁵³ Carbon nanotubes (CNTs) are another nanomaterial widely used for concrete applications. The tensile strength of CNT is almost 100 times higher than that of steel of similar diameter. It also has high thermal conductivity.⁵⁴ With the development of nanotechnology, it is seen that the use of nanoparticle systems in cosmetic products as another sector is increasing day by day.⁵⁵⁻⁵⁷ The characteristic features of nanoparticles such as stability, antimicrobial properties, easy production, preventing moisture loss, and showing less surfactant function stand out in cosmetic products. Examples of cosmetic products in which nanoparticles are used are cream, shampoo, shower gel, toothpaste, lipstick, nail polish, and blush.⁵⁶ Especially the production of silver NPs with green synthesis has shown that their easy production with significant biocompatibility and strong antibacterial effect can be used in biomedical fields.^{5,58-59}

Nanometer-sized materials are used in various fields because they have these superior properties. Some of them are given below:

- * Environment and Energy.
- * Medicine and Health.
- * Nano Electricity and Computer.
- * Materials and Manufacturing Industry.
- * Defense Sector.
- * Aerospace Research.
- * Biotechnology.
- * Agriculture and Food.

2.2 Synthesis of nanoparticles

The desired size, shape, crystal structure, and chemical composition of these materials must be made available in order to explore new physical attributes and practical uses of nanostructured materials.³ Various methods can be used for NPs production, but these methods are generally divided into two basic classes:⁴⁷

- I. Top-Down Approach.
- II. Bottom-Up Approach.

Using physical (such as mechanical) or chemical methods, the size of a suitable starting material is decreased in the top-down approach. A structure is built using the bottom-up method atom by atom, molecule by molecule, or in clusters (Figure 1).

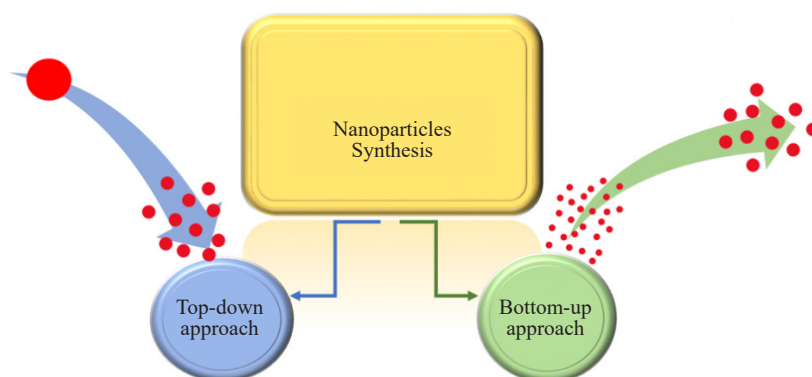


Figure 1. Protocols for nanoparticle synthesis: bottom-up approach and top-down approach

The bottom-up approach is more preferred in nanoparticle synthesis.⁵⁹ Defects may occur in the surface structure of nanoparticles obtained with the top-down approach, which may create disadvantages for the physical properties and surface chemistry of the nanoparticles. Top-down synthesis methods, physical synthesis methods and chemical synthesis methods have harmful effects on the environment because they contain factors such as different toxic chemicals, high temperatures and pressures.⁶⁰ Top-down methods are not suitable for the preparation of very small particles. Biological techniques used in the bottom-up synthesis method have many advantages, such as the use of biological materials such as various plant extracts, algae, and enzymes, the avoidance of toxic chemicals and catalyst materials, more suitable physical environment conditions and easier synthesis.⁶¹ The bottom-up approach has the advantage that it is more likely to obtain nanoparticles with fewer defects and more homogeneous chemical compositions.⁴³ These approaches are divided into subclasses such as biological, physical, and chemical synthesis according to conditions of the reaction and the processes to be applied (Figure 2).

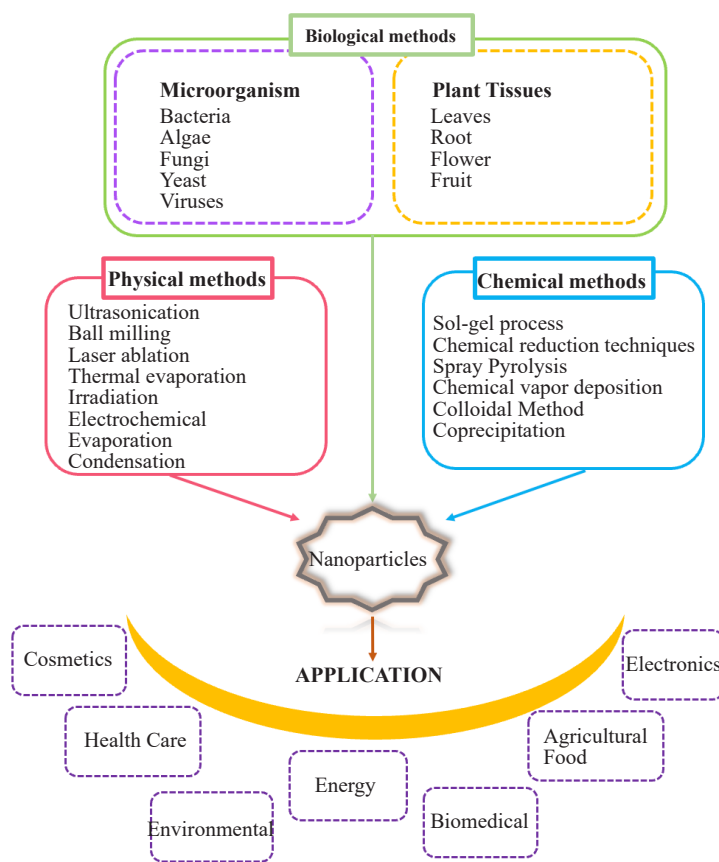


Figure 2. Synthesis methods and applications of nanoparticles

By heating the main bulk material with an electron beam, the physical vapor deposition (PVD) method of synthesizing nanostructures in the gas phase is achieved.⁶² The foundation of the laser pyrolysis process is the quick heating and cooling of small-sized nanoparticles in an inert gas environment while utilizing a strong laser beam.⁶³ High-purity nanostructural thin films with excellent performance at high temperatures can be created using the chemical vapor deposition (CVD) method,⁶²⁻⁶⁴ whereas nanomaterials with magnetic or optical properties can be created using the sol-gel approach.⁶⁵ Chemical reduction is a type of oxidation-reduction reaction used in synthesis; both organic and inorganic reducing agents are used in the reaction.⁶⁶ The foundation of the microemulsion approach is the homogenous and size-controlled production of metal nanoparticles by the utilization of oil-water and water-oil inorganic phases.⁶⁷ The capacity to quickly synthesize large quantities of homogenous nanoparticles and control particle size and morphology

are two of these technologies' greatest benefits.⁶⁸ Nowadays, while it is possible to synthesize nanoparticles with the desired morphology and unique properties by chemical and physical methods,⁶⁹ these methods also have significant disadvantages such as being expensive due to the need for equipment, the necessity of difficult reaction conditions and the use of various chemicals that harm the environment, and having high toxicity and causing negative and fatal effects on the environment and microorganisms.⁷⁰⁻⁷¹ In addition, the majority of chemical processes include the use of hazardous chemicals and frequently result in non-polar organic solutions and unfavorable environmental byproducts. In contrast, biological synthesis provides a low-cost, creative, dependable, and long-lasting substitute for physical and chemical approaches in NP synthesis.^{1,72} The synthesis method has many advantages compared to traditional synthesis methods including physical and chemical methods. For example, it is non-toxic since it does not use toxic chemicals compared to nanoparticles obtained by chemical synthesis. In addition, this method is simple, environmentally friendly, cost-effective and biocompatible. In addition, there is no need to add any external stabilizer. Because the phytochemical components of plants or microorganisms used in nanoparticle synthesis with the green synthesis method act as stabilizers as well as stabilizers. Another advantage of this method is that the experimental steps in obtaining nanoparticles through chemical synthesis can be eliminated and nanoparticle synthesis can be performed in a single step.⁷³ In other words, nanoparticles produced by green synthesis are generally a cheap, fast and easy method with low toxicity, completely nature and ecosystem friendly, and can be easily produced without the need for various operating parameters such as temperature, high pressure and energy.^{74,75}

3. Green synthesis

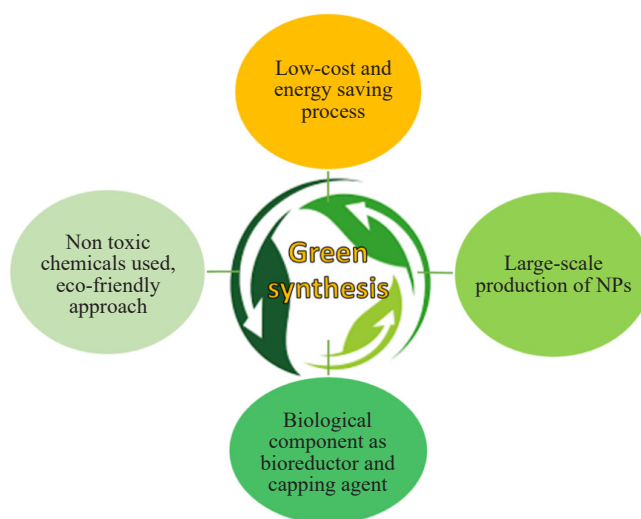


Figure 3. Key merits of green synthesis method

Nanoparticles synthesized using physical or chemical techniques that have been used for a long time are expensive and have high toxicity due to the need for chemicals (as reducing agents; sodium borohydride, hydrazine, coating agents; organic solvents, chloroform, and toluene) and equipment, and they have negative and fatal effects on the environment and microorganisms.⁷¹ Additionally, physical methods (e.g., the use of a tube furnace) often require large amounts of space and are very time consuming. For such reasons, a need for an alternative in nanoparticle production was felt, which gave rise to the concept of green nanotechnology (or green nanobiotechnology). Using a variety of biotechnological instruments, green synthesis is the process of creating nanoparticles from biological resources that include microorganisms, plants, viruses, or their byproducts like proteins and lipids.

Green synthesis produces low-toxicity, inexpensive, quick, and simple nanoparticles that are safe, biocompatible,

and friendly to the environment. It also eliminates the need for several operating parameters, including high pressure, temperature, and energy.⁴ (Figure 3). Because they are created in a single process, nanoparticles made with biological methods are more stable and have the appropriate dimensions. Plant parts, particularly leaves, fruits, roots, stems, and seeds, are frequently employed in green synthesis to create various NPs. Green synthesis; It has several principles, such as avoiding or minimizing waste, reducing pollution, and using renewable raw materials as well as safer (or non-toxic) solvents and auxiliaries.⁷⁶ There are 12 basic principles of green chemistry.⁷⁷

- (1) Waste prevention.
- (2) Maximum atomic economy.
- (3) Less hazardous chemical synthesis.
- (4) Safer solvents and auxiliaries.
- (5) Designing safer chemicals.
- (6) Use energy-efficient design.
- (7) Utilizing sustainable feedstocks.
- (8) Reduce derivatives.
- (9) Catalyst.
- (10) Design for degradation.
- (11) Analysis of pollution prevention in real time.
- (12) Chemistry that is inherently safer to prevent accidents.

With all these, social, economic and environmental sustainability is achieved by reducing waste, materials, risks, environmental impacts and prices.

The synthesis and stabilization of nanoparticles made using biological entities are influenced by a number of variables, including temperature, pH of the surrounding environment, and reaction time. The production of nanoparticles is significantly influenced by the pH of the reaction medium. The size and structure of the nanoparticles vary with different hydrogen ion concentrations. Temperature has a significant impact on the structure and form of nanoparticles during their creation. When *Cymbopogon flexuosus* leaf extract was used to create gold nanoparticles, it was shown that at lower reaction temperatures, nanotriangles formed, whereas at higher reaction temperatures, more spherical nanoparticles were produced with nanotriangles.⁷⁸ It is widely believed that nanoparticles produced through biological synthesis are far superior to those produced through other synthetic methods. The quality and form of nanoparticles are significantly influenced by the reaction time and environment.⁷⁹ For example, as a plant frequently used in biological synthesis, it is generally very easy to obtain and available in large quantities, so a large amount of nanoparticles can be biosynthesized. At the same time, nanoparticles obtained by synthesizing plants have been reported to be both very fast and stable and more economical.⁸⁰ Today, nanoparticle material synthesis studies are being carried out successfully using different parts of plants. There are different studies in the literature in this context. Rolim et al. observed the antibacterial and cytotoxic effects of silver NPs obtained from green tea in a study they conducted.⁸¹ In this study, silver NPs showed a strong antibacterial effect against various pathogenic gram-positive and gram-negative bacteria. In another study, Jemilugba et al. synthesized water-soluble silver NPs from *C. erythrophyllum* leaf extract via green synthesis.⁸² According to TEM images, silver NPs have a particle size of 13.62 nm and are spherical in shape. These synthesized silver NPs were noted to exhibit antibacterial activity against gram positive *S. aureus*, *S. epidermidis* and gram negative *E. coli* and *P. vulgaris*. In another study, researchers synthesized AuNPs using the extract of *Acacia nilotica* twig bark and used the nanoparticles for nitrobenzene detection.⁸³ Ghoreishi et al. synthesized Ag and AuNPs using Rosa damascena flowers. They also modified a glassy carbon electrode with these nanoparticles and used them in electrochemical applications.⁸⁴ There are many studies in the literature using different bacteria for the production of nanoparticles by green synthesis. For example, Hassan and Mahmood used *Escherichia coli* in the synthesis of iron oxide nanoparticles in a study.⁸⁵ In addition, AgNPs of different sizes and structures were successfully synthesized using different bacteria such as *Corynebacterium* sp. SH09, *Corynebacterium glutamicum*, *Escherichia coli*, *Bacillus cereus*, *Morganella* sp., and *Bacillus licheniformis*.⁸⁶ Algae are another preferred biological material for the synthesis of nanoparticles by green synthesis method. For example, AgNPs were obtained with various algae such as *Chlamydomonas reinhardtii*, *Chaetomorpha linum*, *Cystophora moniliformis*, *Leptolyngbya valderianum*, *Oscillatoria willei*, *Sargassum muticum*, while AuNPs were successfully synthesized with Brown & *Sargassum muticum*, *Chlorella vulgaris*, *Chondrus crispus*, *Lyngbya majuscula*, *Padina pavonica*, *Phormidium tenue algae*.⁸⁷ Fungi are widely used in the synthesis studies of

nanoparticles. For example, Zhang et al. used the fungus *Mariannaea* sp. HJ in the synthesis of selenium nanoparticles.⁸⁸

3.1 Green synthesis of metal nanoparticles

Metallic nanoparticles can be produced by natural, synthetic, or biological processes.^{60,89-90} Physical techniques like laser ablation,⁹¹ inert gas condensation,⁹² electric arc discharge,⁹³ and radio frequency (RF) plasma approach⁹³ are frequently employed to synthesize nanoparticles. These physical techniques take a long time to establish thermal stability, use a lot of energy to raise the temperature surrounding the source material, and take up a lot of space in the case of tube furnaces. Consequently, it is not appropriate to produce nanoparticles using the physical synthesis method.⁹⁴ The use of high radiation, reductants with high toxicity levels and agents required for stabilization in physical and chemical syntheses causes negativities for both the environment and living things.⁷⁶



Figure 4. Benefits of green synthesis method of metal nanoparticles

Green synthesis offers several advantages over physical and chemical approaches for the generation of metal nanoparticles. It is cost-effective and environmentally safe, and it doesn't require high pressure, energy, temperature, or the use of dangerous chemical reagents, for example (Figure 4). This approach uses the leaves, roots, flowers, and fruits of the plant as plants, and numerous reductants, including bacteria, plants, algae, yeast, fungi, microalgae, and diatoms, to synthesize metal nanoparticles.

Bacterial cells are considered as a possible biofactory for producing metal nanoparticles by green synthesis. Stressful environments are typically present for bacterial cells all the time. They can endure in competitive settings as a result of these circumstances. As a result, they are somewhat resistant to metallic salt concentrations that are high. The synthesis of nanoparticles by microorganisms can be carried out by intracellular and extracellular methods.⁹⁵ Extracellular synthesis is preferred over these methods. Because the separation process of nanoparticles, which includes cell disruption by mechanical means and removal of cell components by centrifugation, is eliminated with this method.⁹⁶ For example, Patil et al. obtained spherical AuNPs with the *Paracoccus haeundaensis* BC74171T bacterium by extracellular synthesis method.⁹⁶ Extracellular reductase enzymes of bacteria carry out the biological reduction of silver ions to AgNPs. When silver ions are reduced to AgNPs, the enzyme is also oxidized. It has been reported that the rapid extracellular formation of nanoparticles occurs within a few minutes.⁹⁷ Copper nanoparticles, known to have antimicrobial properties, were first produced extracellularly by a copper-resistant strain, *Bacillus cereus*.⁹⁸ Previous studies have determined that copper exhibits better antimicrobial activity in nanoparticle form than in its elemental form.⁹⁹ Three processes take place in the intracellular formation of metal nanoparticles in microorganisms: capture, biological reduction and capping. Cell walls and ionic charges of microorganisms significantly affect the formation of

NPs. Cell enzymes, coenzymes and other molecules of microorganisms provide the passage of certain ions. Not every bacterium may be suitable for nanoparticles of all metals or metal oxides. Metal nanoparticle synthesis using bacteria has been done in many studies. Silver's ability to kill biological organisms is widely recognized. But, silver resistance exists in certain bacteria.¹⁰⁰ *Pseudomonas stutzeri* strain AG259, which was obtained from a silver mine, provided the first indication that bacteria were producing AgNPs.¹⁰¹ *Pseudomonas stutzeri* bacteria that have been isolated using silver precursors have also been reported to have a size range of 16-40 nm and the ability to reduce Ag⁺ ions to create AgNP.¹⁰²⁻¹⁰³

Fungi are an ideal biological system for the synthesis of metal nanoparticles due to their resistance to toxicity, ease of scaling, large surface area, easy and economical applicability, and intracellular metal retention capabilities compared to bacterial systems.⁸⁶ Its ability to bind and absorb intracellular material is comparable to that of bacteria.¹⁰⁴ Using mushrooms increases the synthesis of metal nanoparticles. Because mushrooms are easier to produce and make, and they grow more quickly.¹⁰⁵ Moreover, some fungal species grow rapidly and therefore it is very easy to culture and maintain them in laboratories.¹⁰⁶ Due to all these advantages, fungi are widely used in the synthesis studies of metal nanoparticles. For example, AuNPs have been synthesized with different fungi such as *Rhizopus oryzae*, *Neurospora crassa*, *Fusarium semitectum*, *Fusarium solani*, *Aspergillus foetidus*, *Trichoderma harzianum*, *Phanerochaete chrysosporium*, *Trichoderma viride hypocrealexii*,¹⁰⁷ and AgNPs have been synthesized with different fungi such as *Verticillium* sp., *Aspergillus fumigatus*, *Trichoderma asperellum*, *Phanerochaete chrysosporium*.¹⁰⁸

On the other hand, algae are another preferred biological material for the production of nanoparticles. Algae contain various components such as carbohydrates, proteins, minerals, fats, antioxidants, carotenoids and chlorophylls. These components act as reducing and stabilizing agents in the synthesis of nanoparticles.¹⁰⁹ Microalgae and cyanobacteria have the ability to store and detoxify metal ions in intracellular metal-binding peptides such as phytochelatins and metallothioneins and in polyphosphate structures. The majority of algae can tolerate heavy metals with methods such as enzymatic detoxification, synthesis of metal-binding proteins, and precipitation of metals by forming insoluble complexes. Thanks to their detoxification mechanisms, algae give successful results in the reduction of metal ions to metal nanoparticles. In publications investigating the reducing and stabilizing potentials of algal metabolites, different approaches are tried for the formation of nanoparticles. There are basically two approaches: intracellular and extracellular nanoparticle synthesis. Intracellular production: -Using harvested, wet or dry biomass, taking metal ions into the whole cell, reducing them through intracellular metabolites and forming metal nanoparticles. -Adding metal ions to the culture where growth continues, ensuring storage of ions within the cell and simultaneous formation of nanoparticles with the culture. Extracellular production: -Reducing metal ions to metal nanoparticles through extracellular metabolites contained in the supernatant. -Reducing metal ions to metal nanoparticles through metabolites contained in the crude extract by performing post-harvest extraction. -Synthesis of metal nanoparticles through the reducing properties of purified biomolecules from the crude extract. Algae have been used to create nanoparticles, according to numerous research. According to reports, the blue-green alga *Spirulina platensis* is utilized in protein-mediated gold nanoparticle synthesis, producing uniformly sized particles with an average size of about 5 nm.⁵⁸

Compared to microorganisms, most plants have more regenerative and sustainable qualities.²¹ Plant extracts contain metabolites such as ketones, aldehydes, flavones, amides, terpenoids, carboxylic acids, phenols, and ascorbic acids that are employed in the creation of metal/metal oxide nanoparticles. Metal salts can be reduced to metal nanoparticles by these ingredients.⁷⁶

Biobased green synthesis techniques rely on a range of reaction parameters, including pH, temperature, pressure, solvent, processing time, agitating or static application, reducing agent and substrate amounts. As a result of studies conducted in this field, metal NPs in different shapes, sizes and morphologies can be obtained depending on the biological agent and conditions. The production of metal nanoparticles is significantly influenced by the pH of the surrounding environment.⁸⁹ The production of nucleation sites rises in tandem with pH. With the nucleation center, metallic ions are reduced to metal nanoparticles. The pH of the solution environment affects both the activity of the functional groups in the plant extract and the rate at which metal salts degrade. For example, in a study by Dwivedi and Gopal, they examined the effect of pH (2-10) on the synthesis of silver and gold nanoparticles in *Chenopodium album* plant leaves. They reported that in both nanoparticle synthesis, larger sized NPs were synthesized at pH 2 than after pH 4, and more stable NPs with similar shape and size were synthesized between gold nanoparticles and pH 4-10.¹¹⁰ Sherin et al. examined the effect of pH (5-9) on the AgNPs they synthesized from *Terminalia bellerica* extract. They reported that

large-sized nanoparticles were formed at acidic and neutral pH, while smaller-sized nanoparticles were formed at pH 9.¹¹¹ Large rod-shaped Au nanoparticles (25-85 nm) were formed from *Avena sativa* at pH 2, but smaller nanoparticles (5-20 nm) developed at pH 3 and 4.¹¹² Similarly, utilizing bark extract from *Cinnamomum zeylanicum*, more spherical AgNPs were produced at higher pH values (pH 5 and above).¹¹³

The production of nanoparticles mediated by plants is significantly influenced by the temperature of the reaction medium. Similar to pH, temperature causes an increase in nucleation center development, which speeds up biosynthesis. However, at lower temperatures, the shape of nanoparticles is more likely to be triangular and spherical, while at higher temperatures, they are more likely to take the shape of nanorods and platelets.⁴³ Sun et al. examined the effect of temperature parameter on the synthesis efficiency of AgNPs using tea leaf extract. NPs with sizes of 91, 129 and 175 nm were obtained at various temperature ranges such as 25 °C, 40 °C and 55 °C, respectively. It was concluded that better NP synthesis occurred at 25 °C, but the increase in temperature did not have a significant effect on silver NP synthesis.¹¹⁴

As another factor, plant extract concentration is reported to affect the morphology of NPs. The increase in the concentration of the plant extract not only increases the nanoparticle synthesis rate, but also causes a change in the shape of the nanoparticles. Therefore, it is necessary to determine the optimum concentration in the synthesis process.⁴³ Huang et al. reported that the concentration of *Cinnamomum camphora* leaf extract affected the morphology of synthesized gold and silver NPs. It has been reported that the shape of AuNPs changes from nanotriangle to spherical shape when the amount of extract is changed to 0.1-0.5-1 g.¹¹⁵ Sherin et al. examined the effect of AgNO₃ concentration (1-2.5 mM) on the AgNPs they synthesized from *Terminalia bellerica* extract. According to their results, they observed that the optimum AgNO₃ concentration of 2 mM was suitable, and that higher concentrations than this concentration caused aggregation, resulting in a decrease and broadening of the SPR signal. It has been reported that the nanoparticles obtained under optimum conditions are spherical in shape and have an average size of 29.6 nm.¹¹¹

In the synthesis of metal nanoparticles, reaction time greatly affects the quality, aggregation risk, and morphology of the nanoparticle.⁷⁹ Changes in mixing time, exposure to light, the synthesis method used and storage conditions affect the properties of nanoparticles.¹¹⁶⁻¹¹⁷ Prolonged mixing may cause aggregation or shrinkage of nanoparticles.¹¹⁸ Badoei-dalfard et al. investigated the effect of time on the synthesis of AgNPs using the biological synthesis method. It has been reported that biosynthesis begins after 1 hour and reaches its maximum after 24 hours. It has been reported that no significant AgNP synthesis was detected after 48 hours, therefore NP synthesis after 24 hours is the best time.¹¹⁹ In a study of Li et al., in which Ag nanoparticles were synthesized using the extract of the *Capsicum annuum* (Red Pepper) plant, it was determined that the shape of the nanoparticles was spherical and polycrystalline and the size was around 10 ± 2 nm, when the reaction time was planned as 5 hours under the same conditions. When the reaction time was increased to 9 and 13 hours, it was observed that the sizes of the nanoparticles increased to 25 ± 3 nm and 40 ± 5 nm, respectively.¹²⁰ According to Nazeruddin et al, in a study conducted by Ag nanoparticles, while Ag nanoparticles could be synthesized in 2-4 days using microorganisms, they managed to synthesize the same nanoparticle in 1-2 hours using the *Coriandrum sativum* plant.¹²¹ In the green synthesis method, synthesis with microorganisms is not preferred due to the requirements of high levels of aseptic conditions and their maintenance. Plants are the most preferred sources for NP synthesis because they facilitate large-scale synthesis and synthesis of NPs variable in shape and size.^{122-125,90}

3.2 Green synthesis with lignocellulosic biomass

While it has been known since the early 1900s that plant extracts can reduce metal ions, the composition of the reducing agents is still unclear. Nonetheless, while being extremely straightforward, the production of nanoparticles using plants and plant extracts has garnered a lot of interest lately.^{4,21,89} The reason for this is that plant extracts; are very cheap, allow large amounts of production, do not require special storage conditions, have no risk of contamination, and are very stable against harsh conditions (such as high temperatures, a wide pH range and salt concentrations).¹²⁶ On the other hand, the time to synthesize nanoparticles with plants tends to be faster than the synthesis using fungi and bacteria in green synthesis, is more economical, and is relatively easier to scale up for the production of large quantities of nanoparticles.^{76,127} Additionally, the waste products resulting from the microorganism-based method may be harmful to the environment, depending on the type of microorganisms involved in the synthesis.¹²⁸ There is no need for any unique, difficult, multi-step processes like isolation, culture preparation, and culture maintenance when utilizing microorganisms. It is accepted that synthesized nanoparticles of plant origin are less likely to cause harmful effects in humans compared to chemically synthesized nanoparticles and show advanced biological potential with applications in

food technology, bioengineering, cosmetics, nanomedicine and humans.¹²⁹

The synthesis of metal nanoparticles with plants can be achieved by using different parts of the plant such as leaves, flowers, roots, fruits and seeds. The presence of a wide variety of phytochemicals in plant extracts provides nanoparticle formation by showing natural stabilizing and reducing properties. The process steps of this method are generally prepared as follows:

- I. Collection of plants,
- II. To remove dirt and impurities, washing with pure water,
- III. Drying washed plant parts,
- IV. Size reduction,
- V. Heating in a solvent,
- VI. Filtering the extract by filtration or centrifugation,
- VII. Mixing the resulting extract with metal salt solution,
- VIII. Reduction of metal salt and formation of metal nanoparticles.¹³⁰

In this method, there is no need to add any reducing and/or stabilizing agents to the synthesis medium, because the phytochemical agents found in the plant fulfill both functions.⁷³ The synthesis of many metal nanoparticles such as silver, gold, iron, copper and zinc can generally be carried out using this process.^{5,89,131} The synthesis of nanoparticles, which have therapeutic value and are environmentally safe, is made possible by biomolecules like proteins, amino acids, enzymes, polysaccharides, alkaloids, tannins, phenolics, saponins, terpenoids, and vitamins that are naturally present in plant extracts. The process involves reducing metal ions or metal oxides to 0-valent metal NPs, which are then measured by periodically observing the UV-visible (UV-Vis) spectra of the solution.^{72,132-133} UV-Vis spectrometry is one of the main techniques used in the characterization of metal nanoparticles. UV-Vis spectrometry is a fast and easy technique. It is also selective against different nanoparticles. For all these reasons, it is frequently used in the characterization of nanoparticles. In the UV-Vis spectrometry technique, a beam is sent to the sample solution and the intensity of the beam passing through the sample is measured. Metal nanoparticles have advanced optical properties that are very sensitive to size, shape, agglomeration and concentration changes, and this feature is due to the surface plasmon resonance (SPR) of the nanoparticles.¹³⁴ When free electrons near the surface of nanoparticles are excited by electromagnetic waves, they oscillate collectively and a localized electromagnetic field is formed on the nanoparticle surface. The phenomenon of oscillation of metal electrons in harmony with the electromagnetic field is called SPR.¹³⁵ Upon contact of metal salt with plant extract, the suspension color changes and the color change is due to the excitation of surface plasmon vibrations in metal nanoparticles. Analyzing the suspension using UV-Vis spectrophotometer method usually reveals a band where the absorption peak can be determined and the metal of interest can be confirmed. A gradual increase in the characteristic peak with the increase in the reaction time and concentration of the plant extract with metallic ions is a clear indication of nanoparticle formation.⁴³ As the particles become unstable, a decrease in the observed peak intensity values due to the depletion of stable NPs, a broadening at the peak point or a second peak at longer wavelengths due to the change in the size of the particles due to aggregation can be observed.¹³⁶ The SPR band is affected by the size, morphology, shape, composition of the nanoparticle and the dielectric constant of the medium.¹³⁷ For example, in the measurements taken for AgNPs, plasmon resonance peaks are observed in the range of 420-500 nm, while AuNPs are observed between 500-550 nm.¹³⁸ Particle size and dielectric constant of the medium affect the position (wavelength range) and shape of the plasmon absorption peak in studies with AgNPs.¹³⁹ The blue shift of the peak indicates the reduction of the particle size of the nanoparticle, while the increase in the absorbance value is associated with the increase in nanoparticle formation.¹⁴⁰

There are approximately over 200,000 chemicals in the universe that have been isolated from plants and identified with various structures and classes. There are two categories for these substances: primary and secondary metabolites. Nucleic acids, proteins, carbohydrates, and fatty acids are examples of primary metabolites that are involved in cell maintenance. Although secondary metabolites do not directly participate in photosynthetic or respiratory metabolism, they are known to be necessary for the survival of plants.¹⁴¹ Phenolic compounds are one of the more common secondary metabolites found in plants.¹⁴¹ They are the most common secondary plant metabolites with more than 8,000 known structures. The largest group among phenolic compounds is flavonoids. Structurally, flavonoids consist of a flavan nucleus with 15 carbon atoms arranged in 3 rings, C6-C3-C6, named A, B and C. One of the more prevalent types of secondary metabolites in plants is phenolic compounds.¹⁴¹ They have more than 8,000 identified structures and are the

most prevalent secondary plant metabolites. Flavonoids are the most abundant class of phenolic chemicals. In terms of structure, flavonoids are made up of a flavone nucleus that has 15 carbon atoms organized in three rings, A, B, and C, for example, C6-C3-C6. The predominant polyphenols in human nutrition are flavonoids. Flavones, flavanones, flavonols, flavanols, anthocyanins, and isoflavones are the six subclasses of flavonoids.¹⁴² Due to their bioactive qualities, these substances have been linked to a number of health advantages in humans, including antiviral, cardioprotective, neuroprotective, anticancer, immunomodulatory, antidiabetic, and antibacterial and antiparasitic effects. Since phenolic chemicals are all aromatic, they all exhibit strong UV absorption. All phenols with carboxylic acid functionality are referred to as phenolic acids. The hydroxycinnamic and hydroxybenzoic carbon frameworks are the two distinct forms found in phenolic acids. Due to their significant biological and pharmacological qualities, particularly their anti-inflammatory, antioxidant, and antimutagenic effects, phenolic acids are becoming more and more popular. People consume phenolic acids in their daily diet because they are widely found in plant-based foods.¹⁴³ These phytochemicals, which are non-toxic and have hydroxyl and carboxyl groups that can bind to metals, have unique chemical power to degrade and also effectively encapsulate nanoparticles, thus preventing the agglomeration of nanoparticles.¹³²

One of the first studies on using plants for the synthesis of metallic nanoparticles was the synthesis of AgNPs using alfalfa sprouts. It has been reported that alfalfa roots have the ability to absorb Ag from the agar medium and deliver Ag to the shoots of the plant in the same oxidation state, enabling their association and formation of nanoparticles.¹⁴⁴

For metal nanoparticle synthesis using plants, the method of transporting the ionic form of the relevant metal throughout the plant, translocating it in the plant and reducing the salt form to the element is generally accepted. In this method, in order for metallic NPs to be synthesized in plants, it is important that this metal be soluble, transportable and translocated.

In general, metal nanoparticle synthesis from plants consists of three main steps:

- I. The activation phase, during which metal ions are reduced and reduced metal atoms are nucleated.
- II. The growth phase, during which smaller nanoparticles clump together to form larger particles with increased thermodynamic stability.
- III. The finalization stage, during which the nanoparticles choose the energy-efficient structure that best suits them and decides on their ultimate form. This mechanism results from a plant extract's capacity to stabilize metal nanoparticles.¹³¹

The first stage of the basic mechanism involves reducing the metal ions (M^+ or M^{2+}) in a plant extract sample to metal atoms (M^0) by mixing the sample with a metal salt solution. Reduced metal atoms can nucleate.¹⁴⁵ Subsequently, these small NPs combine with smaller neighboring particles to form larger nanoparticles, which simultaneously increases thermodynamic stability. After this stage, there is a growth period during which further bioreduction of metal ions occurs. The NPs that are generated as growth proceeds combine to form a variety of morphologies, including cubes, spheres, triangles, hexagons, pentagons, rods, and wires.¹³¹ The extract's most stable and energetically advantageous form during the last stage of production is determined by its capacity to stabilize metal nanoparticles.

Biological entities within the plant have limiting and stabilizing agents necessary to act as growth terminators and to inhibit the agglomeration process. Their concentrations, together with those of living things and natural reducing agents, have an impact on the nanoparticles' size and form. Enzymes, proteins, carbohydrates, and phytochemicals such as terpenoids, flavonoids, and phenolics, on the other hand, primarily function as stabilizing and reducing agents.¹

As the first confirmation in metal NP synthesis, it can be checked by looking at the color change in the solution.

Green synthesis using plants has many advantages and the most important advantages are:

- Easy usability,
- Safe use,
- Affordable cost,
- Simple one-step process,
- Plants contain various metabolites that provide reduction,
- Elimination of detailed maintenance of cell cultures,
- Fast synthesis,
- Environmentally friendly,
- Stable nanoparticles,
- Easy control over the size and shape of nanoparticles,

- Suitability for large-scale syntheses.⁴³

Due to these advantages, the synthesis of metal nanoparticles using plants and their studies in different application areas are widely carried out in the literature. For example, in a study by Valsalam et al., they synthesized AgNPs using nasturtium extracts and examined the antibacterial, antifungal, antioxidant and anticancer properties of the synthesized AgNPs.¹⁴⁶ Sharmila et al. synthesized 70-75 nm sized spherical ZnO nanoparticles using the leaves of the yellow trumpet (*Tecoma castanifolia*) plant.¹⁴⁷ In a study by Ghoreishi et al., they synthesized Ag and AuNPs using *Rosa damascena* flowers. They also modified a glassy carbon electrode with these nanoparticles and used it in electrochemical applications.⁸⁴ Thakore et al. synthesized Ag and Cu nanoparticles using the latex of Sapota fruit (*Achras sapota* Linn.).¹⁴⁸ In one study, researchers synthesized AuNPs using the extract of *Acacia nilotica* twig barks and used the nanoparticles for nitrobenzene detection.⁸³ In a study by Dawodu et al., they obtained 25 nm sized AgNPs using the stem part of the *Vigna unguiculata* L. plant. They also used it as an adsorbent in malachite green adsorption studies.¹⁴⁹ In another study, Yu et al. synthesized AuNPs using the extract of *Citrus maxima* fruits and investigated the catalytic properties of the nanoparticles in the reduction of 4-nitrophenol to 4-aminophenol.¹⁵⁰ In another study, Filip et al. successfully synthesized spherical 19 nm sized AuNPs and 16 nm sized AgNPs using cranberry fruit.¹⁵¹ Kumar et al. obtained AuNPs using *Croton caudatus* Geisel leaf extracts and investigated the biological properties of the nanoparticles such as antibacterial/antifungal.¹⁵² Jayaseelan et al. synthesized AuNPs using *Abelmoschus esculentus* seeds and determined their antifungal properties.¹⁵³ In a study conducted by Ebrahimzadeh et al., they carried out synthesis studies of AgNPs using *Crataegus pentagyna* fruits and obtained AgNPs with a spherical structure and sizes of 25-45 nm.¹⁵⁴ Leonard et al. synthesized AuNPs using red ginseng root (*Panax ginseng* C.A. Meyer) and investigated the cytotoxic properties of nanoparticles.¹⁵⁵

As can be seen from the examples above, many studies have been carried out in the literature using different plants and different parts of these plants to obtain metal nanoparticles, and today the number of these studies is increasing. Because the use of plant extracts rather than other biomaterials for the biosynthesis of nanoparticles has been accepted as a more reliable and environmentally friendly method.

3.3 Biosynthesis of AgNPs using lignocellulosic biomass

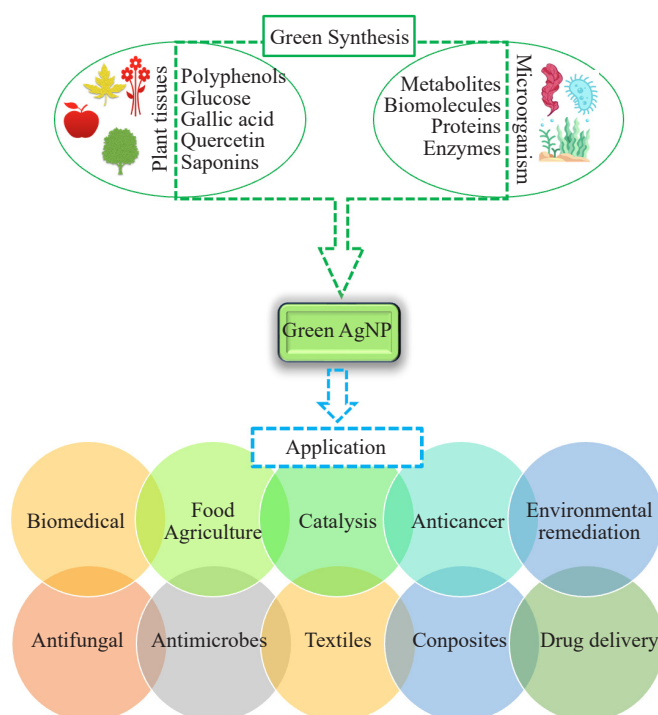


Figure 5. Synthesis of AgNPs through green synthesis and their various applications

Biological sources such as plants, fungi, algae, and bacteria can be used to obtain AgNPs through biosynthesis (Figure 5). The fact that AgNPs obtained from leaves, fruits, roots, flowers, or the entire plant are obtained in larger quantities, are stable, and the synthesis is simple and cost-effective increases interest in synthesis studies using plant sources. Bioactive components such as alkaloids, terpenoids, flavonoids, enzymes, amino acids, phenolics, etc., which constitute the structure of phytochemicals, reduce Ag^+ ions in the aqueous structure and form Ag^0 , forming AgNPs.¹⁵⁶

Obtaining AgNP from lignocellulosic biomass using the green synthesis method is generally as follows:

- I. To get rid of dirt, rocks, and debris that have stuck to the surface, plants are cleaned with clean water.
- II. Plants are let to dry either in the oven or at room temperature.
- III. Dried plant materials are reduced in size to facilitate extraction.
- IV. These prepared plant materials are then extracted in aqueous form by boiling using water or without applying heat.
- V. The plant extracts are then filtered using filter paper.
- VI. The filtered aqueous solutions are then kept for later use at about 4 °C in the refrigerator.
- VII. On the other hand, silver salts are prepared at determined concentrations using the precursor.
- VIII. Then, plant extracts prepared on silver precursor are added in different amounts for nanosilver synthesis.
- IX. Biochemical reduction of silver salts begins immediately.



Stirring continuously homogenizes the prepared solutions. The solutions start to turn from clear to a transparent yellow-brown tint. This hue shift in the nanoparticles generated suggests that metallic silver was successfully synthesized or formed. These hue shifts typically happen even at room temperature, which is 25 °C. Figure 6 illustrates a generic process for the synthesis of AgNPs from various plant extracts.



Figure 6. Synthesis scheme of AgNPs with plants

Two of the most significant formation processes are considered during synthesis: nucleation, which requires high activation energy, and growth, which requires low activation energy. On the other hand, stabilization agents play a very important role in the synthesis. Thanks to this agent, nanoparticles are protected in a way that prevents unexpected aggregation during the control phase of their size and shape (Figure 7). The capping agents, relatively large concentrations of steroids, saponins, carbohydrates, and flavonoids, reducing agents, and phytoconstituents are the agents that give stability to the AgNPs. In general, the green synthesis mechanism of AgNPs occurs in a three-step procedure (Figure 7):

- I. Formation of small particles in the environment.

- II. These newly created small particles grow in size.
 III. Prevention of nanoparticle aggregation by stabilization step.¹⁵⁷

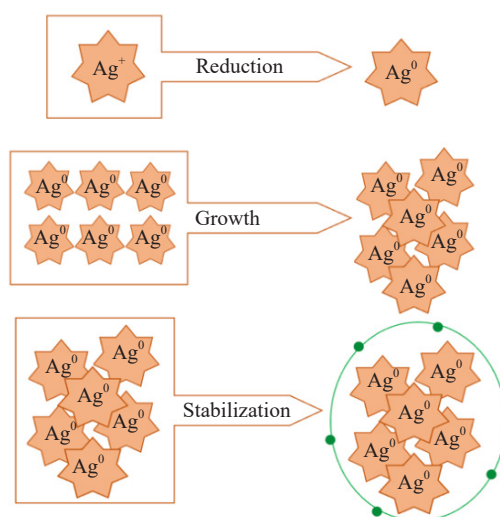


Figure 7. Reduction, growth and stabilization of AgNP

The size, shape, morphology and yield of AgNPs depend on certain factors¹⁵⁸ listed below:

- Heat,
- pH,
- Concentration of plant extracts,
- Volume of plant extracts
- Concentration of silver salt solution
- Reaction time.^{73,110}

AgNPs synthesized with plant extracts form more stable nanoparticles and can be synthesized at a higher rate compared to their synthesis with microorganisms. Studies have reported that the bioreduction potential of plant extracts is relatively higher than that of microbial cultures.¹⁵⁹ Plant-mediated synthesis requires less or almost no contamination, thus reducing the impact on the environment. Table 1 lists some important results of the biosynthesis of AgNPs from plant extracts. When the table is examined, it shows that literature studies mostly prefer the leaf parts of plants and the resulting nanoparticles are spherical in shape. The synthesized nanoparticle sizes are quite variable and range from 5 to 100 nm. Ren et al. confirmed by FTIR that *Ginkgo biloba* leaves contain polyphenols. They reported that thanks to this structure in the plant leaf, macromolecular compounds with hydroxyl groups are oxidized and therefore reduced to Ag^+ AgNP.¹⁶⁰ A different study described a similar response mechanism.¹⁶¹ According to this study, the reduction of Ag^+ is caused by two benzene rings present in the phytochemical. Ag^+ then oxidizes the tannin structures in the environment, resulting in the formation of an intermediate silver complex. Silver ions were finally created. Free electrons help reduce silver ions to zero-valent silver throughout the synthesis process.¹⁶¹ It has been discovered that polyphenolic chemicals included in leaf extracts cause general interactions. The presence of proteins and secondary metabolites in the structure of geranium leaves was observed by Shankar et al. According to their theory, terpenoids in the leaf aid in the reduction of silver ions, which oxidize to form carbonyl groups.¹⁶² It was also reported that *Pedaliium murex* leaf synthesized AgNPs.¹⁶³ The generated AgNPs were round and had an average size of 50 nm, according to TEM micrographs. In a different study, *Mukia maderaspatana* leaf extract was used to create AgNPs with a size range of 58-458 nm.¹⁶⁴ AgNPs were synthesized by Raju et al. using live peanut seedlings.¹⁶⁵ The biosynthesized AgNPs were seen under TEM to have a variety of sizes and forms, including spherical, hexagonal, triangular, square, and rod-shaped. They stated that the majority of the AgNPs that formed were spherical, with an average size of 56 nm. The NPs generated were indeed silver, as validated by the EDX technique.

Table 1. Green synthesis of AgNPs by different researchers using plant extracts

Plant name	Parts used	Size (nm)	Shapes	Ref.
<i>Acalypha indica</i> Linn	-	20-30	Spherical	166
Mulberry fruit (<i>Morus alba</i> L.)	Fruit	80-150	Spherical	167
<i>Annona reticulata</i>	Leaves	7-8	Spherical	168
<i>Cissus quadrangularis</i>	Leave	15-23	Spherical; Cubic	169
Apple, orange, tomato, red pepper, white onion, garlic, radish	-	9-30 ± 2	-	170
<i>Sesbania grandiflora</i>	Leave	10-25	Spherical; Face centered cubic	171
<i>Fraxinus excelsior</i>	Flower	15-115	-	172
<i>Hibiscus cannabinus</i>	Leave	9	Spherical; Face centered cubic	173
<i>Piper nigrum</i> , <i>Ziziphus Spina-Christi</i> and <i>Eucalyptus globulus</i>	Leaves	8-35	Spherical	174
<i>Phyllanthus emblica</i>	Fruit	16.29	Spherical	175
<i>Diplazium esculentum</i>	Leaves	23.385 ± 8.349	Quasi-spherical, hexagonal; Ellipsoidal shapes	5
<i>Abrus precatorius</i> L.	Leave	19	Disk shape; Face centered cubic	176
<i>Blumea eriantha</i> DC		50	Spherical	177
<i>Tectona grandis</i> Linn.	Leave	28	Spherical; Face centered cubic	178
<i>Saraca indica</i>	Leave	23 ± 2	Spherical	179
<i>Camellia sinensis</i>	-	2-4	Spherical	81
<i>Persea americana</i>	Seed	20-40	Spherical	180
<i>Cassia alata</i>	Leave	> 41	Spherical; Face centered cubic	181
<i>Dolichos lablab</i>	Leaves	9	Spherical	182
<i>F. Vulgare</i>	Seed	49.62	Spherical	183
Jasmine flower	Flower	40	Fiber shape	184
<i>Pedaliium murex</i>	Leave	20-50	Spherical; Face centered cubic	185
<i>Alternanthera tenella</i>	Leave	48	Spherical	186
<i>Berberis vulgare</i> , <i>Brassica nigra</i> , <i>Capsella bursa-pastoris</i> , <i>Lavandula angustifolia</i> and <i>Origanum vulgare</i>	Plant	14.7 ± 7.9 to 75.7 ± 17.1	Spherical, Octahedral	187
<i>Araucaria angustifolia</i>	Nuts	91 ± 5	-	188
<i>Azadirachta indica</i>	Leave	34	Spherical	189
<i>Morinda citrifolia</i> L.	Leaves, Fruit Pulp, Seeds	3-11	Spherical	190
<i>Caesalpinia pulcherrima</i>	Leaves	9	Spherical	191
<i>Panax ginseng</i>	Root	5-15	Spherical	192
<i>Ixora coccinea</i>	Leave	13-57	Spherical; Face centered cubic	193
<i>Mimusops elengi</i> , Linn	Leave	55-83	Spherical; Face centered cubic	194
<i>Carya illinoensis</i>	Leaves	12-30	Spherical	195
<i>Mentha piperita</i>	Leaves Extract	35	Spherical	196

Table 1. (cont.)

<i>Plant name</i>	Parts used	Size (nm)	Shapes	Ref.
Felty Germander	Stem and Flower	10 to 1,000	-	197
<i>Tinospora cordifolia</i> Miers	Leave	55-80	Face centered cubic	198
<i>Coccinia grandis</i>	Leave	20-30	Spherical; Face centered cubic	199
<i>Annona squamosa</i>	Aqueous Peel	35±2	Spherical; Face centered cubic	200
<i>Hibiscus rosa sinensis</i>	Leaves	13	Spherical/prism	201
<i>Limonia Acidissima</i>	Leave	20-40	Spherical; Face centered cubic	202
<i>Premna serratifolia</i> L.	Leave	15-100	Cubic; Face centered cubic	203
<i>Strychnos potatorum</i> Linn.F.	Leave	18-60	Spherical	204
<i>Suaeda monoica</i>	Leaves	31	Spherical	205
<i>Cissusquadrangularis</i>	Leave		Spherical; Cuboidal	206
<i>Catharanthus roseus</i>	Leaves	35-55	Cubical	207
<i>Ocimum sanctum</i>	Leaves Extract	10-20	Spherical	208
<i>Ocimum tenuiflorum</i>	Leaves	25-40	Spherical	209
<i>Dillenia indica</i>	Fruit	40-100	-	210
<i>Solanum lycopersicum</i>	Fruit	10	Spherical; Face centered cubic	211
Mango	Peel	7-27	Quasis-spherical; Faced centered cubic	212
<i>Ginkgo biloba</i>	Leaves	15-500	Cubic	213
<i>Argemone mexicana</i>	Leaves Extract	30	Spherical, Hexagonal	214
<i>E. scaber</i>	Leaves	37.86	Spherical	215
<i>Sesuvium portulacastrum</i>	Callus Extract	5-20	Spherical	216
<i>Ocimum sanctum</i>	Aqueous Leave	6-110	Triangle; Face centered cubic	217
<i>Cinnamomum camphora</i>	Sun Dried Leaves	3.2-20	Cubic hexagonal crystalline	115
<i>Chenopodium album</i>	Leave	10-30	Spherical	110
<i>Euphorbia nivulia</i>	Stem Latex	5-10	Spherical; Face centered cubic	218
<i>Astragalus gummifer</i>	Latex	13.1 ± 1.0	Spherical; Face centered cubic	219
<i>Boswellia serrata</i>	Latex	7.5 ± 3.8	Spherical; Face centered cubic	220
<i>Lippia citriodora</i>	Leaves Extract	15-30	Crystalline	221
<i>Citrullus colocynthis</i>	Leaves	31	Spherical	222
Cannonball leaves		28.40	Sphere	223
<i>Piper nigrum</i>	Seed	20-50	Spherical; Face- centered cubic	224
<i>Syzygium aromaticum</i>	Seed	20-149	Spherical	225
<i>Nymphae odorata</i>	Leaves	15 ± 5	Spherical	226
<i>Capparis zeylanica</i>	Leaves	23	Spherical	227
<i>Cocos nucifera</i>	Mesocarp Layer	23 ± 2	Spherical; Face centered cubic	228
<i>Allium sativum</i>	Garlic	7.3 ± 4.4	Spherical; Face centered cubic	229

Table 1. (cont.)

<i>Plant name</i>	Parts used	Size (nm)	Shapes	Ref.
<i>Calendula officinalis</i>	Seed	7.5	Spherical	230
<i>Allophylus cobbe</i>	Leave	2-10	Spherical	231
<i>Tribulus terrestris</i>	Fruit	16-28	Spherical; Cubic	125
<i>Syzygium cumini</i>	Leaves and Seed	29-92	Spherical	232, 233
<i>Macrotyloma uniflorum</i>	Seed	12	Spherical; Face centered cubic	234
<i>Catharanthus roseus</i>	Leave	48-67	Cubical; Face centered cubic	235
<i>Tanacetum vulgare</i>	Fruit	16	Spherical	236
<i>Artemisia capillaris</i>	Water and Ethanol Leave Extrac	Water extract -29.71 Ethanol extract -29.62	-	237
<i>Citrus limon</i>	Leave	8-15	Heterogeneous shape	238
<i>Artemisia nilagirica</i>	Leave	70-90	Spherical; Square; Hexagonal	239
<i>Cycas</i>	Leaves	2-6	Spherical	240
<i>Moringa oleifera</i>	Leave	5-80	Spherical; Face centered cubic	241
Carob	Leaves	5-40	Spherical; Face centered cubic	242
<i>Chrysanthemum morifolium</i>	Leave	20-50	Spherical; Face centered cubic	243
<i>Solanum trilobatum</i>	Leave	15-20	Cubic and hexagonal shape; Cubic and orthorhombic	244
<i>Jatropha curcas</i>	Latex	10-20	Face-centered cubic	245
<i>Acalypha indica</i>	Leaves	20-30	Spherical	246
<i>Butea monosperma</i>	Leave	5-30	Spherical	247
<i>Nelumbo nucifera</i>	Leave	30-40	Spherical	248
<i>Cycas circinalis, Ficus amplissima, Commelina benghalensis and Lippia nodiflora</i>	Leave	13-51	Spherical; Face centered cubic	249
<i>Psidium guajava</i>	Leave	10-90	Spherical	250
<i>Phlomis</i>	Leave	25	Spherical; Face centered cubic	251
<i>Alternanthera bettzickiana</i>	Leaves	5-15	Spherical	252
<i>Abutilon indicum</i> [L.]	Leave	106	-	253
<i>Kalopanax pictum</i>	Leave	30 at 20 °C and < 10 at 90 °C	Spherical; Face centered cubic	254
<i>Petroselinum crispum</i>	Leave	30-32	Spherical	255

These metal nanoparticles obtained by green synthesis using plants are used in the removal of various pollutants including dyes, heavy metals and organic pollutants from water. There are many studies in the literature on the successful use of various mono and bimetallic nanoparticles such as Fe, Au, Ag, ZnO, TiO₂, Fe-Cu, Fe-Pd in the removal of heavy metals, dyes and pharmaceutical active ingredients from water.^{30,32,35,256} Among the pollutants in water, dyes in particular significantly affect aquatic life and the food web even at low concentrations. Dyes used in various industries generally produce colored wastewater with high chemical and biological oxygen demand and different pHs depending on the process and type of dye used. Treatment of wastewater containing these dyes is important because they pose a direct risk to the ecosystem.^{11,257} For all these reasons, studies on dye removal from water using metal nanoparticles have attracted attention in recent years.^{15,17,154,258-261}

3.4 Potential cationic dye removal of AgNPs synthesized using lignocellulosic biomass

Today, water is the most important natural resource. 97.5% of the water in the world consists of salt water. The remaining 2.5% belongs to fresh water resources. Population growth in the world, global warming, drought, irregular urbanization, rapid industrialization and climate changes are important factors that cause a gradual decrease in clean and drinkable water resources. Therefore, control of water pollution is becoming increasingly important. Pollution of existing drinkable water resources will cause problems such as thirst in the future. For this reason, scientists have recently paid great attention to the issues of environmental pollution and water purification.²⁶² Some of the dyes need to be eliminated from wastewater because they are carcinogenic and block sunlight, reduce photosynthesis and cause visual pollution due to the color they create in water.²⁶³⁻²⁶⁴ Great importance is given to the removal of dyestuffs, which are especially harmful to human health, from industrial wastewater. However, while some of these studies remove pollutants in the relevant environment, they may have negative effects on the habitat in other environments. For example, some chemicals used in water purification purify water, but when the chemicals used mix with the receiving environment, the living things in that environment can be negatively affected. The emergence of these problems has led to the development of technologies that will cause less or no harm to the environment. Today, the concept of “green chemistry” emerges as a branch of environmentally friendly approaches. The main goal of green chemistry is to minimize or never allow waste to be generated, rather than eliminating waste after it occurs. Nanoparticle synthesis is highly developed in today’s engineering conditions. These nanoparticles are used as adsorbents and catalysts in environmental engineering, especially in water treatment, and show successful results. However, solvents and reducing agents used in a number of chemical production stages increase environmental concerns. For these reasons, it is very important to follow a path in which the green engineering principle is adopted in adsorbent production.¹³⁰

The use of nanomaterials is rapidly increasing thanks to their high surface area, free surface energy, small size and active atoms, and their high surface area depending on volume ratio also increases the sorption capacity.³⁵ Moreover, some of its attributes are mentioned, including its adsorption capacity, tiny size effect, quantum tunnel effect, macro quantum impact, surface effect at the nanoscale and reactivity which are extremely suitable for the removal of pollutants such as cationic dyes.²⁶⁵ These substances, which have a small size, are formed on a concentrated atomic level surface area, and in this case, it has been stated that these adsorbents increase the reduction capacity. In addition, it has been stated that the reaction can take place in a shorter time with a lower adsorbent requirement.²⁵⁶ Extensive research has been done on the testing and evaluation of plants in the synthesis of AgNPs, and there are many studies in the literature on the removal of cationic dyes (Table 2).

Table 2. Examples of cationic dye removal with AgNPs synthesized from various plant materials

Plant name	Parts used	Size (nm)	Shapes	Dye	Dye removal or Max. Removal capacity	Ref.
<i>Diplazium esculentum</i>	Leaves	23.385 ± 8.349	Quasi spherical;	MB	91%	5
Jasmine flower	Flower	40	Fiber	MB	78%	184
<i>Aegle marmelos</i> leaf	Leaf	N.A.	N.A.	Malachite green (MG)	-	266
<i>Cocos nosifera</i>	Mesocarp of the fruit	30-50	Sphere	MG	-	267
<i>Vigna unguiculata</i> L. stem	Stem	25	Face centered cubic	MG	268.82 mg·g ⁻¹ (21.6% at 200 mg·L ⁻¹)	149
<i>Luffa acutangula</i>	Pale yellow flower	10-30	Spherical; Face centred cubic	MB MG	-	258
<i>Malus domestica</i> -Green Delicious <i>Lagenaria siceraria</i>	Starch	37.59 33.87	Spherical	MG	85.01% 95.90%	268
<i>Morinda tinctoria</i>	Leaves	79-96	Spherical; Rod	MB	95%	19
<i>Imperata cylindrica</i>	-	31	Face centred cubic	MB	92.06%	18
<i>Gymnema sylvestre</i>	Leaves	1 µm to 200-400 nm	Spherica; Rhombohedral	MB	95%	269

Table 2. (cont.)

Plant name	Parts used	Size (nm)	Shapes	Dye	Dye removal or Max. Removal capacity	Ref.
<i>Cynodon dactylon</i> (L.) Pers	Leaves	13	Spherical	MB	75%	16
<i>Chenopodium botrys</i>	Flowers	11.9	Spherical	MB	90.09 mg·g ⁻¹	270
<i>Ruellia tuberosa</i>	Leaves	55.65	Spherical	Crystal Violet (CV)	87%,	271
<i>Carissa carandas</i>	Fruit	23 ± 2	Spherical	CV	93%	272
<i>Sanguisorba officinalis</i>	Leaves and stem parts	10-50	Spherical	CV	90%	273
<i>Kalanchoe brasiliensis</i>	Leaves	17	Spherical	Aniline Blue (AB), Toluidine Blue (TB), Congo Red (CR), Indigo Carmine(IC), Auramine O (AO)	AB- 86.04% CR- 85.95% AO-84.08% TB- 78.85% IC- 70.40%	17
Discarded yerba mate extract	-	24.07 ± 3.00	Spherical	CV, MB, Safranin	For the three dyes tested, more than 70% of removal could be achieved	274
<i>Saussurea costus</i>	Root	5 to 15	Spherical	Safranin	84.6%	259
<i>Urena lobata</i>	Leaves	20	Spherical	MB	87.47% 218.95 mg·g ⁻¹	275
Cauliflower	Leaves	35.08	Spherical	MB	97.57%	260

The synthesis of AgNPs is greatly affected by various factors (green synthesis or traditional methods). These factors include solvent, reductant, size distribution, surface chemistry, morphology, coating materials, surface charge, metal salt concentration, pH, temperature, and time. As a result, we can say that each synthesis method has its own benefits and limitations. In the synthesis of AgNPs used in dye removal, the type of solvent, reductant, and the time required for dye degradation determine the overall efficiency of the synthesized AgNPs.²⁷⁶ On the other hand, according to previous studies, AgNPs synthesized by green methods have better environmental biocompatibility compared to those synthesized by physical and chemical methods.²⁷⁷⁻²⁷⁹ AgNPs were synthesized by green and chemical methods; In the green method, *Mussaenda frondosa* (*M. frondosa*) leaf extract and in the chemical method, sodium citrate were used as reducing and stabilizing agents for the synthesis of AgNPs.²⁸⁰ They characterized the synthesized AgNPs using UV-vis spectroscopy, FTIR, XRD, TGA and TEM. There are some studies, although not many, on the synthesis of AgNPs by different methods and their comparison in the removal of cationic dyes. For example, in a study conducted by Kumar et al. in 2019, they synthesized AgNPs with different methods. They used green tea extract in AgNP synthesis with green synthesis, NaBH₄ solution in AgNP inorganic synthesis, and glucose in AgNP organic synthesis.²⁸¹ When they compared the MB removal of these AgNPs synthesized with different methods, they saw that the removal was 65% with green synthesis. They reported that these AgNPs they synthesized were effective and fast for cationic dye removal at room temperature. In a study conducted by Pandey et al., AgNPs synthesized using κ-carrageenan gum achieved 100% MB removal in only 70 seconds in the presence of NaBH₄.²⁸² This showed an extraordinarily significant degradation activity. These results are quite unusual when compared to the existing literature based on various catalyst systems where the dye degradation kinetics are slow kinetics. In another study, Khodadadi et al. synthesized AgNPs using *Vaccinium macrocarpon* fruit. They fixed these AgNPs, which they synthesized using the green method, onto the surface of clinoptilolite using a green approach. MB degradation with Ag-NPs/clinoptilolite occurred in only 40 seconds.²⁸³ When compared to other methods published in the literature, AgNPs obtained from *Vaccinium macrocarpon*/clinoptilolite were reported to be one of the least time-consuming studies in removing organic dyes. Although the specific reason for this rapid removal is not discussed in the literature, it can be said that the combined effect of bioactive compounds in the extract of the fruit studied in this study, pores in the structure of clinoptilolite and catalytic properties of AgNPs created a synergistic effect in providing rapid dye removal.

For example, Anupama and Madhumitha used *Carissa carandas* dried fruit extract in the synthesis of AgNPs in their study. They characterized the prepared AgNPs with UV-Vis spectrometry, FTIR. They tested their catalytic activity in the degradation of CV dye. In their study, they examined the reduction of only dye, dye + extract and dye + extract +

AgNPs.²⁸⁴

In a study conducted by Saha et al., the biological synthesis of AgNPs was developed using AgNO₃ solution at 1 mM concentration and fruit extract of *Gmelina arborea*. The prepared AgNPs were characterized by UV-Vis spectroscopy, TEM, SAED and EDX. TEM studies showed that the synthesized AgNPs were stable, spherical and crystalline with particle size ranging from 8 to 32 nm. The prepared AgNPs were used in the adsorption reactions of MB dye, and the adsorption process completed within 10 min confirmed the existence of good adsorbent property of AgNPs.²⁸⁵

Sharma et al. synthesized AgNPs by green synthesis using onion juice and tested the degradation of various dyes. As a result of their studies, they stated that onion-coated AgNPs are excellent catalysts in the degradation of dyes such as MB, methylene red, eosin yellow and safranin.²⁸⁶

In a study by Mokhtar et al., an effective solid adsorbent, activated carbon (AgNPs-AC) loaded with AgNPs, was produced to remove CV dye. Substances that function as stabilizers for silver nitrate (AgNO₃) were provided from *Clitorea ternatea* flower extract. AgNPs were reported to have an average size of 16.11 nm. The optimal values of the parameters for the best yield (97%) were AgNPs to AC ratio (1.0 g), amount of adsorbent 30 mg, time of 240 min, and pH 10. Data show that the best condition is an alkaline environment.²⁶¹ The reason for this is that the strong electrostatic attraction between the cationic CV dye and its negative charge on the adsorbent surface in alkaline environment causes an increase in CV dye adsorption.

Yari et al. successfully synthesized AgNPs using *Chenopodium botrys* extract as a simple and environmentally friendly method to evaluate their efficient applicability as a dye-removing nanomaterial. First, for the plant extract, fresh flowers of *C. botrys* were washed with distilled water, dried and powdered. Then, these powders were added to distilled water and mixed at 60 °C to obtain the filtrate. To synthesize AgNPs, *C. botrys* extract was added to the prepared AgNO₃ (0.01 M) solution and mixed mechanically. It has been reported that in the reduction step, silver ions are reduced and stabilized by electrostatic interaction thanks to the biomolecules in plant extracts.²⁷⁰ Here, they reported that the synthesis of AgNPs depends on chemicals such as flavonoids, aldehydes, carboxylic acids, terpenoids, amides and quinine found in the plant extract.²⁸⁷ The change in the color of the solution from yellow to dark brown showed that AgNPs were synthesized. The AgNPs they synthesized were characterized by XRD, FTIR, SEM, TEM, and energy dispersive X-ray spectroscopy (EDAX) techniques. The size of AgNPs was obtained as 12 nm. They investigated the removal of MB and methyl orange (MO) of AgNPs and determined that the color removal of the dyes in the presence of AgNP was 97.5% and 95.0%, respectively. The zero charge point (pHpzc) value of the synthesized AgNP is 7.82. At pH values lower than pHpzc, positive charge distribution occurs due to protonation of the AgNP surface, and the positively charged MB causes repulsion between them, therefore it has been reported that the removal percentage decreases. At pHs higher than pHpzc, due to deprotonation on the surface of AgNPs, the negative charge on the AgNPs surface attracts the positive charge of MB and the dye removal percentage increases.²⁸⁸ As a result of the experiments, the highest removal of MB occurred at pH = 10. Adsorption provided a good fit with the pseudo-second-order kinetic model. It has been reported that equilibrium data are better represented by the Langmuir isotherm. Maximum adsorption capacities of 90.09 and 80 mg·g⁻¹ were obtained for MB and MO, respectively.

In their study, Jyoti and Singh used AgNPs synthesized from *Zanthoxylum armatum* leaves in the removal of hazardous dyes such as Safranin O, Methyl Red, MO and MB. The formation of AgNPs was evaluated by UV-Vis spectroscopy. DLS, SEM-EDX, TEM, SAED and XRD studies showed that AgNPs are crystalline in nature and the size range is between 15 and 50 nm. The removal rate constants of Safranin O, Methyl red, MO and MB in 24 hours are 1.02 × 10⁻³ min⁻¹, 1.03 × 10⁻³ min⁻¹, 1.86 × 10⁻³ min⁻¹ and 1.44 × 10⁻³ min⁻¹ respectively. It was emphasized in the study that AgNPs were observed to be a good catalyst in the removal of hazardous dyes.²⁸⁹

In a study by Anjana and Geetha, the biosynthesis of AgNPs and the dye absorption properties of AgNPs were examined using the leaf extract of *Cynodon dactylon* (L.) Pers and solutions with different AgNO₃ concentrations. UV-VIS, XRD and SEM characterizations were performed to confirm the formation of AgNPs. XRD and SEM analysis showed the presence of spherical and homogeneous AgNPs, with the size of AgNPs approximately 13 nm. Various concentrations of nanoparticles (2.0-10.0 mg) were mixed with water containing MB dye (10 mg/1,000 mL) and removal rates were measured at certain day intervals. The percentage of dye absorption calculated using absorbance values increased as the day increased. Dye solution containing 10 mg of AgNP from various AgNP concentrations used showed 75% dye removal after 5 days of mixing at room temperature.¹⁶

Gowda et al. used the phytochemicals in *Urena lobata* leaf extract to create AgNPs (UL-AgNPs). Results of the study of the leaves of *Urena lobata* revealed the presence of phytochemical substances, including alkaloids, glycosides, tannins, saponins, and phenols, which are assumed to be involved in the synthesis of AgNPs.^{43,62} Plant extract formed AgNPs, which caused the hue to change from pale yellow to brown when combined with silver nitrate solution. UL-AgNP production was measured and found to be 96.72 mg·L⁻¹. The produced UL-AgNPs had a 20 nm diameter and resembled nanospheres. According to reports, the synthesized UL-AgNPs were able to remove 87.47% of the MB dye with a maximum adsorption capacity of 218.95 mg·g⁻¹. This was made possible by their huge surface area. The adsorption of MB by UL-AgNPs obeyed pseudo-first-order kinetics ($k_1 = 0.878 \text{ h}^{-1}$), monolayer deposition was found in the Langmuir isotherm ($R^2 = 0.996$) as the most suitable equilibrium isotherm, chemisorption was dominant, and the R_L value (0.595) showed that the process was spontaneous.²⁷⁵

Ajitha et al. synthesized AgNPs with green synthesis using *Phyllanthus amarus* leaf extract, identified them by UV, FT-IR, XRD and TEM analyses, and tested the microbial effect and catalytic activities of AgNPs. As a result of their studies, they stated that the AgNPs they synthesized would find potential applications in the biomedical field due to their antimicrobial effects. They also stated that they exhibited excellent catalytic behavior in the degradation of rhodamine B dye, and the dye was reduced in 20 minutes.²⁹⁰

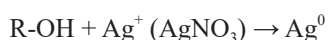
In the study by Kumar et al., AgNPs were synthesized by the green synthesis method using *Gymnema sylvestre* plant leaves aqueous extract. According to SEM analysis, nanoparticles with high agglomeration were found to have spherical, rhombic shapes and size varies from 1 μm -200-400 nm. The removal efficiency of the synthesized AgNPs was examined by adsorption of MB to the surface of AgNPs under sunlight. Green synthesized AgNPs effectively removed approximately 95% of the dye within 7 h of exposure. It was found that the synthesized AgNPs showed excellent adsorption effects against dye molecules and could be used in water purification systems and treatment of dye wastes.²⁶⁹

Vanaja et al. successfully synthesized AgNPs using *Morinda tinctoria* leaf extract and 1 mM AgNO₃ solution at different pH values. The synthesized AgNPs were characterized by UV-Vis spectroscopy, SEM, XRD and EDX. According to SEM analysis, nanoparticles with high agglomeration in the size range of 79 to 96 nm were reported to be spherical and rod-shaped. The absorption capacity of the synthesized AgNPs under sunlight of MB was also examined. It was stated that nanoparticles effectively removed approximately 95% of the dye within a 72-hour reaction time. It has been stated that synthesized AgNPs show high removal efficiency against dye molecules and can be used in water purification systems and treatment of dye wastes.¹⁹

Poiba et al. carried out AgNP synthesis studies using *Grevillea bobusta* leaves and obtained nanoparticles with an average size of 200 nm in 40 minutes at 80 °C. With synthesis, a change in color from light green to brown was observed. They predicted that Ag⁺³ ions would be reduced to AgNPs with the rapid formation of a brown precipitate. They used the AgNPs they obtained in the color removal studies of MB dye. According to the experimental data, at pH < 6, the surface charge becomes positively charged, causing the (H⁺) ions on the surface to compete effectively with the dye cations. As a result, a decrease in the amount of adsorbed dye was observed. It has been reported that the uptake of cationic dyes increases due to the increased electrostatic attraction force with the negative charging of the adsorbent surface at pH 6. For maximum MB removal, contact time of 60 minutes, concentration of 30.0 g·L⁻¹, pH of 6, and dose of 0.4 g·L⁻¹ were obtained.¹⁵

In a study by Rao et al., AgNPs were synthesized using *Grevilla robusta* leaves and 1 mM AgNO₃ solution. The synthesized nanoparticles were characterized using the SEM technique and their dimensions were found to be 200 nm. In the study, the removal of Congo Red dye with nanoparticles synthesized in aqueous medium based on different parameters was examined. The dye removal efficiency obtained under optimum conditions was 96%. The pseudo-second-order kinetic model showed good fit to the experimental study. It has been said that this method can be used for the removal of many other industrial dyes.²⁹¹

Paul et al. carried out AgNPs synthesis studies using the leaves of the *Calendula officinalis* plant (fresh leaves) and synthesized AgNPs at different concentrations (1 mM and 2 mM) of silver nitrate and determined that the average particle sizes were 50-60 nm and 140-150 nm.²⁹² The pale yellow to brown hue shift indicated the production of AgNPs. Through FTIR analysis, the presence of -OH stretching in flavonoids, xantonoids, and phenolic compounds which are thought to be the primary reducing agents for silver ions was demonstrated by the broad peak at 3,338 cm⁻¹, which identified the primary functional groups present in *Calendula officinalis* leaf extract responsible for the synthesis of AgNPs. It was attributed to the reduction of silver ions (Ag⁺) by OH-based products present in the leaves.^{25,26}



These functional groups were reported to be responsible for the reduction of silver ions into AgNPs. The removal of both MB and MO dyes was investigated with these synthesized AgNPs. While 69.79% color removal of MB dye was achieved in 5 minutes, 80% color removal of MO dye was achieved in 8 minutes.²⁹²

Hadi et al. successfully carried out AgNP synthesis studies by reducing AgNO₃ using *Diplazium esculentum* plant extract. As a result of their characterization studies, they revealed that AgNPs with an average particle size of 23.385 ± 8.349 nm, obtained by HRTEM analysis, were formed in spherical, hexagonal and ellipsoidal shapes. It was supported by XRD study that AgNPs have a crystalline and face-centered cubic structure. It has been reported that the presence of functional groups possessed by *D. esculentum* in the FTIR spectrum of AgNPs indicates its function as reductants and stabilizers. They also investigated the effect of AgNPs on MB removal and determined that color removal could be achieved up to 91% in the presence of AgNPs.⁵

In a study by Pandian et al., the removal of MG from aqueous solution was studied using AgNPs synthesized from *Allium sativum* plant using the green synthesis method. Parameters affecting AgNPs synthesis, such as temperature of the environment, plant extract concentration, AgNPs concentration and pH, were optimized. In this study, a maximum of 2.1 g/100 mL AgNPs were obtained. Experimental data were analyzed using Langmuir, Freundlich, Dubinin-Radushkevich, Temkin and Sips isotherm models and it was reported that the Langmuir model provided a better fit. It was stated that in the optimized case, the maximum removal of more than 90% of the MG dye occurred and the Langmuir model showed a better fit with a maximum adsorption capacity of 54.0504 mg·g⁻¹. According to the data obtained, they reported that AgNPs synthesized by *Allium sativum* were a good alternative for MG removal from water.¹⁴

In a study by Fairuzi et al., AgNPs were synthesized by the green synthesis method using *Imperata cylindrica* plant extract and 5 mM AgNO₃ solution. Agglomeration in the microstructure of biosynthesized AgNPs was confirmed by FESEM, and average particle sizes were measured as 31 nm. XRD analysis showed that AgNPs have a face-centered cubic structure. EDX analysis revealed the presence of elemental silver contributing 68.44 wt% of the analyzed sample. The removal of MB by sodium borohydride (NaBH₄) was carried out in the presence of AgNPs as a catalyst. With the addition of biosynthesized AgNPs, the removal of MB from aqueous media increased up to 92.06% within 14 min.¹⁸

Dawodu et al. synthesized AgNPs from *Vigna unguiculata* L. stem extract and investigated the application of these nanoparticles as adsorbents for MG.¹⁴⁹ In the synthesis of AgNPs, AgNO₃ solution was used as a precursor and plant extract was used as a reducing and capping agent. They reported that the alkaloids found in the stem of *Vigna unguiculata* L. played a role in reducing AgNO₃. Bioreduction of Ag⁺ ions to Ag⁰ was confirmed using UV-Vis spectrophotometry. They characterized the AgNPs using UV-Vis, SEM-EDX, FTIR and XRD and reported that the AgNPs showed SPR bands at 455 nm, indicating face-centered cubic crystal structure with an average crystal size of ~ 25 nm. In MG removal studies using synthesized AgNP, it was observed that the adsorption process was exothermic, and it was more in line with the Langmuir isotherm with equilibrium isotherm studies. This showed that monolayer adsorption was achieved. Adsorption capacity, q_e (mg·g⁻¹), was observed to increase from pH 3 to pH 9, from 17.65 to 80.39, maintain constant, and decrease at pH 12. This information was derived from the experimental results. It was discovered that AgNPs have a zero charge point (pHpzc) of 8.6. The adsorbent's surface is primarily negatively charged when the pH of the solution is above pHpzc and net positively charged when the pH is below pHpzc. As a result, at pH values lower than 8.6, the adsorbent surface becomes extremely cationic and there is electrostatic repulsion between the adsorbent and the adsorbate, which reduces the adsorption capacity. However, at pH 9, AgNPs' surface become anionic, which increased the electrostatic attraction force and made it easier for cationic dyes to adsorb in the surrounding environment. The synthesized AgNPs can be used as adsorbents for MG, but they showed low removal percentage (21.6% at 200 mg·L⁻¹) for the removal of MG dye.¹⁴⁹

4. Conclusion

Nanoparticle synthesis is accomplished via physical, chemical, and biological means. The disadvantages of physical and chemical methods such as expensive, toxic and excessive energy consumption encourage researchers to

focus on a cheap, environmentally friendly and safer approach. Increasing cancer cases and environmental pollution have encouraged scientists to look for alternative and natural ways. Synthesis of metal nanoparticles using extracts of lignocellulosic biowastes, which is one of the biological synthesis methods, attracts attention because it is a fast, environmentally friendly, easily accessible, non-pathogenic, economical and one-step technique. Additionally, there is no need to add any external stabilizing agents. When these studies conducted in recent years were examined, it was concluded that plant extracts can be used for the synthesis of AgNPs in the biological synthesis method. Important processes include the synthesis of AgNPs using plant extracts, the manufacture of plant extracts utilizing various plant parts or the entire plant, depending on the extract's content, and the creation of metal salt solutions. Generally speaking, the green synthesis process involves combining plant extract with a metal salt solution at the appropriate pH and temperature to synthesise metal nanoparticles (NPs) using plants. Metal ions bind to compounds found in plant metabolisms and are reduced to metal atoms. By mixing at room temperature, stable nanoparticle formation occurs by surrounding the core with coating and stabilizing agents found in the plant. Especially terpenoids, flavonoids, polyphenols, alkaloids, proteins and carbohydrates found in plant material as phenolic components serve this purpose. AgNPs, metallic nanoparticles and nanocomposite materials produced by biosynthesis method have a wide range of applications in different fields of technology and science. Dyes have the potential to pose a risk to receiving waters because they are not biodegradable and are likely to contain toxic compounds. For this reason, color removal processes containing dyestuffs from wastewater are gaining ecological importance. In particular, AgNP is one of the promising agents for reducing the negative properties of synthetic dyes. The fact that the synthesized AgNPs provide high removal rates in the removal of cationic dyes from aqueous solutions shows their applicability in the waste treatment of pharmaceutical, cosmetics, paint, plastic and especially textile industries.

Conflict of interest

The author declares that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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