

**Review on Environmental friendly Synthesis and characterization of TiO<sub>2</sub> Nanoparticles Using different part of plant Extract and Its application**

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**Abstract**

Recent advances in nanoscience and nanotechnology have radically revolutionized how we diagnose, treat, and prevent disease in all aspects of human life. Among the numerous metallic nanoparticles employed in biomedical applications, silver nanoparticles (AgNPs) are among the most important and intriguing nonmaterials. AgNPs have considerable applications in nanoscience and nanotechnology, particularly in nanomedicine. Although other noble metals have been used for a number of reasons, AgNPs have garnered attention due to their potential applications in cancer detection and antibacterial capabilities. The AgNPs were characterized by UV-Vis Spectroscopy, Scanning Electron Microscopy (SEM), Energy-Dispersive X-Ray Spectroscopy (EDX), Fourier-Transform Infrared Spectroscopy (FTIR), and Dynamic Light Scattering (DLS). Nanoparticles can be produced via physical, chemical, or biological means. In this review, we look at the physical, chemical, and biological techniques of producing AgNPs. The biological synthesis of nanoparticles, also known as green synthesis is an environmentally friendly and cost-effective procedure. We also evaluate the elements that influence AgNP generation and the ways for defining them. More importantly, we discuss the multifunctional bio-applications of AgNPs, including antibacterial, antifungal, antiviral, anti-angiogenic, and anti-cancer agents, as well as the mechanism of AgNP anti-cancer activity. Finally, we will discuss the future prospects for AgNPs.

**Keywords:** green synthesis, silivernano particles, characterization, plant extract, anticancer and antidiabetics

**Introduction**

Nanotechnology can be described as the creation, representation, manipulation, and use of nanoscale structures (Sarsar et al., 2013). Nanotechnology is the most dynamic area of research in material sciences, and the production of nanoparticles (NPs) is growing rapidly around the world. Green nanoparticles (NPs) are tiny molecules that range in size from 1 to 100 nm. Their diverse physical and chemical forms are invisible to the naked eye (Zulfiqar et al., 2022). The presence of a carbon atom distinguishes organic from inorganic nanoparticles. Metallic nanoparticles such as gold and silver are examples of inorganic nanoparticles, as are semiconductor nanoparticles such as zinc oxide, titanium, and magnetic nanoparticles. Inorganic nanoparticles have lately gained popularity in a variety of applications due to their promising features. These metallic particles, for example, have been studied as medical agents for solar energy conversion, catalysis,

water purification, and the treatment of various diseases (Saha et al. 2017). The most common types of nanoparticles are semiconductors, metals, carbon-based, ceramic, polymeric, and lipid-based nanoparticles.

Nanomaterials are also classified as zero-dimensional (0D) (for example, nanoparticles), one-dimensional (1D) (for example, nanotubes & nanorods), two-dimensional (2D) (for example, graphene), and three-dimensional (3D) (for example, nanoprisms & nanoflowers) (Singh Chouhan et al., 2021). Recently, physical (sonication, laser ablation, and radiation), chemical (condensation, sol gel technique, and reduction), and biological approaches have been employed to produce NPs. Toxins may be retained in AgNPs as a result of chemical processes (some et al., 2019). Significant pressure and irradiation may be required for physical techniques (Samuel et al., 2022). Biological techniques that use bacteria, fungi, and yeast to synthesize AgNPs varying in size from 1 to 100 nm may be a safe choice for humans and the environment (Karmous et al., 2020). Green or biological nanotechnology has the potential to improve people's lives in a wide range of fields, including food, biomedical, and agricultural (Ganachari et al 2019).

In recent years, this last aspect has become more relevant in what is known as the green synthesis of chemical substances or reagents, which is defined as taking into account aspects such as the use of harmless chemical or biological precursors, or the reduction or complete absence of chemical residues that can be released into the environment. The green synthesis of nanoparticles (NPs) has four key characteristics: easy methods, non-toxicity, cost-effectiveness, and environmental friendliness. Furthermore, green synthesis procedures do not necessitate high temperatures, high pressures, or costly equipment or reagents (Maribel Santiago 2023). The uses of various natural sources to provide stabilizing agent and cover (coverage) to prevent agglomeration in the synthesis process, and thus contribute to achieving the desired shape and size of the TiO<sub>2</sub> particles, have been reported in the literature (Sethy et al., 2020).

In the green synthesis approach, various sections of plant extracts (root, stem, leaves, floor, seeds) are used to produce materials. The extracts contain a variety of phytochemicals, including flavonoids, malkaloids, phenolics, and others (Ugo et al., 2019), which are high in carbon, hydrogen, and nitrogen molecules. The plant extract reacts with metal salts, forming nanoparticles of varying sizes, shapes, and surface areas. (Shah et al. 2015).

Numerous analytical methods have been employed to assess the synthesized nanomaterials, such as atomic force microscopy (AFM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), dynamic light scattering (DLS), X-ray photoelectron spectroscopy (XPS), X-ray diffractometry (XRD), Fourier transform infrared spectroscopy (FTIR), and so forth (Gurunathan et al., 2015). Although plant leaves are a major source of metabolites and are safe to use in comparison to bacteria, algae, or any other plant tissue because they are preferably nonpathogenic, metal oxide nanoparticles and nanoparticles can be synthesized from seeds, bark, flowers, tubers, and root extracts (Ahmad et al., 2019). Also, a unique aspect that adds on to the property is the leaf extracts have a faster decrease rate than algae or bacteria (Singh et al., 2018). The two major phytochemicals found in plants, terpenoids and flavanones, are essential for stabilizing nanoparticles and serving as capping and reducing agents (Banerjee et al., 2014). Silver salt is reduced to silver nitrate by aqueous neem leaf extract, and these capped nanoparticles with neem extract have antibacterial properties. Most of these reviews focused on several plant and microbial sources for synthesis, several characterization techniques for analysis, certain tabular data representing source, shape and size and information regarding various applications. The

present review, unlike the earlier ones, summarizes the synthesis procedure, parameters, characterizations, applications and predicted antibacterial mechanism in a systematic manner, focusing on various green routes for AgNPs synthesis.

#### Green Synthesis Nano particle

The traditional methods for producing NPs are costly, hazardous, and unfriendly to the environment. To address these issues, researchers identified precise green routes, i.e., naturally occurring sources and their products that can be employed for NP synthesis. Green synthesis can be divided into three categories: (a) the use of microorganisms such as fungi, yeasts (eukaryotes), bacteria, and actinomycetes (prokaryotes), (b) the use of plants and plant extracts, and (c) the use of templates such as membranes, viruses, DNA, and diatoms. The subsequent sections address green synthesis via plants and plant extracts (Rafique et al., 2017).

Silver metal ion solution and a reducing biological agent are the essential requirements for green production of AgNPs. In most situations, reducing agents or other elements present in the cells operate as stabilizing and capping agents, eliminating the need for external capping and stabilizing agents (Srikanth et al., 2016). Green plants and microbes have an amazing ability to produce nanosilver. Biological synthesis employing plant resources and microbes has been shown to be both environmentally benign and cost-effective. Many microorganisms and plant extracts are now employed in the manufacture of nanosilver (Sarsar et al., 2014). Nowadays, green synthesis is regarded as the most suitable synthesis route. Bio-organism extracts may act as reducing and capping agents in the manufacture of silver nanoparticles. The reduction of Ag<sup>+</sup> ions by biomolecules contained in these bio-organism extracts, such as amino acids, enzymes/proteins, polysaccharides, and vitamins (Sharma, 2009), is ecologically friendly yet chemically complex (Sharma, 2009).

Mechanism for the biosynthesis of titanium dioxide nanoparticles from plant extract Sarkar et al. reported the synthesis of silver nanoparticles of various sizes at 100 °C and room temperature using parthenium leaf extract. They illustrated how particle size varies as a function of reaction temperature and duration. Colloidal silver nanoparticles that glow in the dark were generated. After 2 minutes of chemical reaction at a higher temperature of 100 °C, the size of the silver nanoparticles formed ranged between 40 and 160 nm, with an average size of 110 nm (Sarkar et al., 2010).

Mohapatra et al. reported the manufacture of silver nanoparticles using citrus limon extract. The biosynthesized silver nanoparticles range in size from 10 to 30 nm. They demonstrated that the addition of NaOH may be a critical element in the rapid manufacture of stable aqueous dispersions containing large concentrations of silver nanoparticles. As a result, the alkaline environment promotes the manufacturing of silver nanoparticles utilizing Citrus limon (Mohapatra et al., 2015). Prathna et al. produced silver nanoparticles by reducing silver nitrate solution with aqueous extract of *Azadirachta indica* leaves, and the growth kinetics of silver nanoparticles with sizes ranging from 10-35 nm were studied. Aqueous solutions of silver nitrate and natural rubber latex derived from *Hevea brasiliensis* were thermally treated to produce colloidal silver nanoparticles. Silver nanoparticles with diameters ranging from 2 nm to 10 nm were spherical in shape and had a face-centered cubic (fcc) crystalline structure (Prathna et al., 2011).

NP biosynthesis can be divided into three stages (Jameel et al., 2020; Zhang et al., 2020): I. Activation phase: In the initial phase, the metal ion gets converted from mono or divalent oxidation states to a zerovalent state. Nucleation of reduced metal ion occurs.

II. Growth phase: In this stage, small TiO<sub>2</sub>NPs formed aggregate to form larger molecules of NPs having varied shapes such as pentagonal, hexagonal, spherical, cubical, triangular, etc. In this phase, plant extract act as a capping and stabilizing agent to get stable morphology.

III. Termination phase: NP aggregates to form nanotubes, nanoprisms and a variety of other irregularly shaped NPs.

Bioreduction Mechanism:

TiO [OH]<sub>2</sub>+ Plant extracts TiO NPs + byproducts

**Nanoparticle characterization techniques**

To characterize the produced nanoparticles, several approaches were applied. Size, crystal structure, elemental composition, and variety can all be examined using multiple techniques in some circumstances. Table 1 contains information on several approaches for characterizing distinct plant-derived TiO<sub>2</sub> NPs.

Table 1: Details about various methods of characterization of different plant derived Titanium dioxide nanoparticles.

Plant name family	Parts of plant used	Characterization	Size of Titanium oxide (nm)	Shape of Titanium oxide nanoparticles	Citations
Moringaoleifera (Moringaceae)	Leaves	XRD, UV-vis spectroscopy	12.22	Spherical	Swathi et al. (2019)
Ageratinaaltissima (Asteraceae)	Leaves	XRD, FTIR, FESEM, UV-vis spectroscopy	60- 100	Spherical	Ganesan et al. 2016
Kniphofiafoliosa (Asphodelaceae)	Root	Thermo gravitmetric analysis (TGA/DTA), FTIR, TEM, XRD, UV-vis spectroscopy	8-10	Spherical	Bekele et al. 2020
Sesbaniagrandiflora (Fabaceae)		(SEM- EDX), FTIR, UV-vis spectroscopy	160 -220	Spherical diamond shape	Srinivasan et al. 2019
Azadirachtaindica (Meliaceae)	Leaves	SEM, TEM, FTIR	TEM -15-50; SEM- 25-87	Spherical	Thakur et al. 2019
Nyctanthes arbor-tristis (Oelaceae)	Leaves	Scanning electron microscope (SEM), Xray diffraction (XRD), particle size analyzer (PSA)	100-150	Cubic	64Sundrarajan et al. 2011
Trigonellafoenum-	leaf	FTIR, XRD, UV-vis,	25	Spherical shaped	Subhapiya et al.

graceum(Fabaceae)		HR- TEM, HR- SEM		polydisperse nanoparticles	2018
Moringaoleifera	Leaf extract	FTIR, XRD, Uv-Vis, SEM	100	Spherical	Sivaranjani and Philominathan (2016)
	Leaf extract	FTIR, XRD, Uv-Vis, SEM	11-18	Spherical	Sethy et al. (2020)

**UV and visible study**

To determine whether AgNPs are formed or not, the visual and calorimetric appearance of samples was evaluated using a UV-Visible spectrophotometer before and after AgNP formulation at various time intervals. Prior to the manufacture of AgNPs, silver nitrate is colorless, whereas herbal extract has a distinct hue. After interacting with herbal extract, AgNPs produced silver nitrate solution develops a yellowish brown color, as validated by surface Plasmon resonance SPR and UV visible absorption in the 400-475 nm range (Kumar et al., 2014).

**FTIR analysis**

An investigative tool for identifying or conforming functional groups present in the moiety that are distinctive to that molecule is FTIR spectroscopy. By scanning the samples between 4000 and 400 cm<sup>-1</sup>, the main functional moieties found in AgNPs and herbal extract were identified as stated by Singh and colleagues (2013).

**SEM/TEM examination**

The primary application of transmission electron microscopy and scanning electron microscopy is the investigation of the surface morphology of produced AgNPs. Silver nitrate was added to SEM/TEM plates in order to create a solution smear on slides. By creating a thin layer of platinum and coating slides in it, conductivity was added to the system. When the slides were prepared, they were scanned at an accelerating voltage of 20 KV, producing high-quality images.(Kathireswari and others, 2014).

**X-ray diffraction (XRD) examination**

X-ray diffraction is a contemporary technique used to determine whether materials is crystalline or amorphous at different radiation angles. X-ray diffraction identifies crystalline/amorphous phases as well as cell dimension (Baba et al., 2014).

**Energy-dispersive X-ray spectroscopy (EDX)**

EDX is a useful technique for determining the elemental makeup of a material, and its use in nanotechnology has been described. All elements have different atomic structures, resulting in a distinct set of peaks in the X-ray spectrum (Jafarizad et al., 2015), which can be used to investigate the elemental composition of any nanoparticle.

**Dynamic Light Scattering and Zeta Potential**

The dynamic size distribution and surface charge of the produced AgNPs were measured using dynamic light scattering (DLS) and zeta potential, respectively. The AgNP solution was diluted with distilled water as previously described (Vijayabharathi et al., 2018), and 1 mL of the sample was placed in a cuvette for analysis. For each sample, the autocorrelation functions consist of an average of six 10-second runs.

### Factors Influencing AgNP Synthesis

The reaction temperature, metal ion concentration, extract contents, pH of the reaction mixture, reaction duration, and agitation are the primary physical and chemical parameters that influence AgNP synthesis. Metal ion concentration, extract composition, and reaction time all have a substantial impact on the size, shape, and morphology of the AgNPs (Kora et al., 2010).

Table 2 Factors affecting biological synthesis of metal nanoparticles(Zhang et al., 2020)

S. No	Factors	Influence on biological synthesis of metal nano particles	References
	pH	Size and shape of the synthesized nanoparticle	Dubey et al., 2010
	Reaction time	Size and shape of the synthesized nanoparticle	Krzton-Maziopa et al.,2011
	Reaction concentration	Shape of the synthesized nanoparticles	CHandran et al., 2006
	Reaction temperature	Size, shape, yield and stability of the synthesized nanopartic	Song et al., 2009a

### Methods of silver NPs synthesis

#### Physical synthesis

Physical synthesis of silver NPs has a few drawbacks, including the need for a large amount of space, the use of a large amount of energy while raising the ecological temperature around the source material, the need for a long period of time to achieve thermal stability, the consumption of more than a few kilowatts, and a preheating time of a few several minutes to achieve a stable working temperature (Jung et al., 2006).

#### Chemical synthesis

The most common method for creating stable colloidal dispersions of AgNPs in water or organic solvents is chemical reduction. The most commonly used reductant is citrate. Silver is reduced in aqueous solution, resulting in colloidal silver ions of nanoscale. The stability of any colloidal dispersion is crucial, which can be achieved by covering the surface with a stabilizing agent (dodecanethiol). It has the ability to prevent system aggregation and crystal formation. Small changes in parameters (Polymers) during the synthesis of AgNPs produce dramatic differences in size, shape, morphology, polydispersibility index, self-assembling, and zeta potential (Stability).Sunil and colleagues (2021).

The glycol derivatives polyvinyl pyrrolidone (PVP) and polyethylene glycol (PEG) are often used in the synthesis of AgNPs and AuNPs. Polyacrylamide is used in the synthesis of AuNPs as both a reducing and a stabilizing agent (Abou et al., 2010). Surfactants containing functional groups, such as amines, thiois, and acids, are critical for colloidal dispersion stability because they prevent crystal formation, coalesces, and agglomeration. Currently, the modified tollens approach produces AuNPs using saccharides, silver hydrosols, and a reducing agent, generating AgNPs in the 50-200 nm and 20-50 nm ranges, respectively (Yin et al., 2002).

### Biological Synthesis

The biological methods used to create nanoparticles are known as the "Bottom Up" approach. This occurs as a result of the oxidation and reduction of atoms or molecules, which are considered tiny entities. Biological approaches are used to generate nanoparticles from various plant extracts to microorganisms (Srivastava et al., 2021). Biological synthesis is regarded as an environmentally benign and cost-effective method of producing nanoparticles. Nanoparticles are made from plant elements such as the root, fruit, stem, seed, and leaf. The precise method of plant-based nanoparticle formation remains uncertain (unknown).

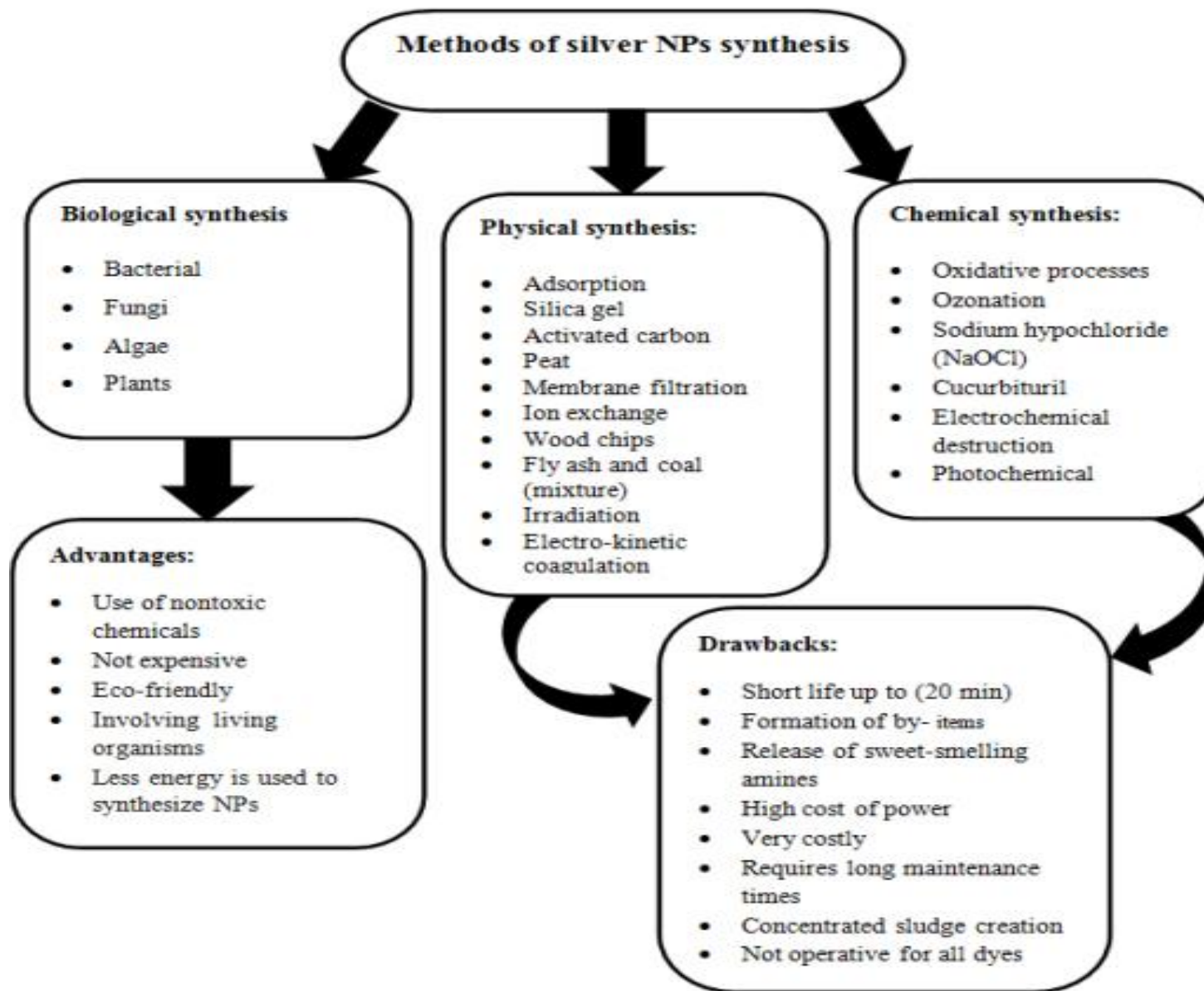


Figure 1: Different methods of silver nanoparticles and its advantages and drawbacks(Iravani et al., 2014)

### Applications of green synthesized titanium nanoparticles

Unique properties of TiO<sub>2</sub>NPs give a gateway to its applications in different fields such as the cosmetic industry, agriculture, pharmaceutical industry and environment. Different applications of TiO<sub>2</sub>NP with different methods and microbes are tabulated below in Table 3.

Table 3: Application of titanium dioxide nanoparticles.

Plant	Part used	Microbes used	Method	Application	Reference
T. cordifolia	stem	E. faecalis and E. coli	Disk diffusion method	Antibacterial Activity	(HarpreetKaur et al.2019)
Citrus limon	leaf	Escherichia coli and Pseudomonas aeruginosa	Disk diffusion method	Antibacterial Activity	(Falook et al.2017)
Sonchusasper	leaf	Staphylococcus aureus, Klebsiella pneumonia and Escherichia coli	Agar well diffusion method	Antimicrobial activity	Babu et al. 2019
Salvia Spinosa	Leaf	-	-	Treatment of pediatric acute leukemia	Xiaoshang et al. 2021
Jasminumofficinale	Flower	Staphylococcus aureus, Klebsiella pneumonia, E-coli	Agar diffusion method	Antibacterial activity	(Aravind et al. 2021)
Sonchusasper	Leaf	Staphylococcus aureus, Klebsiella pneumonia and Escherichia coli	Agar well diffusion method	Antimicrobial activity	Babu et al 2019
CynodonDactylon	Leaf	Escherichia coli	Disk diffusion method (ZOI)	Antimicrobial activity, Cell viability assay (anticancer activity against A549 cell lines)	Hariharan et al. 2017
Curcuma longa	Seed	F. graminearum, T. aestivum(Al Rasheed variety and Tamuze-2 variety)	-	Antifungal and anti-pathogenic activity	Abd et al.(2016)
Azadirachtaindica	Leaf	Bacillus subtilis, Escherichia coli, Klebsiellapneumoniae and Salmonella typhi	Disk-diffusion method	Antibacterial Activity	Thakur et al. (2019)
Kniphofiafoliosa	Root	Staphylococcus aureus, Escherichia coli	Disk diffusion method	Antibacterial Activity	Bekele et al 2020

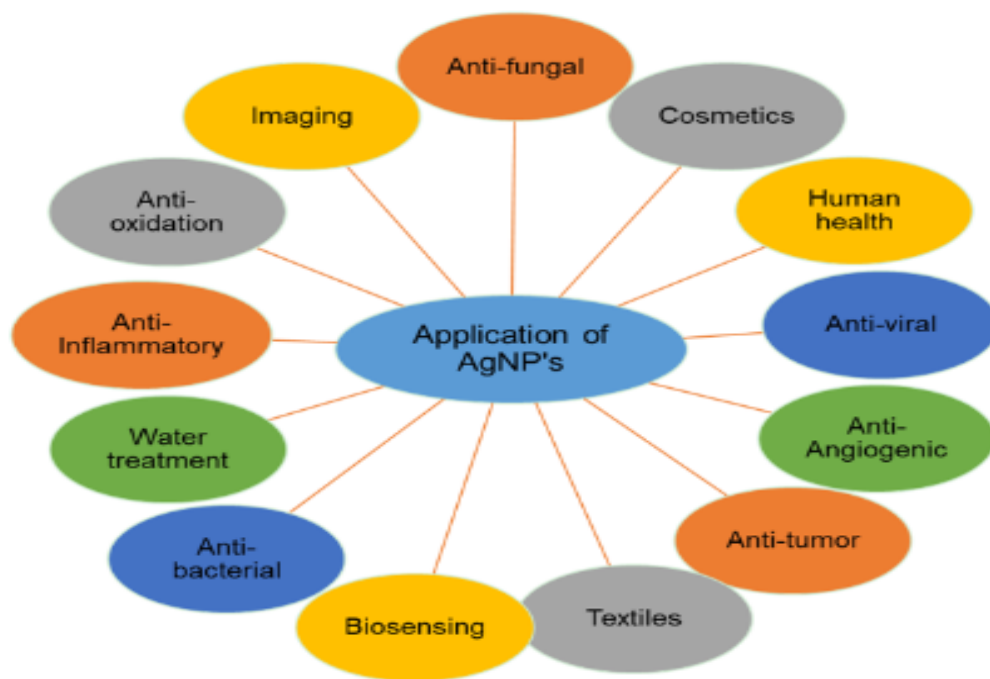


Figure 2: Applications of silver nanoparticles. (Galatage, et al., 2021)

### Cancer Treatment

The silver nanoparticles synthesized by Kuppusamy et al. using *Commeliana nudiflora* L aqueous extract showed a reduced cell viability and increased cytotoxicity against HCT-116 colon cancer cells. Silver nanoparticles composites possessed promising anticancer activity against the A549 (Human lung carcinoma), Hela (Human cervical adenocarcinoma), MCF7 (Human breast adenocarcinoma), MDAMB231 (Human breast adenocarcinoma), and SKBR3 (Human breast adenocarcinoma) cells (El-Naggaret al., 2017).

**Antimicrobial activity:** Silver nanoparticle is reported with antimicrobial activities which is used in disinfectants (Brady et al., 2003). Marslin et al. biosynthesized cream formulation of silver nanoparticle using *Withaniasomnifera* extract and concluded that cream had higher antimicrobial activity, hence might be utilized in low doses and fewer toxic for patient in comparison with AgNO<sub>3</sub> counterparts (Marslin et al., 2015)

**Optical Applications:** Silver nanoparticles can be used to efficiently harvest light and for enhanced optical spectroscopies including metal-enhanced fluorescence (MEF) and surface enhanced Raman scattering (SERS)

**Antifungal Activity:** People with weakened immune systems are more susceptible to fungus infections. This procedure has been shown to be particularly time demanding in nature when attempting to cure fungus-related diseases. The number of antiviral drugs on the market is severely limited (Kim et al., 2008). Antiviral drugs must be non-toxic, biocompatible, and ecologically friendly. Several fungi-caused diseases have been identified to be particularly vulnerable to AgNPs (Gurunathan et al., 2015). *Bipolaris sorokiniana* was effectively combated by biologically produced AgNPs by suppressing conidial germination. Indoor fungal species suppressed by AgNPs include *Penicillium brevicompactum*, *Aspergillus fumigatus*, *Cladosporium cladosporioides*, *Chaetomium globosum*, *Stachybotrys chartarum*, and *Mortierella alpine* cultivated on agar media.

### **Antimicrobial Applications**

One of the most promising nanomaterials for commercialization is silver nanoparticles. Silver nanoparticles have been used as antibacterial agents in a wide range of applications, including cleaning medical devices and household appliances and water purification. AgNPs have the potential to be used in a variety of applications, including cosmetics, food preservation, textiles, and medical equipment. Free silver ions are created at a slower rate with a bigger surface area in antibacterial potential AgNPs, providing a noxious environment, which is the primary rationale for AgNPs' broad spectrum antibacterial potential (Zhang et al., 2018).

### **Conclusions**

Recent technological breakthroughs have resulted in numerous developments in strategies for the biogenesis of NPs in a much more dependable manner. We have highlighted the numerous plant sources that have been employed for the biosynthesis of TiO<sub>2</sub>NPs in this brief review. TiO<sub>2</sub>NPs generated from these plant sources have broader applications and are more dependable due to their lower toxicity. Identification of secondary metabolites and chemical components is required for further advancements in TiO<sub>2</sub>NP production. Each portion of the plant has a wide range of aesthetic values that can be applied to a variety of purposes. TiO<sub>2</sub>NP is well-known in the cosmetic sector for its use in the manufacture of sunscreens. In conclusion, whereas TiO<sub>2</sub>NPs have been widely studied in recent years, much remains to be learned about their potential toxicological consequences to enable risk assessment and management for their application. Various more plants must be explored for the biosynthesis of TiO<sub>2</sub>NPs in order to make future progress in the improvement of TiO<sub>2</sub>NPs. These AgNPs can be produced in three ways: physically, chemically, and biologically. The biological technique is the most popular of the three because it is non-toxic and environmentally benign. It has been determined to be safe and site-specific in the case of cancer treatment. As a result, we can infer that these AgNPs are effective, straightforward, and safe therapies for a wide range of illnesses. AgNPs have a significant potential for usage as drug carriers in cancer therapy, as biosensors for metabolites and pollutants, as catalysts, and so on. Harnessing this potential will require extensive and coordinated study.

### **Future Perspectives**

- Nanotechnology is highly suggested for future views since it replaces hazardous solvents in green synthesis and process techniques, enhances catalytic efficiency and selectivity, is cost-effective, and generates less toxic waste.
- The key benefits of greener techniques include cheap cost and the use of antimicrobial nanoparticle combinations, which allows for the utilization of indigenous plant extracts without the need of toxic chemical reducing agents, as well as new uses such as antibacterial bandages.
- Future challenges and existing achievements related to green perspectives for nanomaterial manufacturing must be addressed, extending laboratory-based compliance to a feasible industrial level while taking current/past issues in terms of health and environmental ramifications into account.
- However, a greener approach strategy based on bio-derived materials or nanomaterials is necessary and will be widely used in the field of environmental remediation as well as other broad fields such as the food, cosmetics, and pharmaceutical industries.

- Furthermore, biomaterials derived from marine plants and algae found in specific locations have yet to be discovered. As a result, there are still several potential for the development of novel green route solutions based on biogenic synthesis.
- To enable the industrial production of such green nanomaterials, a great deal of scientific research is required. The eventual release of such nanomaterials into the environment might cause odd behavior, and this is a concern that must be investigated further.

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