

Synthesis and Characterization of Silver Nanoparticles in Solution via Microwave-Assisted Green Method

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Abstract: Metallic nanoparticles lies in the segment of nanotechnology, compete with the manipulating matter on the scale of one billionth of a meter. Microwave-assisted green synthesis have a great benefit in rapid synthesis and homogenous heating and emerged as eco-friendly, economical and environmental safer alternative relying on intrinsic reducing and stabilizing agents such as plant phytochemicals, bacterial metabolites, proteins, and polysaccharides, but conventional synthesis approach often involve harmful-toxic reagents, harsh solvents, and high energy intake, limiting sustainability and scalability. This method not only combines with green chemistry principles but also synthesized cost-effective and environment-friendly nanomaterials which have many applications in medicine, catalysis, and environmental remediation. In this paper we were discuss about the formation of the silver nanostructures with the average size of 40nm, with the help of Microwave-assisted incorporated with the green synthesis using Giloy (*Tinospora cordifolia*) stem's extract (ark), rich in phytochemicals like alkaloids, flavonoids, proteins, tannins, and phenolic compounds. *T. cordifolia* stems extract works as both reducing, capping and stabilizing agents, converting Ag^+ ions into stable nanoparticles. Microwave parameters used for this synthesis are 900 Watts power, 120 sec. time and 230°C temperature. Synthesis of silver nanoparticles was confirmed by colour changes from yellowish green to brown colour and characteristic SPR (Surface Plasmon Resonance) peak at 420 nm in the UV-Vis spectrum. SEM and EDS analysis analyse respectively size and purity of the synthesized nanoparticles was concluded.

Keywords: Microwave-Assisted, *T. Cordifolia*, Metallic- Nanoparticles.

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I. INTRODUCTION

Metallic nanoparticles (MNPs) – like silver, gold, platinum, copper, and nickel – these are among the most widely studied nanostructures due to of their unique properties. The reason for this lies in their ability to exhibit behaviors that differ significantly from those of their bulk counterparts [1]. For example, metallic nanoparticles are capable of exhibiting surface plasmon resonance, displaying an enormous surface-to-volume ratio, and altering their physical and chemical behavior based upon changes in particle size [2]. As a result, MNPs are being researched for potential applications including microbial eradication, anti-cancer therapy, enhancement of chemical reaction rates, degradation of pollutants, and development of advanced sensors [3].

The primary method used for producing metallic nanoparticles has been through the application of various fabrication techniques including chemical reduction, thermal

decomposition, sol-gel processing, and physical vapor deposition [1]. Although these methods provide a great deal of control over the morphology and structure of individual particles, they also produce several significant disadvantages. These include the necessity for the application of harmful chemicals (e.g., sodium borohydride, hydrazine, or formaldehyde), the need to utilize environmentally hazardous organic solvents, and the requirement of high temperatures and/or pressures. Therefore, many of these methods are not particularly suitable for producing metallic nanoparticles for biomedical and environmental applications. Silver nanoparticles are a good example of this [3]. When produced using conventional methods on a large-scale basis, they create toxic by-product and generate hazardous waste. Thus, the creation of silver nanoparticles and other MNPs through traditional methods may be limited in their use for biomedical and environmental applications [4].

Green synthesis represents an increasingly attractive alternative to traditional methods of synthesizing metallic nanoparticles. Green synthesis represents a cost-effective, energy-efficient and environmentally friendly method for producing materials utilizing natural resources for reducing and stabilizing material. Examples of green synthesis include aqueous-based green synthesis. In aqueous-based green synthesis, water serves as the solvent thereby eliminating the need for potentially toxic organic solvents [2]. Plant phytochemicals, microbial metabolites, proteins, and polysaccharides can contribute to the conversion of metal ions into zero-valent metals and the stabilization of nanoparticles, preventing them from aggregating. Overall, the bio-inspired nature of green synthesis provides an innovative approach for the production of nanomaterials that aligns well with the 12 principles of green chemistry [5].

The stem of *T. cordifolia* has been found to be an excellent source for synthesizing nanoparticles in plant-based materials. Giloy is an herbal plant, well known for their medicinal properties and applications. The plant has many medicinal properties, such as anti-inflammatory activity, antioxidant activity, antimicrobial activity, and anticancer activities. The phytochemicals present in the Giloy plant are flavonoids, tannins, phenolics, glycosides, and alkaloids. The phytochemicals are found to use as reducing, capping and stabilizing reagents [6]. The phytochemicals are capable of reducing metal ions such as Ag^+ , Au^{3+} , or Ni^{2+} into nanoparticles while also providing biofunctional properties to the nanoparticles. The recent publications have shown that the nanoparticles synthesized using the stem of *T. cordifolia* have improved antimicrobial and antioxidant properties. [7].

A very promising development in this regard is the incorporation of microwave-assisted approach, green chemistry and *T. cordifolia* stems extracts. Microwave-assisted green chemistry is a very efficient method for the synthesis of nanoparticles. Microwave fields can rapidly and uniformly heat reaction mixtures in a very energy-efficient manner. Reaction times can be reduced from hours to minutes. The microwave fields can accelerate the reaction between phytochemicals and metal precursors. The uniform nucleation and growth of the nanoparticles can be achieved [5]. The synthesized nanoparticles can have a narrow distribution and high crystallinity. The microwave-assisted method can reduce solvent consumption and increase the reproducibility of the reaction. The microwave-assisted approach can be very attractive for the large-scale synthesis of nanoparticles. For example, silver nanoparticles synthesized by using *T. cordifolia* stems extracts and microwave irradiation can show high antimicrobial activities against bacteria and fungi. [1, 3].

II. PRINCIPLES OF GREEN SYNTHESIS IN SOLUTION

Green synthesis of metallic nanoparticles is associated with the 12 principles of green chemistry, which highlight safety, sustainability, and environmental accountability [5]. On the other hand conventional chemical methods depend

on toxic reducing agents and hazardous solvents, but in the green synthesis we used naturally derived biomolecules in aqueous solutions, so that have minimum ecological outcomes [2, 4]. In this study we were associated green chemistry with the microwave-assisted approach. In the solution-based green synthesis, three major components play critical role:

➤ *Reducing Agents –*

Plant phytochemicals and microbial metabolites like flavonoids, polyphenols, alkaloids, terpenoids, and proteins used like an eco-friendly reducing agents. These biomolecules successfully convert metal ions (M^{n+}) into their zero-valent metallic nanoparticle [5].

➤ *Stabilizing Agents –*

The same biomolecules with polysaccharides and proteins acts as capping and stabilizing reagents. They prevent nanoparticle aggregation, regulate surface charge, and control morphology, ensuring long-term stability and functionality [8, 9].

➤ *Solvent System –*

In the green synthesis Water is used as the universal solvent, that is replacing high risk organic solvents. Its abundance, safety, and compatibility with biological systems make it the most sustainable medium for large-scale nanoparticle production [3].

➤ *Plant-Mediated Green Synthesis:*

T. cordifolia stems are among plant constituent's extracts that are the most common reducing agent used in green synthesis because they contain a large variety of phytochemicals and are readily available. Phytochemicals include flavonoids, tannins, phenolics, and glycosides that serve as reducing and capping agents for metallic nanoparticles [4]. Biomolecules within *T. cordifolia* stems are highly efficient at reducing metal ions to form metallic nanoparticles and then capping their surfaces to increase their stability [10]. While the phytochemicals in *T. cordifolia* stems do more than simply reduce metal precursors to nanoparticles, they also determine the average size, particle's shape and surface chemistry of the resultant nanoparticles. Such bio-directed control of nanostructure is highly valuable for tailoring nanoparticles toward specific applications, ranging from antimicrobial therapies to catalytic and sensing technologies [11].

• *Preparation of Plant Extract:*

Fresh stems are collected from agriculture field of the boys' hostel of R. B. S. Engineering Technical Campus Bichpuri, Agra. Stems are rinse 3-4 times with the tap water and two times with distilled water for removal of the pollutants and dust particles from the stems. After that stems are cutting into 2-3 inches small pieces and dry in shade for 2-3 days. After two rest of the moisture in the stems is remove by dry them into hot air oven for 20 min at 80°C. Dried stems are converted into fine powder by crushed them into mortar & pestle and grinding mixer. 3g of powder pour into 100 ml or deionised water and boil for 30min at 90°C. Cool them and filter with whatman filter no. 1 and

centrifuge for 20 min at 3000rpm for residual extraction. Extract is Stored at 4°C for uses [12].

- **Synthesis of Silver Nanoparticles:**

To synthesize silver nanoparticles, a 5 mM solution of silver nitrate (AgNO_3) was first prepared and used as the metal precursor. From this stock solution, 30 ml was measured and poured into a 100 ml beaker. A constant volume (30 ml) of an extract from the Giloy stems was then added slowly into the beaker while maintaining a steady rate of adding one drop per minute by means of a burette with the constant speed of 500 rpm for the 30 minutes. Maintaining this steady dropwise addition will ensure adequate contact time for interaction to occur between the silver ions and the bioactive compounds within the extract in the slightly acidic medium. The reaction mixture was mechanically stirred during the course of the entire experiment at a consistent speed to provide uniform distribution of the reactants and to optimize the efficiency of reduction and stabilization [5, 8].

Following the completion of the addition of the extract, the reaction precursor was placed in a standard household microwave-oven for irradiation in order to increase the rate of the chemical reaction. The reaction mixture was subjected to microwave radiation with an energy output of 900 watts for the duration of 120 seconds as per previous studies. The microwave radiation provided sufficient thermal energy to rapidly heat the reaction mixture and enhanced the process of nucleation and rapid growth of the silver nanoparticles as the result of the increased rate of energy transfer [10]. Visual evidence of the formation of silver nanoparticles was evident upon completion of the microwave treatment. The starting colour of the reaction precursor solution changed from a yellowish colour to a dark brown colour, and finally became blackish after approximately one hour. These sequential colour changes are indicative of the formation of silver nanoparticles and can be seen in Figure 1. The observed colour changes are visible due to the surface plasmon resonance (SPR) exhibited by the formed silver nanostructures [7], and therefore confirm that the Giloy stem extract effectively behave as a reducing, capping and stabilizing reagent for the silver ions and enabled the production of stable silver nanoparticles.



Fig 1 Methodology of Silver Nanoparticles Synthesis

III. CHARACTERIZATION, RESULT AND DISCUSSION

The synthesis of silver nanostructures (AgNSs) via microwave-assisted approach using the extract of the stem of Giloy (*T. cordifolia*) has been achieved and the obtained

nano-materials have been characterized by Ultra Violet-Visible spectroscopy and energy dispersive X-ray (EDX) analysis [8].

➤ *UV-Vis Spectroscopy:*

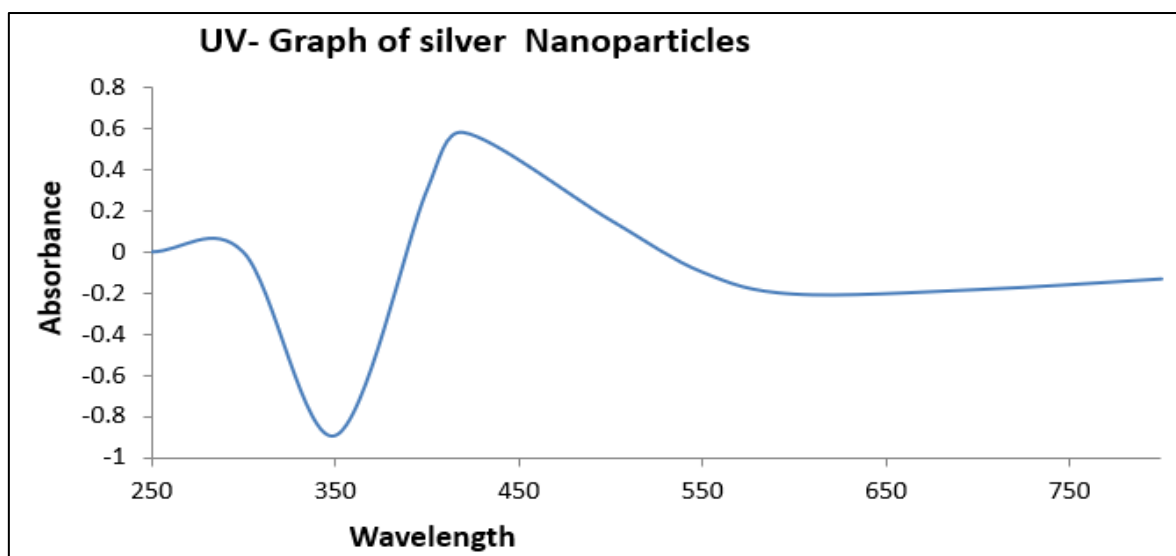


Fig 2 UV- Spectra of Synthesized TcAgNPs

UV-Vis absorption spectrum of the prepared AgNPs (figure 2), exhibited a prominent SPR peak at approximately 420 nm, which is a typical indicator of silver nanoparticles[2]. Sharp SPR peak shows that the silver ions were efficiently reduced by phytochemicals present in the *T. cordifolia* stems extract, like alkaloids, flavonoids, and polyphenols that acts like both reducing and stabilizing

agents [10]. Furthermore, SPR at 420 nm suggest that the formed nanostructures were nearly spherical in the shape and had an average diameter of approximately 40 nm, in accordance with other previous research studies on green synthesis [7].

➤ *Scanning Electron Microscopy (SEM) Analysis*

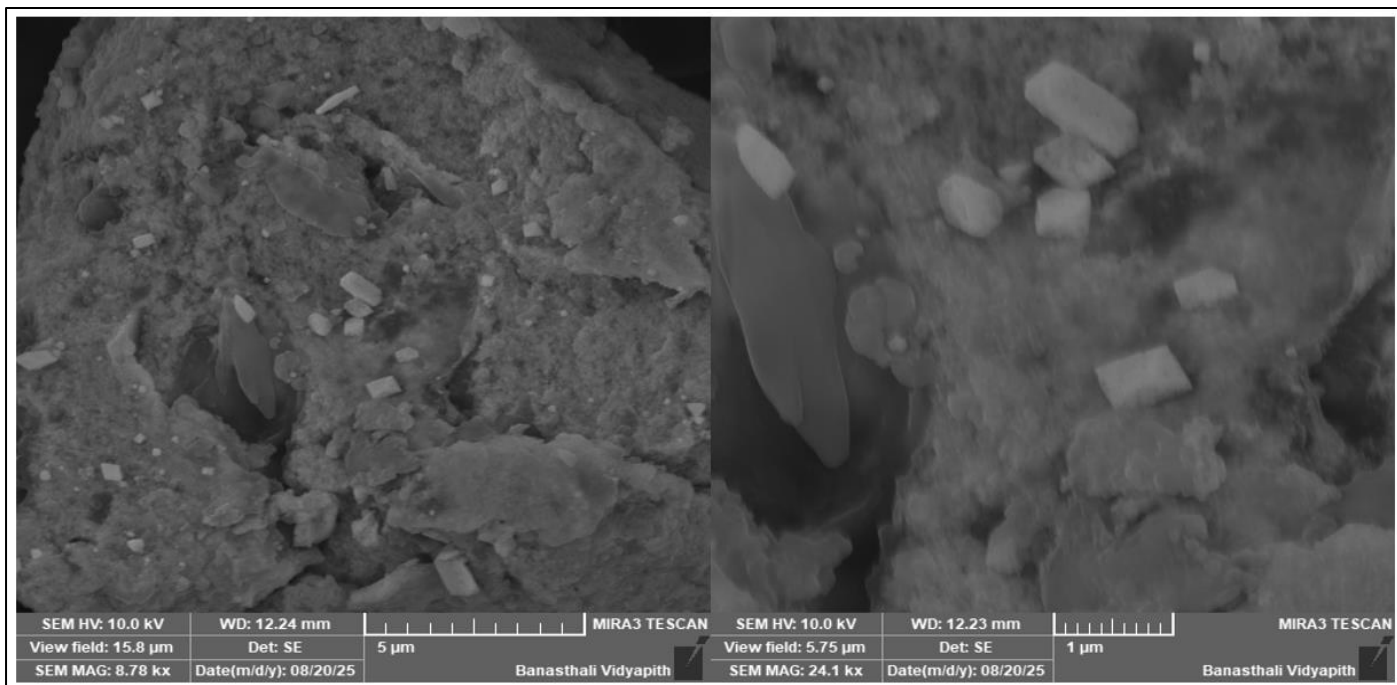


Fig 3 SEM Images of Synthesized TcAgNPs.

SEM image (figure 3) of the silver nanoparticles produced from the *T. cordifolia* stems extract under microwave irradiation clearly shows the formation of well-defined nanoparticles. Particles show a primarily spherical morphology and were found to be evenly dispersed without any significant aggregation, further indicating the

phytochemicals presence in the plant extracts effectively stabilized the particles. Average particle diameter was estimated to be approximately 40 nm, which is similar to what was determined by UV-Vis analysis (SPR peak at 420 nm) [7]. Narrow size distribution of the particles, signifies that the microwave-assisted synthesis produces

homogeneous nanoparticles compared to traditional methods. The fact that the particles exhibit a smooth morphology and a nanoscale size demonstrates that the bioactive compounds from the *T. cordifolia* stems functioned not only as reducing agents but as capping agents to prevent aggregation of the particles. Spherical AgNPs

with a diameter of ~40 nm show improved surface activity, antimicrobial efficacy and plasmon behavior compared to larger AgNPs [13].

➤ *EDX (Energy Dispersive) Analysis*

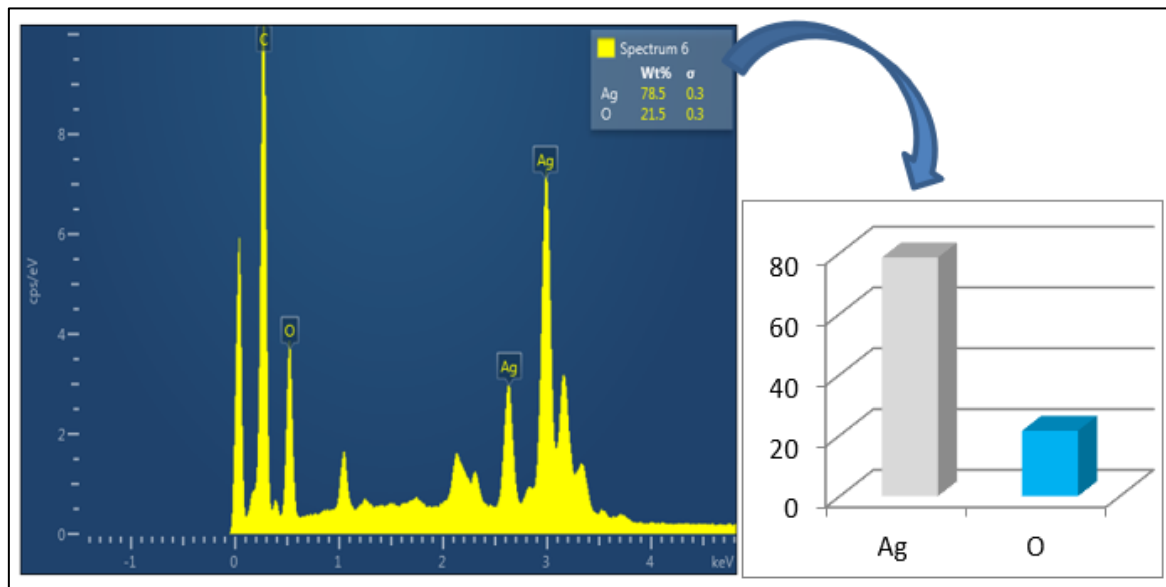


Fig 4 EDX Image of Synthesized TcAgNPs.

The EDX spectrum (figure 4), provided an insight into the chemical composition of the synthesized nanoparticles. A strong and high peak of Ag was found at approximately 3 keV, this is the energy needed to break up the silver atoms and thus indicates that the Ag nanoparticles were successfully formed [4]. Along with the large peaks for Ag, smaller peaks for C and O were also seen on the spectrum. C and O are often linked to phytochemicals derived from plants, thus it appears that the compounds present in the Giloy stems extract participated in capping, stabilizing and

inhibiting aggregation of the nanoparticles during the synthesis. Presence of C and O combined with lack of any notable impurities in the spectrum demonstrate that the biosynthesized nanoparticles have very high purity. In summary, Energy Dispersive X-ray analysis confirmed both the composition of elemental and success of the green synthesis route employed to produce stable silver nanoparticles [14].

➤ *Mechanism of Microwave-Assisted Green Synthesis*



Fig 5 Schematic Diagram of Microwave Mechanism

The microwave radiation rapidly accelerated the reduction process (in figure 5) through the generation of uniform heat and rapid chemical reaction kinetics, resulting in the formation of nanoparticles that are significantly smaller and are more evenly distributed than those generated by conventional methods[8]. Phytochemicals present in the *T. cordifolia* stems extract donated electrons to convert Ag^+ into Ag, while concurrently adsorbed onto the surface of the nanoparticles to inhibit agglomeration [15]. The dual roles of phytochemicals (stabilization and reduction) help to understand why the formed AgNPs exhibit both a high level of uniformity and good stability. In comparison with other methods of producing AgNPs by means of thermal treatment, the rapid, environmentally friendly, and reproducible production method using *T. cordifolia* stem extracts as reducing agents under microwave radiation

allows the formation of stable and relatively small AgNPs (approximately 40 nm), which have a potential use in the fields of biomedical, antimicrobial and catalytic applications [2, 16].

IV. APPLICATIONS

Metallic nanoparticles have opened up a vast variety of applications in all types of disciplines because they can be compatible with biological systems, function on their surfaces and can be tailored to various physical and chemical characteristics [2]. Metallic nanoparticles have opened up a vast variety of applications in all types of disciplines because they can be compatible with biological systems, function on their surfaces and can be tailored to various physical and chemical characteristics [16].

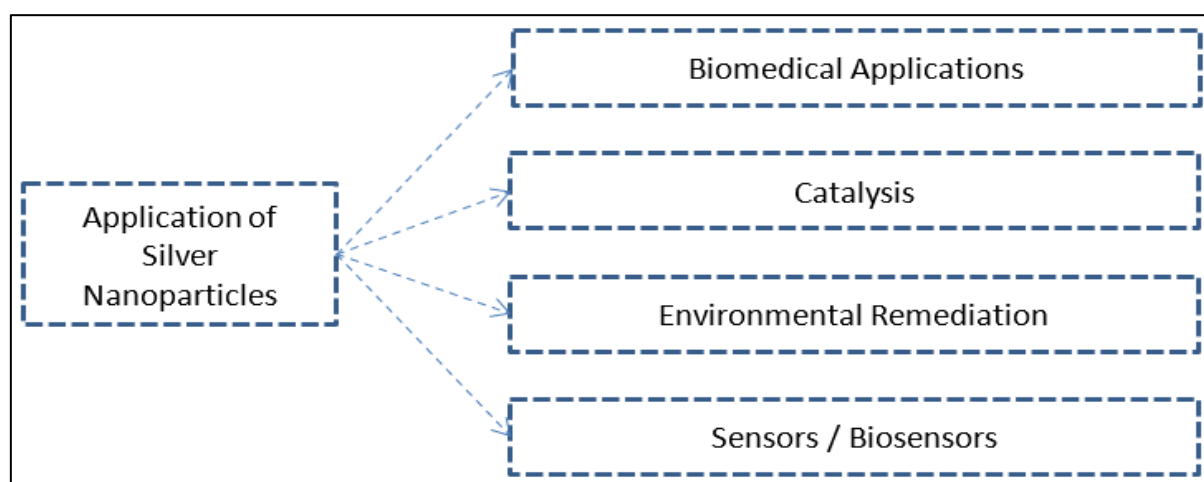


Fig 6 Schematic diagram of Application Synthesized of TcAgNPs.

➤ *Bio-Medical Applications:*

Biomedical applications of synthesized nanostructures include their inherent biocompatibility, and the surface functionalization with phytochemicals that allow for compatibility. In addition to their biocompatibility and surface functionalization with phytochemicals, the antimicrobial activity of these nanoparticles has demonstrated efficacy against a wide range of microbial pathogens; thus, offering potential alternatives to conventional antibiotics for the treatment of infections happened by multi-drug resistant bacteria. They have also shown great promise as anticancer agents in cancer therapy, through the induction of oxidative stress, and through their ability to target cytotoxicity [7]. Moreover, their ability to attach to other molecules (such as drugs) enables them to be used as effective drug delivery mechanism, enabling controlled discharge and reduction of side effects [11]. Finally, synthesized nanostructures have also been gaining attention for their use in biosensing and bioimaging, where their optical properties enable greater sensitivity and specificity than traditional methods [9, 15].

➤ *Catalysis:*

Metallic particles have proven to be very effective as catalysts due of their high ratio of surface area to volume and their number of active surface sites. Metallic particles

are able to reduce many of the most common industrial pollutants (nitroaromatics) into less harmful by-products. In addition, metallic particles can be used for the photodegradation of dyes in wastewater making it a sustainable method of water purification [2]. Finally, metallic particles will likely play an important role in future renewable energy systems through their ability to facilitate chemical energy conversions (i.e., hydrogen production from water electrolysis, fuel cells etc.) [14].

➤ *Environmental Remediation:*

Nanoparticles synthesized from natural sources have been gaining increasing interest as a material to remove heavy metals using adsorption and redox processes[4]. In addition to heavy metal removal, they can be used to degrade organic dyes under UV or catalytically active conditions. Their application in water purification systems has demonstrated potential for large-scale cleanup of contaminated sites [11].

➤ *Sensors:*

Due to their strong surface plasmon resonance, these nanoparticles are excellent platforms for surface-enhanced Raman scattering (SERS), enabling ultrasensitive molecular detection[3]. In addition, their electrochemical properties

support the development of next-generation sensors for environmental monitoring and medical diagnostics [15].

V. CHALLENGES AND FUTURE DIRECTIONS

Although a wide variety of advancements have occurred with respect to “green” methods for synthesizing metal nanoparticle (MNP), several significant issues remain prior to the industrialization of MNP. One primary challenge associated with the usage of plant extracts for making MNPs is the uncontrolled nature of the process. Plant extracts contain numerous biomolecules that act as reducing, capping and stabilizing reagents during nanoparticle synthesis [12]. The exact impact of these biomolecules on the resulting MNP product is unknown. Consequently, it is highly difficult to synthesize uniform-sized MNPs using a plant extract-mediated process. Uniformity in size will directly affect the optical, catalytic and biological characteristics of the MNPs [5].

Another major problem is the variable nature of natural extracts. The variability of the phytochemical composition of the extracts results from the influence of a variety of factors including geographic location, harvest time, extraction procedure, and storage conditions. As a result of this variation, it is often difficult to reproduce the same types of particles in the same quantities using plant extracts. Furthermore, scaling up the synthesis of nanoparticles by the help of plant extracts is also a problem [9]. Despite the fact that small-scale laboratory tests are possible when employing plant extracts to create nanoparticles, problems associated with particle agglomeration as well as/and reduced surface activity as well as/and loss of function may occur when transitioning to large-scale industrial production. These problems limit the potential for use of biologically synthesized green nanoparticles in medical and catalytic applications [1].

Therefore, the next step in research should be to develop methods to standardize the preparation of plant extracts and reaction conditions so that less variability exists and greater reproducibility is achieved. A further strategy could be to integrate the green synthesis of nanoparticles into continuous flow microreactors. Continuous flow microreactors allow a kinetic control over the reaction rates and thus enable the manufacture of highly pure products at significantly lower waste levels than batch reactors [11]. Furthermore, scientists should work on developing protocols to synthesize multimetallic and hybrid nanocomposite materials, which possess improved functional properties in comparison to single metal nanoparticles. The overcoming of these obstacles will contribute toward the realization of green nanoparticle synthesis in an industrial setting, which has been up to now only established in a laboratory-based environment [13].

VI. CONCLUSION

The development of metallic nanoparticles in solution through green synthesis methods has become an area of research that can potentially align nanotechnology with the

green chemistry principles and sustainable development. These nanomaterials have been shown to have unique characteristics like tunable size, shape and surface area to volume ratio, surface functionality that provide value added uses for these materials in the field of biomedical applications, catalysis, sensing and environmental remediation. Silver nanoparticles synthesized from Giloy (*T. cordifolia*) stems extract exhibited a well-defined SPR peak at 420 nm and have an average particle size of approximately 40 nm indicating that they are both uniform and stable. EDS analysis shown the purity of the synthesized silver nanoparticles with 78.5 % elemental detection and Oxygen are present with 21.5% in the sample. While the green synthesis of metallic nanostructures offers many opportunities to advance nanotechnology, there remain several major challenges that need to be overcome before this technology can become widespread. Challenges such as reproducibility of the process, variability of the natural extracts and scale up of the process are all still to be resolved. A number of initiatives are underway that may provide some solutions to these challenges. Advances in the standardization of plant extracts, enhanced understanding of the appliances of action and integration of the green synthesis of metallic nanoparticles into microwave-assisted processes may ultimately lead to the development of commercially viable, scalable, sustainable and functional nanomaterials.

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