

Chapter 29

Synthesis Techniques and Applications of Diverse Nanoparticles

Muhammad Adil¹, Farrah Deebea¹, Bushra Mumtaz², Marwa Abbas², Sayeda Nimra Jabeen^{3*}, Hassan Raza⁴, Rabia Ali², Saba Imtiaz⁴, Saifuddin² and Maham Naseer⁵

¹Department of Clinical Medicine & Surgery, University of Agriculture, Faisalabad, Pakistan

²Institute of Microbiology, Faculty of Veterinary Science, University of Agriculture, Faisalabad, Pakistan

³Department of Chemistry, Government College Women University, Faisalabad, Pakistan

⁴Department of Chemistry, University of Agriculture, Faisalabad, Pakistan

⁵ National Institute of Food Science & Technology, University of Agriculture, Faisalabad, Pakistan

*Corresponding author: syedanimrajabeen@gmail.com

ABSTRACT

Nanotechnology involves the creation of nanomaterials, including nanoparticles, which exhibit unique properties because of their nanoscale size (1-100nm). These particles differ significantly from bulk materials in their chemical, optical, physical, and electrical characteristics, primarily because of their large surface area-to-volume ratio and quantum mechanical effects. Nanoparticles can be synthesized using various techniques, including chemical methods (sol-gel, hydrothermal, solvothermal, vapor synthesis), biological methods (microbial, plant-based synthesis), mechanical methods (milling, mechanical alloying), and both top-down and bottom-up approaches. The characterization of nanoparticles deals with determining their morphology, structure optics, and physiochemical properties by means such as electron microscopy, X-ray diffraction spectroscopy (XRD) BET(Brunner-Emmet-Teller) surface area analysis to calculate particle size, shape crystalline structure, and elemental composition. From cancer therapy to drug delivery, environmental cleanup, and even energy devices, nanoparticles have a wide scope of uses because of their unique characteristics and ability to be manipulated. Nanoparticles also have some benefits and threats to health and the environment, which can be overcome by using the techniques of green synthesis that include non-toxic eco-friendly reagents. Nanoparticles are classified, synthesized, and characterized and their application in various fields along with prospects have been included in this chapter.

KEYWORDS

Nanoparticle, Microscopic, Biological, Chemical, Mechanical, Metallic

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INTRODUCTION

Nanotechnology is the creation and formation of nanomaterials that are functionalized in less than or equal to 100nm range (Adil et al., 2023). Because of their small size, between one and a hundred nanometers (nm) in diameter, these particles have unique chemical, optical, physical, and electrical properties (Bhattacharjee and Bose, 2021). Nanoparticles themselves are synthesized and developed by nanotechnology. Nanoparticles mainly consist of a core that is encapsulated by several layers of nanomaterials and, as a result, shell form with an exterior surface that is commonly functionalized (Bolokang et al., 2015). Nanoparticles are substantially different from bulk materials in terms of their surface-area-to-volume ratio, interfacial layer, solubility into the solvent phase, type of coating quantum mechanical effects, diffusion rate, and last modifiers such as metal ions (Bhoi et al., 2016). Surfactants act as modifiers for nanoparticle properties for all kinds of applications (Fanhoefer et al., 2004).

Synthesis Techniques of Nanoparticles

Chemical Methods

Nanoparticles can be synthesized through different chemical methods, such as sol-gel, precipitation, hydrothermal, thermal breakdown, solvothermal, and vapor synthesis methods (Rane et al., 2018). The sol-gel technique is very fundamental in creating nanostructures. Here, the precursors are dissolved in a solvent to solve with characteristics like a gel. It is then heated to produce nanoparticles, and the solvent is also evaporated through gelation (Bokov et al., 2021). The wet chemical precipitation technique is still another method that one can use; it is quite fast and efficient. High

pressure and temperature, especially in aqueous solvents such as water, led to heterogeneous treatment known as hydrothermal treatment. The nanoparticle properties can therefore be controlled by the pressure, pH, and temperature that are used to synthesize the nanoparticles (Darr et al., 2017). These nanoparticles are hydrophilic and, therefore, appropriate for the biotechnological field. Thermal breakdown involves the heating of a solid material until it melts, that is the release of binding forces, the formation of molecules, and a gaseous effect. Solvothermal synthesis involves the utilization of solvents with medium to high pressure to create materials like metals, semiconductors, and polymers (Sasikala et al., 2017). The solvothermal method comparatively works at controlled temperature, leading to the formation of stable nanoparticles. Different surfactant stabilizers enhance nanodot formation from cationic precursor kinds (Li et al., 2021). Zinc oxide, zinc selenide, and cadmium selenide obtained by this process are used in biotech and magnetic corporations (Balakrishnan and Kadam, 2021). In vapor synthesis, the gaseous state of the material carries out a chemical reaction and forms a phase that condenses to induce particle growth, and, in this phase, the temperature has a direct influence on the process. The methods of condensing inert gases, vaporizing supersaturated material by using a pulsed laser, sputtering by unreactive gaseous ions, spark discharge, and chemical methods including Chemical Vapor Deposition, Photothermal Method, Flame Synthesis, and Spray Pyrolysis are some ways which can cause homogeneous nucleation (Danielson et al., 2020). Titania, carbon, and silica nanoparticles can be produced using this method, although the process can be time-consuming. Flame synthesis is applied in the commercial synthesis of silica, carbon black, optical fibers, and titania. While it may be possible to obtain agglomerated particles, the ones derived from the conversion of gases in the furnace reactors or hot walls are rather clean (Zhao et al., 2024).

Biological Methods

Biological nanoparticle synthesis using microorganisms represents a clean, uncontaminated, and environmentally sustainable method (Samrot et al., 2021). This approach has produced various nanoparticles, particularly oxides that include ferrous, silver, nickel, copper, and zinc (Ezealigo et al., 2021). The synthesis site depends on whether the production occurs extracellularly or intracellularly (Chan et al., 2022). For intracellular nanoparticle synthesis, the ions are transported into microbial cells through the action of enzymes and the nanoparticles are synthesized within the cells (Rana et al., 2020). Extracellular fungi with large secretion glands located outside the cell wall are used in this method. Microorganisms such as bacteria and fungi are highly effective in the synthesis of nanomaterials due to their cost effectiveness, non-toxicity, and ability as scavengers. Plant-based nanoparticles suitable for biological systems make use of abundant and stable natural resources by excluding the use of toxic synthetic agents (George et al., 2021). The pH variations affect the geometric aspects of plants, binding capability, and the concentration of metal ions for biosynthesis. The sources, synthesis methods, and applications of nanoparticles are shown in Fig. 1 (Jamkhande et al., 2019). Generating nanoparticles by using Biogenic methods such as utilizing microorganisms and waste materials is both eco-friendly and cost-effective (Mughal et al., 2021).

Mechanical Methods

Mechanical techniques are used for making nanoparticles, such as mechanical alloying, milling, and mechanochemical processes (Yadav et al., 2012). When contacting for a chemical reaction, the milled sample should be improved, and this should especially apply to cases where the milling was done at low temperatures. Using continuous welding processes to help select milling materials and minimize occurrences of agglomerations here will again use mechanochemical methods. The source material stoichiometry, thermal treatment conditions, reaction mechanism, and milling parameters should be optimally controlled for high production. Mechanical techniques are used to make oxide, iron, nickel, silver, and cobalt nanoparticles (Iqbal et al., 2016).

Top-Down Approach

The top-down method of preparing nanoparticles comprises the fission of large particles and converting them into particles of the required size (Priyadarshana et al., 2016). It is detrimental and uses enhanced breakdown techniques like Physical Vapor Deposition (PVD), grinding, and Chemical Vapor Deposition (CVD) (Abid et al., 2022). Milling was also engrossed to extract the nanoparticles from coconut resemblance shells and the crystallite size was decreased with time. This process is used to synthesize nanoparticles of cobalt (III) oxide, carbon, iron oxide, and dichalcogenides (Harish et al., 2022).

Bottom-up Approach

The bottom-up approach involves the assemblage of nanoparticles from fundamental elements, and this procedure is slow. This technique is quite accurate, inexpensive, relatively safe for humans and the bio of insects, and safe for the environment (Modan and Plăișu, 2020). The methods comprise spin coat-ting, green synthesis, biochemical synthesis, sol-gel synthesis, and other reduction and sedimentation methods. Through this approach, titanium dioxide, gold, and bismuth nanoparticles are synthesized (Ambre et al., 2023).

Nanoparticle synthesis can also involve biological or chemical procedures. Chemical synthesis processes consist of microwave methods, wet chemical synthesis, sol-gel, hydrothermal methods, and thermal decomposition (Hachem et al., 2022). Conversely, biological methods utilize enzymes, microbes, plant extracts, and fungi.

Characterization Methods for Nanoparticles

Thorough characterization of nanoparticles is essential to determine their shape, size, surface morphology, crystalline nature, light absorption, and other properties (Cuenya, 2010). *Some of the methods used to characterize nanoparticles include:*

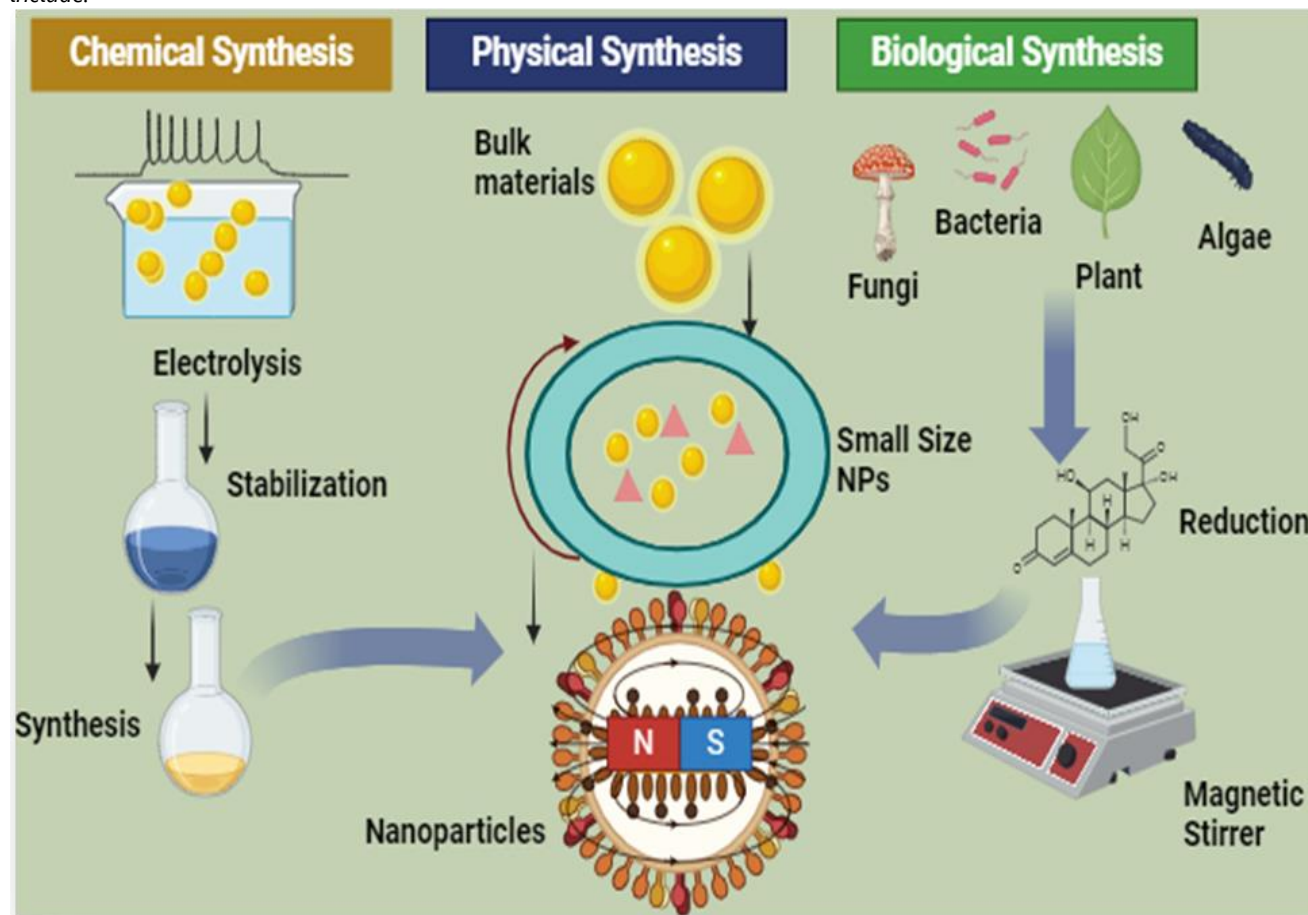


Fig. 1: Methods of Synthesis of Nanoparticles

Morphological Features

An important factor influencing the characteristics that nanoparticles exhibit is their shape. Particularly scanning probes or electron microscopy are used on nanoparticles (Ngoi et al., 2021). The dispersion and shape of nanoparticles can be studied at the nanoscale and surface using a scanning electron microscope (SEM) (Tang et al., 2020). Measurements of individual particles are made using destructive microscopy techniques. Utilizing electron transmittance, transmission electron microscopy (TEM) may obtain large amounts of data at both high and low magnifications (Zhang et al., 2020). Because nanoparticles are smaller than the limit of light diffraction, the optical microscopic approach does not apply to them. Combining electron microscopes and spectroscopic methods would be possible for elemental studies (Taghavi Fardood et al., 2020).

Optical Studies

Optical methods are innovative techniques used to characterize the photochemical, luminescence, transmittance, and reflectance characteristics of nanoparticles that unmask these properties. Spectroscopy is a method that appraises the density of nanoparticles and their size and shape by monitoring how they react to electromagnetic waves or light (Gudkov et al., 2023). Nanoparticle spectroscopies are more prominent as IR, UV-VIS, NIR, and PL DRS (photoluminescence) than MRI (Mmelesi et al., 2024). The band gap energy can be achieved by using the method of DRS (diffuse reflectance spectroscopy) and this could only be done via specialized, high-sensitivity instrumentation. PL studies show that the excitability of photons, half-life and charge results produced by recombination work differently due to emissivity and absorptivity. The size-mobilized optical properties and application aspects of nanoparticles incorporated into bioimaging tools (Wang et al., 2020).

Structural Analysis

In this technique, the nanoparticles are directly linked through the type of inter-atomic bond and this crystal structure

projects, onto a nanoscale level within bulk material so that distinctive qualities of bulk are shown. Through this technique characterization of nanoparticles like the composition and structure of the synthesized selenium nanoparticles was done by X-ray diffraction (XRD), infrared (IR) spectroscopy, Brunauer-Emmett-Teller (BET) surface area measurements, and other electron microscopy imaging techniques. Findings on the phases, size, and type of nanoparticles will be characterized by powdered crystal X-ray diffractograms (Khan et al., 2020).

Elemental Studies

The elemental composition of nanoparticle techniques for characterization are Energy dispersive X-ray (EDX), Raman Spectroscopy, and Fourier transform infrared (FT-IR) spectroscopy, are included. This technique provides information about the nanoparticles present in the powder form (Patil et al., 2022). The exact composition of elemental particles can be studied with their proper ratios. In this regard, X-ray photoelectron spectroscopy (XPS) is the most sensitive method for finding out all dimensional characterizations. The vibrational effect of Raman and FTIR, respectively, shows the functionalization of those peaks and provides particle information (Castro and Zuazo, 2024).

Size Estimation

Scanning Electron Microscopes, X-ray diffraction instruments, Transmission Electron Microscopy, Atomic Force Microscopes, and so on can be used to measure the sizes of the nanoparticles (Selvan et al., 2021). Sizing distribution curves are employed in the process of measuring the sizes of particles, and the value of such magnitudes becomes correct when they are merged with the digital models. Adsorption and desorption processes are the procedures making use of BET to characterize the surface area, which has been pointed out by (Hou et al., 2020).

Physicochemical Characteristics

The physicochemical features of nanoparticles that make them appropriate for industrial applications include their mechanical character flexibility and their optical activity to absorb sunlight. While lighting, electrons on the surface of nanoparticles become much free. Therefore, their motion is very active, and they can lose congregation (Kishore et al., 2023). Magnets are characterized by magnetism at the nanoscale because crystalline planes of the materials where NPs have a random orientation. This orientation is regulated by the synthesis method used. They are abundant in catalytic machines, biomedicine, and nucleotide resonance imaging (Roostae and Sheikhshoaie, 2020). The mechanical properties of nanoparticles, including stress, adhesiveness, friction, hardness, strain, and surface coatings, are crucial for understanding their behavior and significantly affect surface quality (Mehmood et al., 2023). Nanoparticles, especially those on their surfaces, demonstrate superior thermal conduction.

Application Areas of Nanoparticles

Because of their unique properties, nanoparticles are used in a variety of fields, including cancer treatments, vaccines, disease management, cancer detection, mechanical industries, electronics, optical devices, energy harvesting, manufacturing, cell imaging, and delivery systems (Aghebati-Maleki et al., 2020). They also play a role in environmental protection by helping to remove toxins from water during purification (Pooja et al., 2020).

The application areas of nanoparticles have been given in Table 1 (Singh et al., 2021).

Table 1: Application areas of some nanoparticles.

<i>Nanoparticles</i>	<i>Application areas</i>
Silver	Fitness centers, medicine, electronics, automotive, agriculture, food packaging, textile industries, clothing
Gold	Medicine, food, environmental products, cosmetics
Carbon nanotubes	Cosmetics, construction, medicine, electronic components, textiles, integrated circuits.
Titanium dioxide	Skin coatings, H ₂ O purifiers, paints
Zinc oxide	Food, cosmetics, agriculture, automotive, home appliances
Cerium oxide	Applying in biomedical equipment, electronic and energy devices
Nickel oxide	Gas sensing devices, water treatment and catalytic systems, supercapacitors, dye-sensitized solar cells, batteries
Iron (Fe ⁺⁺)	Applying in optics instruments, H ₂ O purifiers
Calcium (Ca ⁺⁺)	Applying in agronomy, automotive, and nutrition

Although nanoparticles (NPs) are extensively used, their introduction into the body through various pathways or their release into the environment can cause toxic and adverse effects. Furthermore, organic compounds can interact with nanoparticles, resulting in their agglomeration. Using green synthesis techniques can reduce the toxic affects of NP synthesis, particularly when creating nanoparticles of gold, silver, iron, and copper, among other metals (*Dikshit et al., 2021*). Green synthesis uses a variety of capping materials, including polysaccharides and biomolecules. Utilizing reagents such as sugars, polymers, vitamins, and plant extracts, green procedures are non-toxic, eco-friendly, and compatible with biological systems (*Ahmed et al., 2022*). Due to their low cost, simplicity, ease of replication, and high stability, plant-based extracts such as latex, leaves, seeds, roots, or stems are better suited for bioprocesses. By calculating the rates at which the

particles nucleate, models can be created to reduce the challenges related to dispersing the particle size and NP formation (Shrestha et al., 2020).

Classification of Nanoparticles

Nanoparticles can be categorized into artificial, engineered, metallic, non-metallic, organic, or inorganic types. Non-metallic nanoparticles encompass materials such as silica and carbon nanotubes, whereas nanoparticles that are metallic in nature include elements like copper, magnesium, zinc, gold, titanium, and silver (Khan et al., 2022). Anthropogenic nanoparticles arise as by-products from industrial processes, while engineered nanoparticles are specifically produced through manufacturing techniques (Barhoum et al., 2022). Table 2 provides a summary of various nanoparticles and their characteristics (Khan et al., 2019; Khan and Hossain, 2022).

Table 1: Nanoparticles and their implications

Nanoparticles	Features
Silver	Extremely potent, excellent antibacterial activity, wide use
Gold	Effective for diagnosing cancer and other microorganisms, good at detecting protein interactions, helpful in tracking fingerprints, and able to detect antibiotics and malignant cells.
Iron	Suitable for medication administration, gene analysis, cancer treatment, and stem cell sorting, biocompatible
Quantum dot	less than 10 nm in diameter, semiconducting, and size-dependent
Carbon nanotubes	Strong electron bonds, excellent electrical conductivity, sp ² hybridized carbon atoms, and effective catalysts.
Copper	Good-quality nanoparticles are produced by a broad absorption spectrum and unique optical characteristics.
Ceramics	Inorganic amorphous solids are widely used in photocatalysis, imaging devices, and other fields. They can be polycrystalline, porous, amorphous, or dense.
Semiconductor	Excellent for electronic equipment and water splitting due to its large and adjustable band gap nature.
Polymeric	Mostly organic materials that can readily be operational.
Fat-type	Include components like fat and employ surface-active agents as central stabilers.

Conclusion and Future Perspective

Researchers are actively working on developing nanoparticles in response to the growing need for eco-friendly or green materials that can be simultaneously utilized in biological systems. At the nanoscale, nanoparticles are prevalent due to their potential as stable, eco-friendly materials that can integrate harmoniously with biological systems. This chapter commences by detailing the classification, advantages, and limitations of nanoparticles, followed by an overview of synthesis and characterization methods. Numerous cost-effective and efficient techniques for synthesizing nanoparticles exist, including chemical and biological synthesis, top-down and bottom-up approaches, as well as mechanical methods. Multiple approaches for characterizing nanoparticles are developed to extract information about their shape, structure, optical characteristics, mechanical properties, and physiochemical properties. The most important aspects of nanoparticles depend on the synthesis and characterization techniques used. In the areas of medicine, drug delivery, cosmetics, optics, electronics, and solar energy devices, nanoparticles prove to be beneficial specifically.

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