TECHNIQUES FOR NANOPARTICLE SYNTHESIS

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ABSTRACT

Innovative Nanoparticles synthesis techniques are very important for the development of Nanotechnology and Nanoscience. The scope for new synthetic methods for nanomaterials preparation has been ever demanding with innovative contribution. Various traditional physical and chemical methods are being used for fabrication of nanomaterials but more recently the advantageous use of biological means for nanoparticles synthesis is gaining importance. As these methods are ecofriendly, nontoxic, economical and easily controllable. In this paper, we discussed different synthesis routes for the fabrication of Nanoparticles.

Keywords: *Nanotechnology, Nanoparticles, Micro-organisms, Phytonanotechnology*

1. Introduction

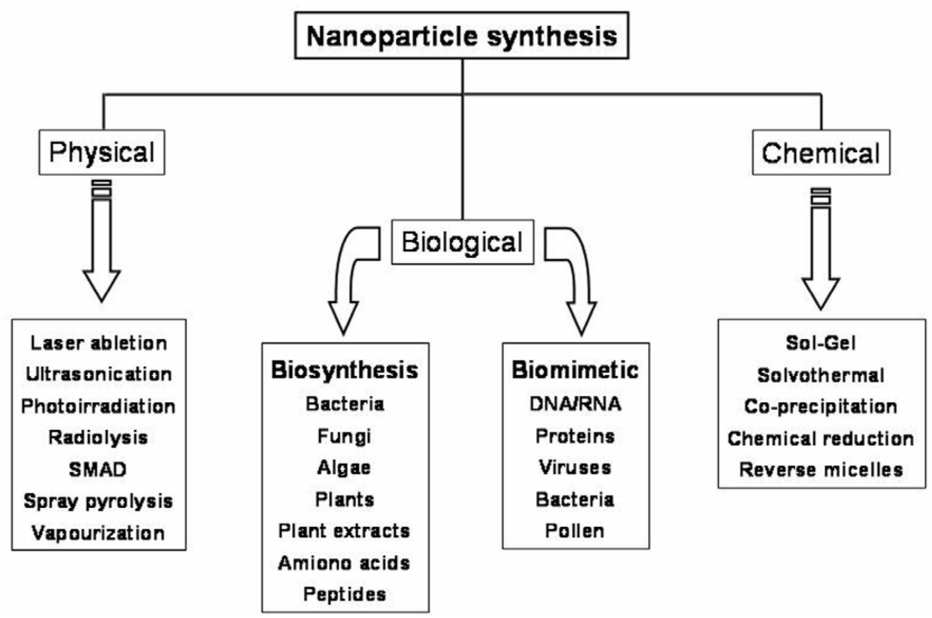
Nanoparticles—particles having one or more dimensions of the order of 100nm or less—have attracted great attention due to their unusual and fascinating properties, and applications advantageous over their bulk counterparts [1]. Although the synthesis of nanoparticles was found in ancient Indian medical and chemical science, where different kinds of Bhasma like Suvarna (gold), Rajat (silver) Bhasma, were used in ayurvedic medicine for the treatment of diseases. However, Michel Faraday is considered as the first to chemically synthesize gold nanoparticles, in solution from aqueous chloroauric acid and phosphorous dispersed in CS_2 [2]. Since then, various physical, chemical and biological methods have been formulated for the fabrication of inorganic nanoparticles with a range of compositions, sizes and shapes. Some of the very successful physical methods for the synthesis of nanoparticles include photoirradiation[3], radiolysis[4], ultrasonication[5], spray pyrolysis, solvated metal atom dispersion[6], chemical vaporization[7], and electrochemical methods[8]. Chemical methods for the synthesis of inorganic nanoparticles are reduction or oxidation of metal ions, or by the precipitation of the necessary precursor ions in the solution phase. The control of size, shape, stability and the assembly of nanoparticles can be achieved by incorporating different capping agents, solvents and templates. Various capping agents ranging from simple ions, to polymers to biomolecules are routinely used for the capping and stabilization of nanoparticles[9]. As a solvent, either water or non-aqueous organic solvents are used for the synthesis of nanoparticles depending on the ultimate application of nanoparticles.

On the other hand, biological methods utilize nature's most efficient machines i.e. living cells for the synthesis of nanoparticles. Biological methods also involve the use of biomolecules as templates or scaffolds for synthesis and assembly of nanoparticles. Many soft and rigid templates such as micelles[10], polymer materials [11], DNA[12], and mesoporous materials[13] have been employed to facilitate control over the formation of desired shape, size and assembly of nanoparticles.

1. Physical Synthesis Methods

1.1 Evaporation methods: Physical vapor deposition(PVD), sputtering and chemical vapor deposition (CVD) are the commonly used methods to form inorganic nanomaterials [14].

(i) PVD involves condensation from the vapor phase which is composed of three main steps: (a) formation of vapor phase by evaporation or sublimation of the material, (b) transporting the material from the source to the substrate, and (c) formation of the particle and/or film by nucleation and growth. Different techniques have been used to evaporate the source such as electron beam, thermal energy, sputtering, cathodic arc plasma, and pulsed laser. Nanowire, nanorod, nanobelt, nanosheet, nanoribbon, and nanotube, etc., have been synthesized using PVD.



(ii) In CVD, the carrier gases containing the elements of the desired compound flow over the surface to be coated. This surface is heated to a suitable temperature to allow decomposition of the carrier gas and to allow the mobility of the deposited atoms or molecules on the surface. It consists of three steps: (a) mass transport of reactants to the growth surface through a boundary layer by diffusion, (b) chemical reactions on the growth surface, and (c) removal of the gas-phase reaction byproducts from the growth surface.

(iii) In sputtering, a discharge of non reactive ions such as argon is created which fall on the target and break the surface atoms, which are collected on the surface to be coated.

1.2 Laser Ablation Method: In this method, metal atom desorption occurs, when intense laser pulses are focused on a metal target. In a Laser ablation experiment, a bulk metal is immersed in a solvent containing surfactant. During the laser irradiation, the metal atoms will vaporize and are immediately solvated by the surfactant molecules to form nanoparticles in solution [15].

1.3 Solvated Metal Atom Deposition (SMAD) Method: In SMAD, a bulk metal is evaporated under vacuum and the vapors of the metal are co-condensed with vapors of organic solvents like acetone to form nanoparticles in solution using a physical method [16]. Evaporation of metal is achieved by electrically heating a metal wire under vacuum. The resulting solution would consist only of colloids and solvent with no byproducts.

1.4 Photolytic and Radiolytic Methods: These methods involve the reduction of metal salts by radiolytically produced reducing agents such as solvated electrons and free radicals and the photolysis of metal complexes in the presence of some donor legands [17]. Radiolysis of aqueous solutions of metal ions gives solvated electrons that may directly react with the metal ions or with other dissolved materials to produce secondary radicals, which then reduce the metal ions to form nanoparticles.

On irradiation of UV light, alcohols form radicals which can reduce the metal ions to form Nanoparticles. When UV light is irradiated on mixture of aqueous metal ions and alcohols metallic nanoparticles like gold and silver are synthesized.

2. Chemical Synthesis Methods

2.1 Chemical precipitation: It is a very simple method of synthesis of nanoparticles.The kinetics of nucleation and particle growth in homogeneous solutions can be adjusted by the controlled release of anions and cations. Careful control of precipitation kinetics can result in monodisperse nanoparticles. Once the solution reaches a critical supersaturation of the species forming particles, only one burst of nuclei occurs. Thus, it is essential to control the factors that determine the precipitation process, such as the pH and the concentration of the reactants and ions. Organic molecules are used to control the release of the reagents and ions in the solution during the precipitation process.

By this method, very complicated nanostructures can also be constructed such as CdS/HgS/CdS, CdS/$(HgS)_2$/CdS and HgTe/CdS quantum well systems and other core/shell structures [18].

2.2 Sol-gel Method: This method is based on inorganic polymerization reactions. It includes four steps: hydrolysis, polycondensation, drying and thermal decomposition [18]. Precursors of the metal or nonmetal alkoxides hydrolyze with water or alcohols according to the hydrolysis process

$$M(OR)_x + mH_2O \rightarrow M(OR)_{x-m}(OH)_m + mROH$$

where if *m* is up to *x*, the reaction is total hydrolysis, followed by either a water condensation or alcohol condensation. Any acid or a base can also help to hydrolyze the precursor.

The solvent must be removed after the solution has been condensed to a gel. Higher temperature calcination is needed to decompose the organic precursor. The size of the sol particles depends on the solution composition, pH, and temperature.

2.3 Hydrothermal Synthesis: Water at elevated temperatures plays an essential role in the precursor material transformation because the vapor pressure is much higher and the structure of water at elevated temperatures is different from that at room temperature. The properties of the reactants, including their solubility and reactivity, also change at high temperatures. The changes mentioned above provide more parameters to produce different high-quality nanoparticles and nanotubes, which are not possible at low temperatures. During the synthesis of nanocrystals, parameters such as water pressure, temperature, reaction time, and the respective precursor- product system can be tuned to maintain a high simultaneous nucleation rate and good size distribution. Different types of oxides and sulfides nanoparticles such as TiO_2, $LaCrO_3$, ZrO_2, $BaTiO_3$, $SrTiO_3$, $Y_2Si_2O_7$, Sb_2S_3, CrN, α-SnS_2, PbS, Ni2P, and SnS_2 nanotubes, Bi_2S_3 nanorods, and SiC nanowires have been successfully synthesized in this way. The solvent is not limited to water but also includes other polar or nonpolar solvents, such as benzene, and the process is more appropriately called solvothermal synthesis in different solvents. [14,18]

3. Biosynthesis of Nanomaterials by Living Organisms

Living organisms, especially micro-organisms like Bacteria, Fungi, Algae, Actinomycetes, Yeast, and Virus etc. have a remarkable ability to form exquisite inorganic structures often in nanodimensions. This ability of living creatures has lured material scientists towards these biological systems to learn and improve the skills for the precise fabrication of nanomaterials at ambient conditions. There exist several examples in biological systems demonstrating not only the efficient synthesis of macroscopic materials like bones and teeth with precise positioning [19] but also in making functional structures in mesoscopic and nanometer dimensions.

Generally synthesis of inorganic nanomaterials by microorganisms has been classified in two categories such as – biologically controlled synthesis and biologically induced synthesis [20]. Biologically controlled synthesis of inorganic materials can often be considered as biomineralization as it is known to occur naturally in few specific organisms. Biogenic nanomaterials commonly have attributes which distinguish them from their inorganic counterparts. The vast array of organisms are now known to synthesize inorganic materials [20], most of them are calcium carbonates, calcium phosphates, silicates, iron oxides and iron sulfides [21].

During biologically controlled synthesis of inorganic materials, inorganic phases grow within or on organic matrix or vesicles inside the cell, allowing the organism to exert a strict control over the composition, grain size, habit, and intracellular or surface location of the produced minerals [20, 22]. Examples of such synthesis include silica biosynthesis in diatoms [23], sponges [24] and radiolarians [25], calcareous structures in coccoliths [26], gypsum in S-layer bacteria [27] and the nanocrystals of magnetite and greigite in magnetotactic bacteria [28].

In Biologically induced synthesis of inorganic materials, organisms modify its ambient microenvironment and create conditions suitable for extracellular precipitation of minerals[28a]. Deliberate synthesis of inorganic nanoparticles is possible because of the specific resistant mechanism exerted by micro-organisms against the high metal ion concentration. At higher concentration of metal ions micro-organisms can cope with the toxic effect of metal ions by one of the defense mechanisms such as effluxing of metal ions by efflux pumps, alteration in the solubility of metal ions, alteration in redox state, extracellular compelxation and extracellular precipitation of metal ions etc [29].

3.1 Mechanism of Synthesis of Nanoparticles

Different biological agents react differently with metal ions leading to the formation of Nanoparticles so the precise mechanism for synthesis through biological routes is yet to be conceived. Generally, nanoparticles are biosynthesized when the microorganisms grab target ions from their environment and then turn the metal ions into the element metal through enzymes generated by the cell activities. It can be classified into intracellular and extracellular synthesis according to the location where nanoparticles are formed [30, 31]. The intracellular method consists of transporting ions into the microbial cell to form nanoparticles in the presence of enzymes. In the intracellular synthesis of nanoparticles, the cell wall of the microorganisms plays an important role. The mechanism involves electrostatic interaction of the positive charge of the metal ions with negative charge of the cell wall. The enzymes which are present within the cell wall reduce the ions to nanoparticles and these nanoparticles get diffused off through the Cell wall. The extracellular synthesis of nanoparticles involves trapping the metal ions on the surface of the cells and reducing ions in the presence of enzymes [32]

3.2 Synthesis of Nanoparticles using Micro-organisms

The various techniques for the synthesis of nanoparticles by the microorganisms include alteration of solubility and toxicity through reduction or oxidation, lack of specific metal transport system, biosorption, extracellular complexation or precipitation of metals, bioaccumulation and efflux system [33]. Nanomineral crystals and metallic nanoparticles having properties identical to chemically synthesized nanomaterials can be synthesized using microorganisms. Different size, shape and composition of the nanoparticles can also be controlled by using microorganisms.

In recent research, bacteria, including Pseudomonas deceptionensis [34], Weissella oryzae [35], Bacillus methylotrophicus [36], Brevibacterium frigoritolerans [37], and Bhargavaea indica [38,39], have been explored for silver and gold nanoparticle synthesis. Similar potential for producing nanoparticles has been shown by using several Bacillus and other species, including Bacillus licheniformis, Bacillus amyloliquefaciens, Rhodobacter sphaeroides [40-42], Listeria monocytogenes, Bacillus subtilis, and Streptomyces anulatus [42, 43]. Various genera of micro-organisms have been reported for metal nanoparticle synthesis, including Bacillus, Pseudomo-nas, Klebsiella, Escherichia, Enterobacter, Aeromonas, Corynebacterium, Lactobacillus, Pseudomonas, Weissella, Rhodobacter, Rhodococcus, Brevibacterium, Streptomyces, Tricho-derma, Desulfovibrio, Sargassum, Shewanella, Plectonemaboryanum, Rhodopseudomonas, Pyrobaculum, and others [44]. These investigations suggest that the main mechanism of the synthesis of nanoparticles using bacteria depends on enzymes [45]; for instance, the nitrate reductase enzyme was found to be responsible for silver nanoparticle synthesis in B. licheniformis. Using Fungi for synthesis of Nanoparticles i.e., Mycosynthesis, is another route for achieving stable and easy biological nanoparticle synthesis. Most fungi containing important metabolites with higher bioaccumulation ability and simple downstream processing are easy to culture for the efficient, low-cost, production of nanoparticles [46]. Moreover, compared with bacteria, fungi have higher tolerances to, and uptake competences for metals, particularly in terms of the high wall-binding capability of metal salts with fungal biomass for the high-yield production of nanoparticles [46,47]. Three possible mechanisms have been proposed to explain the mycosynthesis of metal nanoparticles: nitrate reductase action; electron shuttle quinones; or both [46]. Fungal enzymes, such as the reductase enzymes from Penicillium species and Fusarium oxysporum, nitrate reductase, and /-NADPH-dependent reductases, were found to have a significant role in nanoparticle synthesis [48], similarly to the mechanism found in bacteria. The synthesis of nanoparticles using actinomycetes has not been well explored, even though actinomycetes-mediated nanoparticles have good monodispersity and stability and significant biocidal activities against various pathogens [49]. The synthesis of silver, copper, and zinc nanoparticles using Streptomyces sp. has demonstrated that the reductase enzyme from Streptomyces sp. has a vital role in the reduction of metal salts [50]. Similar to other micro-organisms, yeasts have also been widely investigated for the extracellular synthesis of the nanoparticles on a large scale, with straightforward downstream processing [51-54]. Further-more, virus-mediated synthesis of nanoparticles is also possible. Viruses can be used to synthesize nanowires with functional components that are assembled for various applications, such as battery electrodes, photovoltaic devices, and supercapacitors [55].

3.3 Nanoparticle Synthesis Using Plants

Recently, phytonanotechnology has provided new avenues for the synthesis of nanoparticles and is an ecofriendly, simple, rapid, stable, and cost-effective method. Phytonanotechnology has advantages, including biocompatibility, scalability, and the medical applicability of synthesizing nanoparticles using the universal solvent, water, as a reducing medium [56]. Thus, plant-derived nanoparticles produced by readily available plant materials and the nontoxic nature have significant roles in metal salt reduction and, furthermore, act as capping and stabilizing agents for synthesized nanoparticles [57].

4. Conclusion

Physical and chemical methods for synthesis of nanoparticles are more popular and able to produce large quantities of nanoparticles with a defined size and shape in a relatively short time. But they are complicated, outdated, costly, inefficient and produce hazardous toxic wastes that are harmful, not only to the environment but also to human health[58].

While nanoparticles synthesized by biological process are far superior, in several ways, to those particles produced by chemical methods. Biological methods are reliable, nontoxic, and eco-friendly. With an enzymatic process, the use of expensive chemicals is eliminated, and is not as energy intensive as the chemical method. Nanoparticles synthesis through 'Green' route is easier as it is easy to tailor the size, shape and nature of NPs by simply modifying the culture conditions like temperature, pH, pressure or the nutrient media. They are economically viable as plants and microbes are easily/readily available. The particles generated by these processes have higher catalytic reactivity, greater specific surface area, and an improved contact between the enzyme and metal salt in question due to the bacterial carrier matrix [59, 60]

However, most micro-organism-based synthesis for nanoparticles are slow with low productivity, and the recovery of nanoparticles requires downstream processing. Furthermore, problems related to micro-organism based synthesis for nanoparticles also include the complex steps, such as microbial sampling, isolation, culturing, and maintenance[61].

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