

Methods of Preparation of Nanoparticles

Mr Sharad Kamble, Miss Kaveri Bhosale, Mr Mahesh Mohite, Mrs Swapnali Navale

Nootan College of Pharmacy, Kavathe Mahakal. Sangli, India

Abstract: *The size, shape, and material qualities of nanoparticles can be used to classify them into several categories. Some classifications distinguish between organic and inorganic nanoparticles; nevertheless, the classification of nanoparticles is often determined by their applications or may be connected to how they were formed. Nanoparticles can be found in nature and are also produced as a result of human activity. Nanoparticles have unique material properties due to their sub-microscopic size, and they may find practical uses in a range of fields. A nanoparticle is a distinct nano-object with all three Cartesian dimensions smaller than 100 nm, according to the International Organization for Standardization (ISO). Two-dimensional nano-objects and one-dimensional nano-objects are both described in the ISO standard. However, the definition is later changed. Nanoparticles can also be classed as hard, such as silica particles and fullerenes, or soft, such as nanodroplets. For millennia, nanometres have been used to study biological systems and to develop a variety of materials such as colloidal dispersions, metallic quantum dots, and catalysts. For example, more than a thousand years ago, the Chinese used Au nanoparticles as an inorganic dye to provide red colour to their ceramic porcelains. Although a complete study on the creation and properties of colloidal gold was only published in the middle of the nineteenth century, its use has a long history. Colloidal Faraday's gold dispersion, was created in 1857. Nanotechnology is a technology for designing, fabricating, and applying nanostructures and nanomaterials in general. Fundamental knowledge of the physical properties and phenomena of nanomaterials and nanostructures is also required for nanotechnology. Nanoscience is the study of basic links between physical characteristics and events in nanoscale scale materials. Nanotechnology is described in the United States as materials and systems with nanoscale structures and components that display innovative and considerably improved physical, chemical, and biological properties, phenomena, and processes. Here are some of the techniques for making nanomaterials.*

Keywords: Nanoparticles

I. INTRODUCTION

A nanoparticle is a small particle that stages from 1 to one hundred nanometres in size. Undetectable through the human eye, nanoparticles can show off considerably distinctive bodily and chemical. [1] A nanoparticle is a small particle that stages from 1 to one hundred nanometres in size. Undetectable through the human eye, nanoparticles can show off considerably distinctive bodily and chemical homes to their large fabric counterparts. [2] Petrie's to their large fabric counterparts. Nanoparticles (NPs) and nanostructured accoutrements [NSMs] represent an active area of exploration and a techno-profitable sector with full expansion in numerous operation disciplines. [3] NPS and NSMs have gained elevation in technological advancements due to their tuneable physicochemical characteristics similar to the melting point, wettability, electrical and thermal conductivity, and catalytic exertion. light immersion and scattering performing in enhanced performance over their bulk counterparts. [4] A nanometre (nm) is an International System of Units. A unit that represents 10^{-9} meters in length. In principle, NMs have described accoutrements with lengths of 1–1000 nm in at least one dimension still. they are generally, however; they are generally defined to be periphery in the range of 1 to 100 nm. [5]

Methods of Nanoparticles:

1. Mechanical Methods

Synthesis of Nanomaterials by High Energy Ball Milling:

A ball mill is a type of grinder that is used to mix or grind materials for a variety of purposes. The impact force allows the feed material to be reduced in size. The horizontal axis of the cylindrical shell rotates. [6] The hard and tiny balls in

the ball mill are the most important component for grinding. Steel is commonly used for these balls. Abrasion-resistant material is applied to the hollow shell's interior surface.[7] As the shell spins, the balls are raised up and then dropped at a 60° angle. This free-falling movement has the effect of reducing the size of the particles. Metal ores can also be ground with a ball grinder. Ball Mill with a High Energy. [8]

A ball mill is a type of grinder that is used to mix or grind materials for a variety of purposes. force reduces the size of the feed material. The horizontal axis of the cylindrical shell is rotated. The hard and tiny balls in the ball mill are the essential component that allows for grinding. Steel is commonly used to make these balls. The hollow shell's inner surface is covered with an abrasion-resistant substance. As the shell spins, the balls rise and then fall at a 60° angle. This free-falling movement has an influence, reducing the size of the particles. Metal ores can also be ground with a ball mill.[9]

Three Roll Milling is better than conventional mixing methods since it produces materials with:

- Smaller Particle size
- Sizes are evenly distributed.
- Improved ointment consistency
- Quality control is simple.[10]

High-energy ball milling has the following benefits:

- Installation costs are low.
- Grinding at a low cost
- By simply changing the balls, the capacity and fineness of the process may be modified.
- Batch and continuous operations are both possible.
- Grinding in both open and closed circuits is possible.
- It can be used on any material, regardless of hardness.
- Applications:
- Paint Dressing Processes Mechanical Alloying Refractory Material Production
- Selected Laser Sintering (SLS) is a method of sintering

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- Mechanical Alloying
- Refractory Material Production
- Paints
- Dressing Processes
- Selective Laser Sintering

2. Melt Mixing as Method to Disperse Carbon Nanotubes into Thermoplastic Polymers:

Two methods of incorporating nanotubes into polymer matrix were used in melt mixed composites. In the first case, commercially available masterbatches of nanotube/polymer composites are used as starting materials, which are then diluted by the pure polymer in a subsequent melt mixing process (masterbatch dilution method), whereas nanotubes are directly incorporated into the polymer matrix in the second case. [11] Composites of polycarbonate with MWNT, which were manufactured using a Brabender PL19 single screw extruder, are provided as an example of the master batch dilution method. Electrical percolation was seen in this system at a concentration of 0.5 wt.% MWNT. [12] The nanotube dispersion detected using TEM is rather homogenous. In composites of polycarbonate with MWNT and SWNT, the direct incorporation approach is explored. Depending on the aspect ratio and purity of the materials, commercial MWNT percolation was found to be between 1.0 and 3.0 wt.% [13] Percolation of HIPCOSWNT from CNI yielded 0.25 to 0.5 wt.% SENT. The addition of nanotubes to composites alters their stress-strain behaviour significantly: modulus and stress are increased, while elongation at break is reduced, particularly above the percolation concentration.[14]

3. Laser Ablation

In this method, the vaporization of the material is affected using pulses of the laser beam of high power. The setup is a high vacuum system equipped with an inert gas introduction facility and laser beam. Clusters of any material of which a solid target can be made are possible to synthesize. [15] The laser which gives UV wavelength such as excimer laser is required as other wavelengths like IR or visible are often reflected by some of the metal surfaces. [16] A powerful beam of laser evaporates the atoms from a solid source and atoms colloid with inert gas atoms and cool on them forming clusters. They condense on the cooled substrate. This method is known as laser ablation. [17]

Laser ablation is a technique for creating nanoparticles such as semiconductor quantum dots, carbon nanotubes, nanowires, and core-shell nanoparticles. Nanoparticles are created in this process by the nucleation and development of laser-vaporized species in a background gas. [18] The ability to produce high-purity nanoparticles in the quantum size range [10 nm] is aided by the quick quenching of vapour. The production mechanism of nanoparticles by laser ablation is summarised at this point. [19] The problems for functional nanoparticle synthesis using sophisticated laser ablation technology are then highlighted, as well as recent developments in nanoparticle size management [20]

Chemical Methods:

Colloids synthesis:

These are the phase-separated sub-micrometre particles in the form of spherical particles, rods, tubes and plates etc. These are the particles suspended in some hot matrix. Metal, alloy, semiconductor and insulator particles of different sizes and shapes can be synthesized in an aqueous or non-aqueous medium. [21] The synthesis of colloids is a very old method. M. Faraday synthesized gold nanoparticles by wet chemical route. The particles are so stable. Colloidal particles are synthesized in a glass reactor. Glass reactor has a provision to introduce some precursors, and gases as well as measure temperature, pH etc; during the reaction [22]

It is possible to remove the products at suitable time intervals. The reaction is carried out under an inert atmosphere to avoid any uncontrolled oxidation of the product. Chemical methods provide an easy way to synthesize silver nanoparticles (Ag NPs) in solution. [23] These metal nanoparticles have great potential for biomedical applications as antibacterial, antifungal, and antiviral agents or in wound healing. The adjustment of the parameters involved in these reactions permits precise control over the size, shape, monodispersity and surfaces of the nanoparticles. These nanoparticles are being used in the design of new hybrid organic-inorganic or inorganic nanomaterials for biomedical applications. [24]

Methods Based on Evaporation

Physical Vapour Deposition:

This method usually involves the use of materials of interest as sources of evaporation. Inert gas or reactive gas for collisions with material vapour. A cold finger on which nanoparticles can condense, a scraper to scrape nanoparticles and a piston anvil. [25] All the processes are carried out in a vacuum chamber so that the desired purity of the end product can be obtained. Generally, high vapour pressure metal oxides are evaporated from filaments of refractory metals like W, Ta, and Mo in which the materials to be evaporated are held. [26] The density of the evaporated material close to the source is quite high and particle size is small [5 nm] such particles would prefer to acquire a stable lower energy state. Due to small particle-particle interaction, bigger particles can be formed. [27] Hence, they should be removed away from the source as fast as possible. This is done by forcing an inert gas near the source, which removes the particles from the vicinity of the source. In general, the rate of evaporation and the pressure of gases inside the chamber determine the particle size. Evaporated atoms and clusters tend to collide with gas molecules and make bigger particles, which condense on cold fingers. [28]

The preparation of thin films by physical vapour deposition methods is described. At first, the different processes for the ejection of particles into the vacuum and the characteristic properties of the particles (ionization degree, kinetic energy etc.) are discussed. The influence of the growth parameters on the initial growth and the growth after coalescence is reported. The interrelations are illustrated by experimental findings and computer simulations. [29]

Synthesis of Metal Nanoparticles by Colloidal Method:

This process is done by the reduction of some metal salt or acid. For example, copper particles can be obtained by reducing Chloroauric acid (HAuCl_4) with trisodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$). The reaction will be, $\text{HAuCl}_4 + \text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \rightarrow \text{Au} + 3\text{C}_6\text{H}_5\text{O}_7^- + 4\text{HCl} + 3\text{NaCl}$. The reaction will be carried out in water. [30] Obtained nanoparticles exhibit colour depending upon the particle size. i.e. (intense red colour for gold metal). In a similar way Silver, Gold, Palladium and a few other metal nanoparticles can be synthesized using appropriate precursors, temperature, pH, and duration of synthesis. [31]

Sol-Gel Method:

In this method two types of materials or compounds 'sol' and 'gel' involves. This process is a low-temperature process, hence less energy consumption and less pollution. Sols are solid particles in a liquid. They are a sub-class of colloids. Gels are nothing but a continuous network of particles with pores filled with liquid. A sol-gel process involves the formation of sols in a liquid and then connecting the sol particles to form a network. By drying the liquid, it is possible to obtain the International Journal of Trend in Scientific Research and Development [IJTSRD] powders and thin films. This method is useful to synthesize ceramics or metal oxides, sulphides, borides and nitrides. [32]

Sol-gel synthesis involves hydrolysis of precursors, condensation followed by polycondensation to form particles, gelation and drying process by various routes. Precursors are to be chosen so that they tend to form gels. Both alkoxides and metal salts can be used. It is also possible to synthesize nanoparticles like nanorods, nanotubes etc. by sol-gel technology. [33]

Biological Methods:

Synthesis using Plant Extracts: The use of plants in the synthesis of nanoparticles is quite a less studied area as compared to the use of microorganisms to produce nanoparticles. There are a few examples which suggest that plant extracts can be used in the synthesis of nanoparticles. To obtain gold nanoparticles from geranium plant extract is discussed here. Finely crushed leaves are put in an Erlenmeyer flask and boiled in water just for a minute. Leaves get ruptured and cells release intracellular material. The solution is cooled and decanted. This solution is added to the HAuCl_4 aqueous solution, and nanoparticles of gold start forming within a minute. [34]

Bio-based methods

Several reports prevailed in the literature indicate that the synthesis of nanoparticles by chemical approaches is eco-unfriendly and expensive. Thus, there is a growing need to develop environmentally and economically friendly processes, which do not use toxic chemicals in the synthesis protocols. This has conducted researchers to look at the organisms. The potential of organisms in nanoparticle synthesis ranges from simple prokaryotic bacterial cells to eukaryotic fungi and plants. Some examples of nanoparticle production include using bacteria for gold, silver, cadmium, zinc, magnetite, and iron NPs; yeasts for silver, lead and cadmium NPs; fungi for gold, silver and cadmium NPs; algae for silver and gold NPs; plants for silver, gold, palladium, zinc oxide, platinum, and magnetite NPs. [35]

Bio-based protocols could be used for the synthesis of highly stable and well-characterized NPs when critical aspects, such as types of organisms, inheritable and genetical properties of organisms, optimal conditions for cell growth and enzyme activity, optimal reaction conditions, and selection of the biocatalyst state have been considered. Sizes and morphologies of the NPs can be controlled by altering some critical conditions, including substrate concentration, pH, light, temperature, buffer strength, electron donor (e.g., glucose or fructose), biomass and substrate concentration, mixing speed, and exposure time. In the following section, we discussed the synthesis of NPs. [36]

Tollens's method:

A simple one-step process, Tollens's method, has been used for the synthesis of silver NPs with controlled size. This green synthesis technique involves the reduction of $\text{Ag}(\text{NH}_3)_2^+$ (as Tollens's reagent) by an aldehyde. In the modified Tollens procedure, silver ions are reduced by saccharides in the presence of ammonia, yielding silver nanoparticle films (50-200 nm), silver hydrosols (20-50 nm) and silver NPs of different shapes. In this method, ammonia concentration and the nature of the reducing agent play an important role in controlling the size and morphology of silver NPs. It was

revealed that the smallest particles were formed at the lowest ammonia concentration. 37] Glucose and the lowest ammonia concentration (5 mM) resulted in the smallest average particle size of 57 nm with an intensity maximum of surface plasmon absorbance at 420 nm. Moreover, an increase in NH_3 from 0.005 M to 0.2 M resulted in a simultaneous increase in particle size and polydispersity. Silver NPs with controllable sizes were synthesized by reduction of $[\text{Ag}(\text{NH}_3)_2]^+$ with glucose, galactose, maltose, and lactose [38].

The nanoparticle synthesis was carried out at various ammonia concentrations (0.005-0.20 M) and pH conditions of 11.5-13.0 resulting in average particle sizes of 25-450 nm. The particle size was increased by increasing (NH_3) , and the difference in the structure of the reducing agent (monosaccharides and disaccharides) and pH (particles obtained at pH 11.5 were smaller than those at pH 12.5) influenced the particle size. Polydispersity also decreased by lowering the pH. Produced silver NPs were stabilized and protected by sodium dodecyl sulphate (SDS), polyoxymethylene sorbinatemonooleate (Tween 80), and polyvinylpyrrolidone (PVP 360). [39]

Irradiation methods

Silver NPs can be synthesized by using a variety of irradiation methods. Laser irradiation of an aqueous solution of silver salt and surfactant can produce silver NPs with a well-defined shape and size distribution. Furthermore, the laser was used in a photo-sensitization

synthetic method of making silver NPs using benzophenone. At short irradiation times, low laser powers produced silver NPs of about 20 nm, while an increased irradiation power produced NPs of about 5 nm. Laser and mercury lamps can be used as light sources for the production of silver NPs. In visible light irradiation studies, photo-sensitized growth of silver NPs using thiophene (sensitizing dye) and silver nanoparticle formation by illumination of $\text{Ag}(\text{NH}_3)^+$ in ethanol have been done.[40]

Electrochemical synthetic method:

The electrochemical synthetic method can be used to synthesize silver NPs. It is possible to control particle size by adjusting electrolysis parameters and to improve the homogeneity of silver NPs by changing the composition of electrolytic solutions. Polyphenolpyrrolecoated silver nanospheroids (3-20 nm) were synthesized by electrochemical reduction at the liquid/liquid interface. This nano-compound was prepared by transferring the silver metal ion from the aqueous phase to the organic phase, where it reacted with the pyrrole monomer. In another study, monodisperse silver nanospheroids (1-18 nm) were synthesized by electrochemical reduction inside or outside zeolite crystals according to the silver exchange degree of compact zeolite film-modified electrodes. [41]

Furthermore, spherical silver NPs (10-20 nm) with narrow size distributions were conveniently synthesized in an aqueous solution by an electrochemical method. Poly N-vinylpyrrolidone was chosen as the stabilizer for the silver clusters in this study. Poly N-vinylpyrrolidone protects NPs from agglomeration, significantly reduces silver deposition rate, and promotes silver nucleation and silver particle formation rate. Application of rotating platinum cathode effectively solves the technological difficulty of rapidly transferring metallic NPs from cathode vicinity to bulk solution, avoiding the occurrence of flocculates in the vicinity of the cathode, and ensures monodisperses of particles. The addition of sodium dodecyl benzene sulfonate to the electrolyte improved particle size and particle size distribution of silver NPs.[42]

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