*Section A-Research paper*

# A Comprehensive review on methods of preparation for Nanoparticles

**Surender[a], Rakesh Kumar[b], Chandra Mohan[c,*], Amit Kumar Rawat[d]**

[a,c]Deptt. of Chemistry, SBAS, K R Mangalam University, Gurugram, India

[a,b]Deptt. of Chemistry, Maharaja Surajmal Institute of Technology (GGSIPU), New Delhi, India

[d]Deptt. of Chemistry, Hansraj College, University of Delhi, New Delhi, India

***Corresponding Author:**

gurgaonmohan@yahoo.co.in

**Abstract**

Nanoparticles (NP's) have attracted material science researchers due to their distinctive properties and diverse applications. These materials have firmly occupied their space in various disciplines like chemistry, material science, physics and engineering. Preparatory method for nanoparticles can be chemical, physical or biological using bottom-up or top-down approach. A lot of developments were made in last couple of decades in synthetic methods for nanoparticles. The present review mainly covers important chemical, physical and biological methods for nanoparticles synthesis.

**Keywords:** nanoparticles, nanocomposites, top-down, bottom-up, polydispersity, green, biological.

**Introduction:**

The nanomaterials are the game changers of the material world because of their useful properties and multidimensional applications. The researchers from various disciplines like chemistry, material science, physics and engineering are continuously working to enhance their. The valuable properties of nanoparticles are mainly affected by their coefficient of polydispersity, standard deviation and mean diameter. The important parameters of nanoparticles i.e., chemical, physical properties and size, are determined by the growth mechanism which is further affected by temperature, viscosity, concentration of medium etc. The various methods of nanoparticle synthesis are chemical, physical and biological (Figure 1) and the present review covers all important methods under these sections.

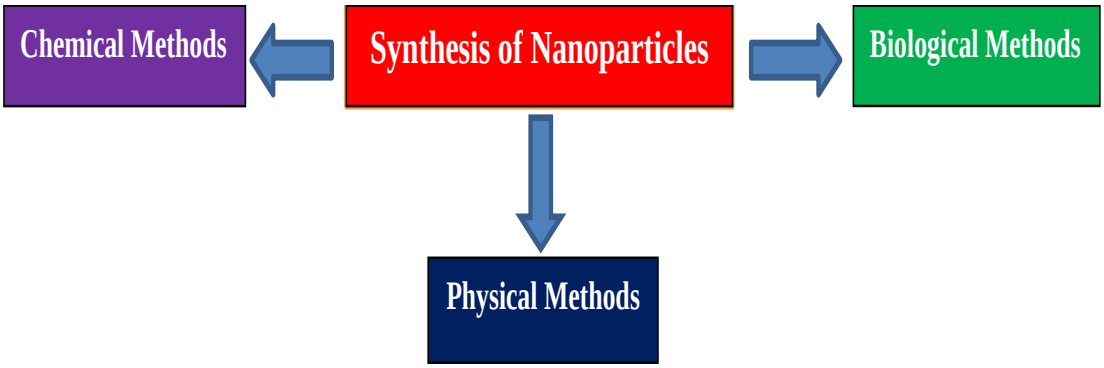


Figure 1. Methods of preparation for nanoparticles.

***Chemical methods of preparation for NP's:***

These methods involve chemical processes which may be top-down or bottom-up and yields pure nanoparticles with homogenous morphology (Figure 2 & Table 1). The chemical methods can be used to produce nanoparticles at large scale but cost is high.

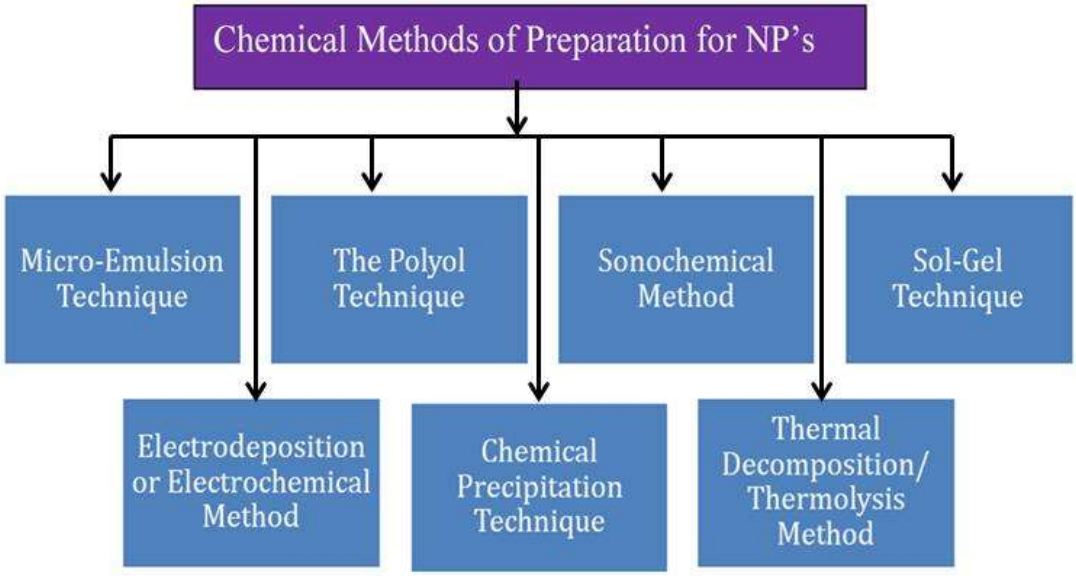


Figure 2. Chemical methods of preparation for nanoparticles.

**Thermal Decomposition/ Thermolysis Method:**

It's is a top-down approach mainly applied for preparation of both metal as well as metal oxide nanoparticles. The target material is decomposed chemically by application of heat. The heat so applied breaks chemical bonds resulting decomposition of the precursor compound. The decomposition process is generally endothermic although in some cases it may be exothermic. Chaudhary et al. prepared chromium (monoclinic) nanoparticles via thermal decomposition technique using chromium (III) chloride precursor. They evaluated thermal decomposition technique as effective, simple and fast. The nanoparticles so obtained were reported as monoclinic crystals having MPS (mean particle size) 45.20 nm [1]. Sanchez et al. synthesized MnxGa1−xFe2O4 (x = 0-1) nanoparticles using tetraethylene glycol mediated thermal decomposition technique. The supermagnetic nanoparticles (SMNPs) so obtained were reported to have inverse spinel crystalline structure, near about spherical morphology and MPS (mean particle size) 5.6± 1.5 nm. They recommended these materials for their potential biomedical applications in future [2]. Mohadesi et al. prepared mercuric oxide (HgO) nanoparticles by heating powdered mercury (II) acetate up to thermal decomposition (sublimation) temperature (150 - 180 °C) for 2 hours. The formation of pure mercuric oxide nanoparticles formation was reported orthorhombic geometry [3]. Darezereshki et al. synthesized zinc oxide (ZnO) nanoparticles (NP's) using Zn4(SO4)(OH)6•1/2 H2O salt precursor. The zinc salt precursor was thermally decomposed by one hour open calcination process at temperature 825 °C. Pure ZnO nanoparticles of spherical shape were prepared with estimated size 87 nm and 92 nm. They suggested that the methodology can be applied at industrial scale because of its low cost [4]. Tomar et al. synthesized CoFe2O4 nanoparticles by thermal decomposition of Co-Fe glycolate which in turn has been synthesized by refluxing of the metal salts of Co and Fe in ethylene glycol. The effect of varying the temperature and strength of ethylene glycol on morphology of nanoparticles was also investigated. The magnetic nanoparticles were reported to show enhanced magnetic properties with excellent chemical stability and thermal stability [5].

**The Polyol Technique:**

A bottom-up approach used for preparation of metal, metal oxide, semiconductor and bimetal¬lic alloy nanoparticles by employing non aqueous solvent (polyol). The polyol acts as solvent, reducing and passivating agent. The polyol helps to control shape, size and texture of nanoparticles with mini¬mized surface oxidation and agglomeration. The solvent used is usually ethylene glycol for preparations of metal oxide nanoparticles due to its useful properties i.e., high boiling point, high dielectric constant, crosslinking ability and strong reducing capability. Koventhan et al. prepared manganese cobaltate ($MnCo_2O_4$) nanoparticles with hexagonal geometry in presence of ethylene glycol as reductant and stabilizer using polyol technique. The fabricated manganese cobaltate electrode using $MnCo_2O_4$ nanoparticles reported to exhibit selectivity, stability, reproducibility and excellent sensitivity [7]. Kim et al. synthesized spherical silver nanoparticles of various standard deviations and sizes by polyol technique. Two methods were used for preparation viz. precursor heating method and precursor injection method to compare the effect of various reaction parameters on nanoparticle size [7]. Park et al. prepared iron rich Fe-Co core - cobalt rich Fe-Co oxyhydroxide layer of different particle size by differential rate of reduction of Fe and Co using facile modified polyol technique. Among all sizes Fe-Co 173 reported to exhibit good stability and enhanced catalytic properties. They recommended FeCo electrodes for hydrogen production by electrochemical oxidation of water [8]. Oh et al. synthesized superparamagnetic (SPM) $Fe_3O_4$ nanoparticles of 11 nm - 338 nm range by polyol technique. They investigated impact of varying concentration of sodium acetate and iron precursor on size of $Fe_3O_4$ nanoparticles and reported the decrease particle size with of increase sodium acetate concentration while particle size found to decrease with decrease in iron precursor concentration. The nanoparticles so obtained by this method recommended for biomedical applications such as hyperthermia treatment, MRI and drug delivery [9]. Julio Jimenez et al. prepared $Fe_{55}Co_{45}$MNPs (magnetic nanoparticles) of 19.1nm - 28.6 nm range using green chemical polyol technique. They developed green methodology by preparing aqueous extract of leave of Mangifera Indica. The green polyol technique was reported to produce $Fe_{55}Co_{45}$nanoparticles quickly with superior saturation magnetization (92.44 emu/g) values. They recommended these magnetic nanoparticles obtained by this technique for water treatment and various biomedical uses such as drug delivery devices [10].

**Micro-Emulsion Technique:**

It's is a bottom-up approach and widely applied for synthesis of inorganic nanoparticles. The emulsions are liquid in liquid dispersions formed by reduction of interfacial tensions with the help of energy and adding sufficient amount of surfactant. The surfactant stabilizes the particles and helps in size control. The polymers also produce emulsions in liquid state which are of three of types depending on the droplet size i.e., micro-emulsions, mini-emulsions and macro-emulsions. The metallic nanoparticles are synthesized using water in oil micro-emul¬sion by mixing of micro-emulsions of a metal salt with other micro-emulsions containing a reducing agent. The collisions among the reactants in the mixture of two micro-emulsions due to Brownian motion results into mixing, coalescence and fusion. The formation of metal nuclei by the reaction among solubilizates. The nanodroplets size, shape and type of surfactant used greatly affect the nanoparticles morphology. Chin et al. prepared starch nanoparticles using precipitation in water-in-oil micro-emulsion technique. They evaluated various parameters affecting nanoparticles properties: oil phases, ratios of oil/co-surfactant, stirring rates, co-surfactants, and ratios of water/oil. The direct nanoprecipitation technique was reported to yield nanoparticles with MPS (mean particle size) of 109 nm while, precipitation in micro-emulsion method yielded nanoparticles with MPS (mean particle size) of 83 nanometer [11]. Barad et al. prepared maghemite ($Fe_2O_3$) nanoparticles by water-in-oil micro-emulsion technique using $FeCl_3$ and $FeSO_4$as metal precursors in presence of surfactant SDS (Sodium dioctylsulfosuccinate). The synthesized

magnetic nanoparticles were coated with oleic acid and then applied for batch mode adsorption of Cr (VI) [12]. Mangaiyarkarasi et al. synthesized palladium (Pd) nanoparticles with MPS (mean particle size) ~20 nm by using in- situ ILC (Ionic liquid crystal) micro-emulsion technique. They prepared three micro-emulsion namely ammonium ILC (I) (containing a cholesterol core), imidazolium ILC (II) (bearing a cholesterol core) and imidazolium ILC (III) (containing a biphenyl core). The palladium (Pd) nanoparticles prepared by three different micro-emulsions were used as catalyst in Heck reaction and catalyst prepared by ILC (II) micro-emulsion was reported to exhibit better activity as compared with the other two i.e. ILC (I) and ILC (III) [13]. Akbari et al. prepared Fe-Co/ MgO nanoparticles with uniform size distribution using micro-emulsion method. The nanoparticles so obtained were applied first time as catalyst in Fischer-Tropsch process. They investigated effect of different calcination conditions on catalytic performance, physic-chemical properties and product selectivity [14]. Chen et al. synthesized CaO-CuO nanostructured composites by micro-emulsion technique for CO2 uptake applications. The effect of various parameters like water to surfactant molar ratio and precursors molar concentration micro-emulsion system were evaluated. The study showed that with increase of water to surfactant molar ratio nanoparticles size also increases while high precursor molar concentration increases CO2 uptake. The resulting CaO-CuO nanostructured prepared by micro-emulsion technique reported to show fewer declines in activity in successive cycles as compared to reference material [15].

**Electrodeposition or Electrochemical Method:**

Electrochemical synthesis is the bottom-up approach for preparation of metal nanoparticles in an electro¬chemical cell. This method involves use of aprotic solvent for dissolution of a metallic anode. The target metal ions then reduced electrochemically to produce nanoparticles. The nanoparticle size can be controlled by using diverse counter electrodes and current density. The electrochemical synthesis method has some important advantages like accurate tune ability with preferred potential and rejection of the potential wasteful alternative half-reaction [16]. Jiangfeng et al. synthesized ZnO nanoparticles by electrodeposition approach using solution precursor. They investigated important parameters such as concentration of the electrolyte which affects surface morphology, shape and size were. The well-aligned nanorod arrays formation was reported at low concentration while anomalous hexangular nanoparticles formed at higher concentration [17]. Pan et al. synthesized silver (Ag) nanoparticles (NPs) films by novel in-situ electrodeposition of carboxylated chitosan on substrate. The carboxylated chitosan acts as a component in the film at the same time it acts green reductant and stabilizing agent. The formation of homogeneous film nanocomposite film with MPS (mean particle size) 10 nm with antibacterial properties was reported [18]. Rasouli et al. synthesized zinc oxide nanotube arrays decorated silver (Ag) nanoparticles (NP's) on TiO2 (fluorine doped) using electrodeposition method. The nanotube arrays so obtained were used for photo-electrochemical splitting of water under UV light. They evaluated ZnO nanotube arrays decorated with silver (Ag) shows enhanced performance because of reduced band gap. The electrodeposition method was reported simple and cheaper as compared to other methods [19]. Alkuamet et al. prepared CdS (Cadmium sulphide) thin film and CdS (Cadmium sulphide) nanoparticles using electrodeposition method and chemical bath deposition method. The CdS thin films and nanoparticles were used for fabrication of solar cell devices. They evaluated electrodeposition technique as a potential technique for fabrication of CdS thin films at large scale [20]. Zhang et al. synthesized silver (Ag) nanoparticles (NP's) on AZO (aluminum-doped zinc oxide) using electrodeposition method under ambient conditions. The aluminum-doped zinc oxide glass reported to show better properties as compared to bare AZO glass because of their localized surface plasmon resonances property. They recommended the Ag on AZO glass for optoelectronics applications mainly in solar cells due to superior reflection property [21].

**Chemical Precipitation Technique:**

The bottom-up approach in which nanoparticles size is controlled through arrested precipitation methodology. It involves in situ synthesis of nanomaterials which doesn't allow physical changes and helps in size control by blocking aggregation. The Oswald ripening and thermal coagulation can be controlled by use non-aqueous solvents by inducing double layer repulsion of tiny crystallites at lower temperatures. The role of added surfactant is to maintain space among the particles. The nanoparticles so formed can be recovered using centrifugation are washed and vacuum dried. Li et al. prepared NixNb1-xO nanoparticles with sponge and fold like structures. They varied Ni:Nb ratio to synthesize a series of NixNb1-xO nanostructures. The surface area of synthesized nanoparticles was reported 173 m2/g with investigation of catalytic hydro-conversion of lignin derived substances using Ni0.92Nb0.08O catalyst [22]. Viruthagiri et al. synthesized ceria (CeO2) doped ZrO2-CuO nanoparticles using chemical precipitation technique. They reported formation of nanoparticles with MSP (Mean particle size) 5-6 nm range [23]. Kuriakose et al. prepared La2(WO4)3 (Lanthanum tungstate) nanoparticles in aqueous medium by chemical precipitation technique. The La2(WO4)3 nanoparticles so obtained were investigated for their photo luminescent and photo catalytic properties. They recommended these materials suitable for treatment water containing organic pollutants like dyes and fabrication of LED lights [24]. Patel et al. synthesized Ni1-xFexO (x = 0.00 to 0.06) nanoparticles using chemical precipitation in presence of PVP (polyvinyl-pyrrolidone) and nitrate salt of metal [25]. Lassoued et al. prepared α-Fe2O3 nanoparticles using chemical precipitation technique. The effect of precursor concentration variation was investigated on morphology, crystalline phase and size of α-Fe2O3 nanoparticles. They obtained nanoparticles with particle size range of 21 and 82 nanometer [26].

**Sol-Gel Techniques:**

It is a bottom-up technique mainly used for preparation of Silicon, Yttrium oxide and Zirconium nanoparticles. This technique involves interaction of colloidal suspension called sol and gelatin to obtain continuous liquid phase network called gel. The solvent used is mostly alcohol while metal alkoxides and aloxysilanes are used as starting materials. The sol gel technique starts with a homogeneous alkoxides solution. The pH of process controlled using a suitable catalyst. The Sol-gel method is comprises of following stages:

(1) Hydrolysis - replacement of [OR] group with [OH-]group
(2) Condensation - formation of low to high order rings
(3) Growth and Agglomeration of particles – formation of nanoparticles.

Dixit et al. synthesized silica nanoparticles in 20 minutes using a sol-gel technique. The precursor tetraethyl orthosilicate (TEOS) in equi-volumetric ethanol-water was heated at 20 oC for 20 minutes in presence of sodium hydroxide (Catalyst) .They used emulsification technique to remove unreacted TEOS and polysilicic acid chains. The chemilumniscent molecule Luminol, has been successfully entrapped between the layers of silica [27]. Ayesha et al. synthesized Zinc-Tin Oxide (ZTO) nanoparticles (NP's) by sol-gel technique at ambient conditions. The synthesized ZTO NP's were fabricated in to thin films on to glass substrate. They investigated photocatalytic performance of ZTO-NP's thin films by photo-degradation of organic dyes. The photocatalytic efficacy of ZTO-NPs was reported 73 % and 62 % for MO and MB respectively [28]. Pavithra et al. prepared NiTiO3 nanoparticles by sol-gel technique. The NiTiO3 ceramics nanoparticlesso obtained showed semiconductor behaviour with activation energy 0.04eV. They recommended NiTiO3 nanoparticles for supermagnetic applications at room temperature [29]. Zorkipliet al. synthesized NiO NPs (Nickel oxidenanoparticles) at pH 11 and 450 °C by sol-gel technique. The NiO NPsobtained by this technique were evaluated as pure with cubic structure. They calculated NiO: Ni: O ratios and average diameter of

nanoparticles (32.9) nm by morphological analysis [30]. Nescakova et al. prepared SiO2-CaO based MBGNs (mesoporous bioactive glass nanoparticles) and Zn2+ ions doped Zn-MBGNs nanoparticles using micro-emulsion mediated sol-gel technique. The prepared Zn-MBGNs nanoparticles were reported to show good dispersity, spherical appearance and size range of 130 ± 10 nm. The doping of MBGNs with zinc precursors was reported to enhance specific surface area without affecting the morphology of nanoparticles. The nanoparticles so obtained were evaluated as suitable for many biomedical uses such as drug delivery, wound cure, soft tissue repairing and bone regeneration [31].

**Sonochemical Method:**

It's a bottom-up technique for preparation of metal nanoparticles. It involves interaction of matter and energy by ultrasound irradiation of precursors (sonochemical synthesis) in presence or absence of some stabilizer. The stabilizer so used is starch, gelatin polyvinyl, pyrrolidone (PVP) and polyethylene glycol (PEG). The process is consisting of three steps: formation, growth and in the end bubbles collapse implosively. There slight increase in pressure and temperature for few seconds due to bubbles collapse followed by cooling. The important properties of nanoparticles viz. particle shape, particle size, and purity can be controlled by solvent, sonication power, chemical species and temperature. Vivekanandan et al. synthesized nickel manganous oxide (crump-like) nanoparticles (Fig. 6) using high-intensity ultrasonic sonication technique for preparing NiMnO@r-GO nanocomposites (NC's) with reduced graphene oxide (r-GO). They explored sensing ability of NiMnO@pr-GO NC by modifying on to a glassy carbon electrode sensor (GCE). The study reported an optimized sensitivity value of 1.220 µA µM-1 cm-2 for the sensor [32]. Zonarsaghar et al. prepared CeVO4 (cerium vanadate) nanoparticles by using sonochemical technique. The NH4VO4 and Ce(NO3)3.6H2O were used as reactants and N2H4 as source of hydroxide ions were allowed to react in presence of ultrasonic radiations and absence of ultrasonic radiations. The various factors were investigated like surfactant, time, solvent, and sonication power with control on reaction using ethylenediamine and hydroxide sources. The nanoparticles so obtained were used for electrochemical storage H2 with excellent capacity [33]. Dinesh et al. synthesized Cu0 nanoparticles (zero valent copper NPs) by sonochemical method using Hibiscus rosa-sinensis extracts as stabilizer and reductant. The Cu0 nanoparticles were reported to have amphoteric nature which exhibit excellent activity of 93.2 % for organic drugs degradation [34]. Nithya et al. prepared bismuth (III) phosphate (BiPO4) nanocrystals by sonochemical technique. The impact of various factors like pH, sonication power & irradiation time on morphology of nanocrystals was investigated. The synthesized BiPO4 nanocrystals reported to have monoclinic structure and recommended for pseudocapacitors applications. They changed matrices to improve cycling stability of bismuth (III) phosphate nanocrystals and obtained maximum with activated carbon [35]. Pugazhenthiran et al. synthesized porous nickel (II) titnate (NiTiO3) nanorods by sonochemical method. The surface morphology of material so obtained was tuned by calcination at different (100 to 600 °C) temperatures. They examined formation of rod shaped and porous nickel (II) titnate nanorods at 600 °C. The porous NiTiO3 nanomaterils were investigated for their photocatalytic activity by degrading CFS (ceftiofur sodium) in sunlight. Further photocatalytic activity was reported to increase in presence of peroxomonosulfate [36].

**Table 1. Chemical methods for NP's synthesis with applications.**

| S.No. | Technique | Type | Applications |
|---|---|---|---|
| 1 | Thermal Decomposition/ Thermolysis Method | Top-down | Metal and Metal oxide NP's |
| 2 | The Polyol Technique | Bottom-up | Metal, Metal oxide, Alloys and Semiconductor NP's |
| 3 | Micro-Emulsion Technique | Bottom-up | Metal and Metal oxide NP's |
| 4 | Electrodeposition or Electrochemical Method | Bottom-up | Metal and Metal oxide NP's |
| 5 | Chemical Precipitation Technique | Bottom-up | Metal and Metal oxide NP's |
| 6 | Sol-Gel Technique | Bottom-up | Metal and Non-Metal oxide NP's |
| 7 | Sonochemical Method | Bottom-up | Metal, Metal oxide and Hydrocarbon NP's |

***Physical methods of preparation for NP's:***

These methods involve physical processes which generally top-down approach based (Figure 3 & Table 2). The physical methods are low cost but yields inhomogeneous nanoparticles.

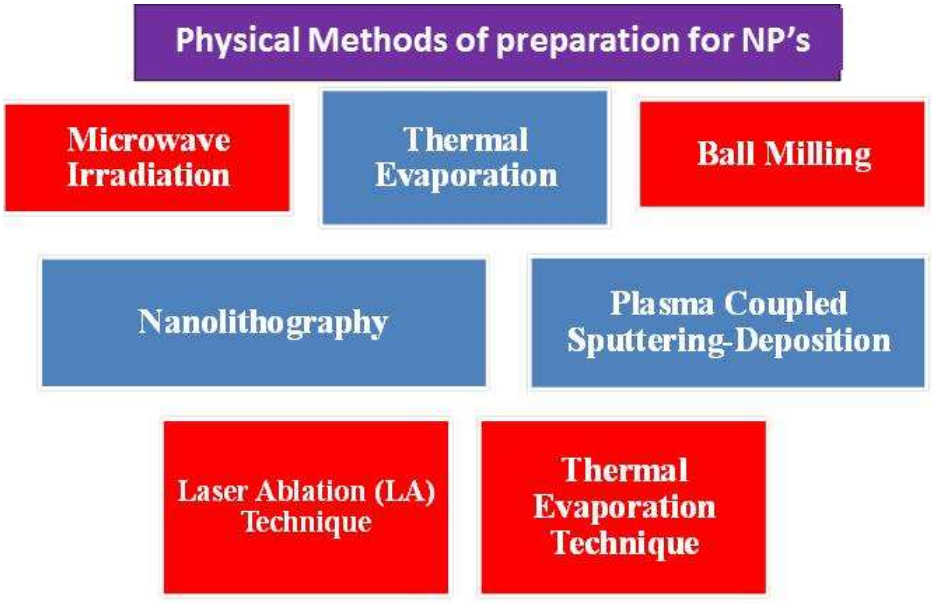


Figure 3. Physical Methods of preparation for nanoparticles.

***Ball milling (Mechanical Method):***

It is a top-down mechanical approach for synthesis of nanoparticles. It involves milling (crushing) of solid materials by balls made up of tungsten carbide (WC), hardened steel and silicon carbide (SiC) in a stainless steel container. The milling process is completed in 100 to 150 hours to yield uniform powder. The advantages of this technique are: simple, low cost and fine nanoparticles of 2-20 nm range but lack of control on purity and shape are some shortcomings. Carvalho et al. synthesized magnetite ($Fe_3O_4$) nanoparticles of size range 12-20 nm using high energy ball milling technique. The synthetic methodology was achieved by

using different stoichiometric combinations of iron powder and distilled water. They investigated effect of milling time on purity and particle size of $Fe_3O_4$ nanoparticles [37]. Zhang et al. prepared magnetic nanoparticles of iron encapsulated with carbon using high energy ball milling of $Fe(NO_3)_3$ (ferric nitrate) and $C_8H_{11}NO_2$ (dopamine). They also investigated effect of dopamine content on carbon content of magnetic nanoparticles. They reported formation of magnetite, martensite and iron nanoparticles iron nanoparticles due increase in carbon content with increase in dopamine content [38]. Suwanboon et al. synthesized Titanium-doped ZnO (zinc oxide) nanoparticles (NP's) using high-energy ball milling method by simultaneous milling of ZnO and $TiO_2$ powders at 400 rpm. The Ti-doped ZnO nanoparticles obtained by milling process carried out for 20 hours were reported to have hexagonal structure with single phase. They investigated the antibacterial properties of ZnO and Ti-doped ZnO NP's and reported effective against *P. aeruginosa, E. coli, S. aureus, P. mirabilis* and *S. typhi* [39].

***Thermal Evaporation Technique:***

A top-down approach mainly used for preparation of metal and alloy nanoparticles. It is among some primitive techniques which were invented for synthesis of nanoparticle and involves evaporation of solid target material using an electron beam evaporation devices or Joule heated refractory crucibles in vacuum at 1-50 milli bar pressure. The gas phase collision and condensation (nucleation) processes yields ultrafine metal and alloy nanoparticles. This technique is simple and economical but formation of inhomogeneous nanoparticles due to non-uniform heating is the main shortcoming [40]. Abdallah et al. synthesized lead sulphide (PbS) doped zinc oxide (ZnO) Nanowires films by thermal evaporation technique on Silicon (100) & glass substrates. The PbS doped ZnO nanowire thin films so obtained were tested for photoluminescence applications. They reported dependence of photoluminescence activity on band gap and the band was found to increase with increase of PbS concentrations [41]. Xie et al. prepared silicon carbide (SiC)/ silicon oxide ($SiO_2$) nano-chains on a carbon fibre substrate by thermal evaporation technique in catalyst free Ar/CO atmosphere. The CO atmosphere was identified as promoter in formation of $SiO_2$ and $SiC/SiO_2$ nano-chains were formed by cooling. The findings were reported as a catalyst in further studies of 1D Silicon carbide based materials [42].

***Microwave Irradiation Technique:***

It is top-down method similar to thermal evaporation & condensation technique which involves heating and evaporation of solid target material using microwave radiations. The microwave radiations provide uniform heating without heating the environment and produces homogeneous nanoparticles. Wang et al. synthesized copper oxide (CuO) nanoparticles using $(CH_3COO)_2Cu$ and NaOH as starting materials in ethanol (solvent) by microwave irradiation technique. The prepared CuO nanoparticles reported to have band gap 2.43 eV, high purity, regular shape and narrow size distribution [43]. Parthibavarman et al. prepared cobalt (Co) doped stannous oxide ($SnO_2$) nanoparticles by microwave irradiation. The synthesized nanoparticles were tested for optoelectronic application. The nanoparticles so obtained were used as ethanol gas sensors with investigation of doping effect of Co on $SnO_2$ and reported that Co doping of $SnO_2$ significantly enhances the sensitivity of sensor [44]. Alle et al. synthesized Au NP's (gold nanoparticles) using CNC (cellulose nanocrystals) as supporting and reducing agent by microwave irradiation technique. The methodology was reported as sustainable, cost-effective and eco-friendly. The size of AuNP's was reported to to alter with CNC concentration. The AuNPs-CNC nanocomposite films were synthesized which showed excellent degradation properties and applied for degradation of Rhodamine B, Allura red and Congo red [45].

***Plasma Coupled Sputtering-Deposition Technique (PCS-DT):***

A top-down technique for preparation of nanoparticles involving erosion of target surface (cathode) with high energy partially ionized noble gas (Argon) particles (Plasma Spray-PS). The target material ions so formed by bombardment are deposited on the substrate at anode end in the form of thin film (Physical Vapour Deposition-PVD). The plasma is created at $10^{-1}$-$10^{-3}$ milli bar pressure. The target material can be used in variety of shapes i.e. rectangular, circular, tubular, delta etc. Mekasuwandumrong et al. synthesized a series of copper/ titanium oxide nanoparticles ($Cu/TiO_2$ NP's) by PDC (pulsed direct current) magnetron sputtering. The $Cu/TiO_2$ NP's were obtained by sputtering of copper in inert (Ar) atmosphere using $TiO_2$ supports as anode. The $Cu/TiO_2$ nanoparticles reported to show excellent photocatalytic activity as compared uncoated $TiO_2$ [46]. Zhu et al. prepared grapheme (Gr) nanodots-encaged porous gold (Au) electrode using ion beam sputtering deposition. They used Au and Al-Gr composite target material and sputtered simultaneously onto anode with glass substrates using beam of Argon ions, followed corrosion. The Gr nanodots-encaged porous Au electrodes so obtained were tested for detection of heavy metal ions ($Cu^{2+}$ and $Pb^{2+}$). The metal detection activity was reported to saturate at 40 nm electrode thickness [47]. Huang et al. synthesized boron-doped microcrystalline silicon thin films using coupled plasma coupled RF magnetron sputtering deposition technique. They used pure boron as cathode sputtering target in silane and argon (Ar) mixture and observed effect of sputtering power from 0.0 to 350.0 W. The structural and electrical properties were reported to improve with increase with target power from 0.0 to 300.0 W but sudden decline beyond 300.0 W. The study was evaluated as relevant for microcrystalline silicon-based *p–i–n* junction based solar cells [48].

***Laser Ablation (LA) Technique:***

It is top-down method in which a laser beam is focused on to solid (occasionally liqiud) target material to ablate (remove) nanoparticles. The characteristics of nanoparticles so formed are dependent on laser parameters such as pulse duration, repetition rate, wavelength and flounce etc. They reported increase in rate of ablation process with increase in laser energy. The advantages of laser ablation technique over the other techniques are: can be carried out at room temperature, no chemical solution required, simple and safe techniques, and high purity, wide range of target materials and nanoparticles with complicated stoichiometry can be produced. The only disadvantage of laser ablation is its high cost. Menazea et al. prepared silver (Ag) nanoparticles (NP's) and copper oxide (CuO) nanoparticles (NP's) embedded in grapheme oxide (GrO) using laser ablation (LA) technique. The nanoparticles so obtained were investigated for photo-activated antibacterial activity against *E. coli*. The photo-activated antibacterial activity of GrO nanoparticles reported to significantly enhanced by addition of Ag and CuO nanoparticles [49]. Tsuji et al. synthesized silver (Ag) nanoparticles (NP's) by primary laser ablation technique onto silver plate as anode in aqueous solution PVP (polyvinylpyrrolidone) and secondary laser irradiation of obtained colloidal solutions. The effect of PVP on particle size reduction was also investigated with prominent size reduction in secondary irradiation step [50]. Gondal et al. prepared NiO (nickel oxide) nanoparticles by pulsed laser ablation (PLA) technique in 3% hydrogen peroxide solution. They reported this methodology very effective in terms of size control, purity and lesser chemical wastes as compared to other wet methods [51].

***Nanolithographic Method:***

A top-down technique supplemented by self-organization, thin film deposition and self-assembly techniques. It involves tightly focusing of a particle beam (ions, electron and light) on to the target surface. There any many types of nanolithographic techniques depending on the type of beam used i.e. Electron-beam lithography, Extreme ultravoilet lithography, Ion-beam lithography, X-ray lithography, Ion-track lithography

and Nanoimprint lithography. Bullen et al. prepared $TiO_2$ (titanium oxide) nanoparticle arrays on glass substrate using nanosphere lithography. Two evaporation masks i.e. monolayer and bilayer were generated using hcp polystyrene nanospheres with different nanoparticle arrays of $TiO_2$. The monolayer and bilayer masks yielded different $TiO_2$ nanoparticles with diameters 169 ± 12 nm and 140 ± 13 nm. The absorption edge of nanoparticle arrays was reported blue-shifted from single crystal rutile [52]. Bera et al. synthesized iron oxide ($Fe_2O_3$) nanoparticles (NP's) on support of Si (100) via spin-coating using reverse micelle (RM) nanolithography. The process involved plasma oxidation and annealing at high temperature. The $Fe_2O_3$ NP's so obtained were unimodal in size with mean diameter ∼14 nm [53]. Li et al. utilized positively charged AUT (11-1-undecanethiol) template on gold (Au) substrate by dip-pen nanolithography (DPN) to synthesize single Au nanoparticle array. The AUT template used were reported to organize nanoparticlese eg. gold and grapheme oxide. They evaluated the electrostatic interactions as the factor which ensures the adsorption of gold and grapheme oxide only at designated AUT areas [54].

***Wire Explosion Method:***

A top-down approach based on Joule's heating effect which involves passing a high power current through a metallic wire. The wire disintegrates due to excess of heat produced by high power current and produces nanoparticles. The simplicity and low cost are the advantages associated with this technique but control over particle size is lacking. Kumar et al. prepared aluminium-magnesium (Al-Mg) binary eutectic alloy nanoparticles in inert ambience using wire explosion technique (WET). The Al-Mg alloy wire was sublimated and nanoparticles were formed by condensation. The kinetic and thermodynamic studies were also carried for the process [55]. Lerner et al. synthesized (Al) and Aluminium/ Aluminium nitride (Al/AlN) composite nanoparticles using explosion of aluminum wires in argon (Ar) and nitrogen ($N_2$). The various parameters affecting mean size of nanoparticles were evaluated: gas medium and aluminum nitride content in the powder. The mechanism believed to take place by coagulation of the primary particles generated in the electrical explosion [56]. Ghosh et al. prepared TiC (Titanium carbide) nanoparticles (NP's) using explosion of titanium (Ti) wire in methane ($CH_4$) as well as diluted acetylene ($C_2H_2$) atmosphere by varying pressures. The dependence of size of nanoparticles with increase in the applied energy was evaluated and found to decrease with increase in energy. The synthesized TiC NP's were tested for $CO_2$ adsorption at low pressure (≤1 bar) and different temperatures. The $CO_2$ adsorption was reported to be physical and exothermic in nature [57].

Table 2. Physical methods for NP's synthesis with applications.

| S.No. | Technique | Type | Applications |
|---|---|---|---|
| 1 | Ball milling (Mechanical Method) | Top-down | Metal oxide NP's |
| 2 | Thermal Evaporation Technique | Top-down | Metal oxide and Semiconductor NP's |
| 3 | Microwave Irradiation Technique | Top-down | Metal and Metal oxide NP's |
| 4 | Plasma Coupled Sputtering-Deposition Technique (PS-PVD) | Top-down | Metal, Metal oxide and Semiconductor NP's |
| 5 | Laser Ablation (LA) Technique | Top-down | Metal oxide NP's |

| | | | |
|---|---|---|---|
| 6 | Nanolithographic Method | Top-down | Metal and Non-Metal oxide NP's |
| 7 | Wire explosion Method | Top-down | Metal, Metal oxide and NP's |

***Biological methods of preparation for NP's:***

The biological approach of nanoparticles synthesis incorporates biotechnology in nanotechnology and utilizes biological resources which are unicellular as well as multicellular like algae, bacteria, yeast, fungi, plants and viruses (Fig. 4 & Table-3). These methods are mainly used for preparation of metal nanoparticles are high-yield, cheaper and eco-friendly.

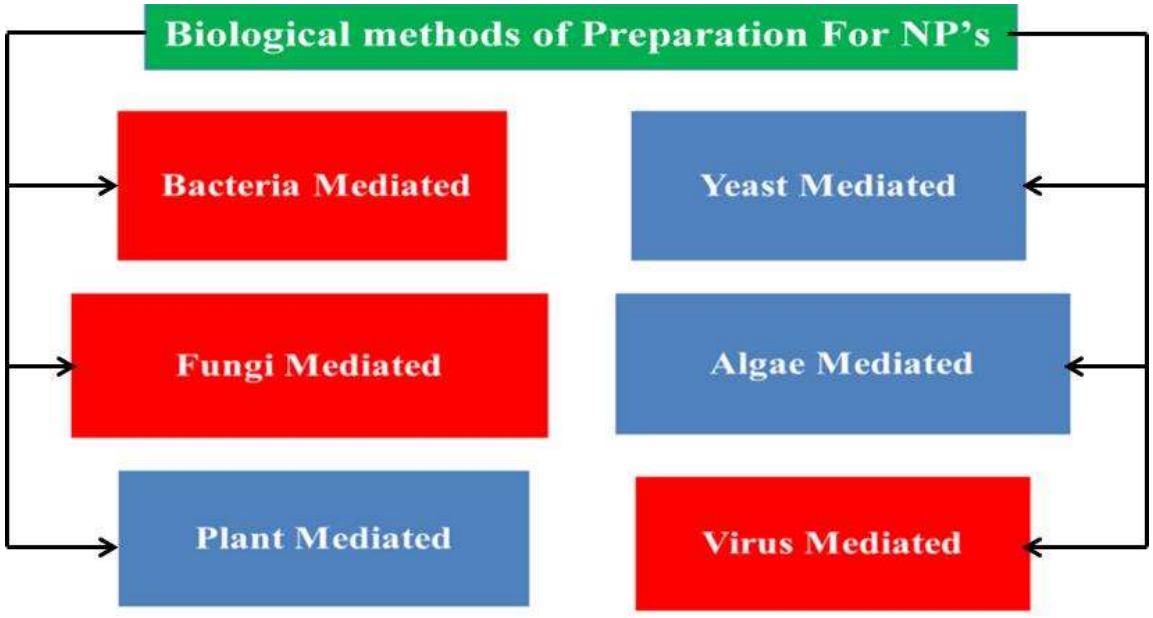


Figure 4. Biological Methods of preparation for nanoparticles.

*Synthesis of nanoparticles using Bacteria:*

The prokaryotic bacteria were identified as most suitable biological resource for nanoparticles synthesis among the millions of biological resources. The bacterial assisted technique is preferred due the ease of manipulation and low cost. The nanoparticles synthesis may take place by intracellular mechanism involving transport of ions into bacterial cell followed by the enzymatic action which forms the nanoparticles while; extracellular mechanism is believed to take place by trapping of metal ions onto the cellular surface and reduction of ions by enzymes. Kanmani et al. evaluated synthesis of silver (Ag) nanoparticles (NP's) using bacterial exopolysaccharide as reducing as well as stabilizing agent. The AgNP's were reported to multidispersed with varied shape and mean diameter of 10.0 nm (2.0 to 15.0 nm). The silver nanoparticles so obtained were investigated for their antibacterial *(P. aeruginosa* and *K. pneumonia*) and antifungal (*Aspergillus and Penicillum)* activity by agar well diffusion method [58]. Saravanan et al. prepared silver (Ag) nanoparticles (NP's) using bacterial exopolysaccharide (EPS). The AgNP's were reported to have spherical shape, mean size of 35 nm with excellent thermal stability up to 437.1 °C. The AgNP's so obtained were investigated for degradation of azo dyes by electron transfer mediated chromophore destruction method. They reported EPS-AgNP's as low cost, green and efficient materials for dye degradation [59]. Ameen et al. synthesized silver (Ag) nanoparticles (NP's) using heavy metal resistant *Cuprividus* sp. Bacteria isolated from soil. The AgNP's so obtained were crystalline having particle size range from 10-

50 nm. The probable mechanism of nanoparticles formation was identified as extracellular enzymatic reduction of Ag (I) ions to Ag (0). The AgNP's were investigated for their antibacterial properties against pathogenic bacteria *Enterobacter xiangfangensis, Stenotrophomonas pavanii, Proteus mirabilis* and *Aeromonas* enteropelogenes [60].

*Synthesis of nanoparticles using Yeast:*

Yeast are non-pathogenic micro-organisms which were extensively used in breweries, bakeries and food industry for their sugar fermentation ability. These are eukaryotic micro-organisms which not only have resistance against toxicity of heavy metals but also have ability to detoxify the same by redox enzymes or structural proteins through extracellular or intracellular mechanism. In recent times yeast are used for ecofriendly preparation of metallic nanoparticles like gold (Au), silver (Ag), titanium (Ti), palladium (Pd), and selenium (Se). The yeast mediated synthesis is advantageous because of low cost and easy handling of biomass. However, manipulation of genetic material of eukaryotic organisms is difficult as compared to prokaryotes. Fernandez et al. investigated synthesis of silver (Ag) nanoparticles (NP's) by using two yeasts supernatants: Cryptococcus laurentii and Rhodotorula glutinis. The yeasts were reported to heve enzyme nitrate reductase which produce nanoparticles. The Ag NP's so obtained were different sizes, disperse and stable. They investigated the synthesized Ag NP's for their antifungal activity against phytopathogenic fungi and reported to have nearly same efficiency to iprodione (conventional fungicide) at 3 ppm concentration [61]. Faramarzi et al. prepared selenium (Se) nanoparticles (NP's) using *Saccharomyces cerevisiae* yeast. The Influences on yeast growth with change in precursor concentration (sodium selenite: 5, 10, 15, 20, and 25 µg) was evaluated at 32 °C. The Se NP's reported to have particle size range from 75 to 709 nm, polydispersity index from 0.189 to 0989 and zeta potential range from-7.06 to -10.3 mV respectively [62]. Zhang et al. investigated novel preparation of gold (Au) nanoparticles (NP's) by using *Magnusiomyces ingens* LH-F1 yeast cells. They reported adsorption of some biomolecules on surface of nanoparticles which were identified as probable candidate for formation of AuNP's. The synthesized AuNP's were evaluated to show excellent catalytic reduction activities of nitrophenols to aminophenols in presence of $NaBH_4$. The *Magnusiomyces ingens yeast cells* were reported as an efficient biofactory for eco-friendly preparation of metal nanoparticles [63].

*Synthesis of nanoparticles using Fungi:*

Fungi are eukaryotic micro-organisms which have metal bioaccumulation and detoxification ability through enzymes or structural proteins binding by extracellular or intracellular mechanism. These are used for ecofriendly preparation of metal nanoparticles of gold (Au) and silver (Ag) mainly. The process has some advantages such as easy to handle biomass and low cost and. However, similar to yeast manipulation of genetic material of eukaryotic organisms is difficult as compared to prokaryotes. Tyagi et al. prepared silver (Ag) nanoparticles (NP's) using *Beauveria bassiana* an entomopathogenic fungus. The methodology was identified as non-toxic, simple and rapid in vitro method of extracellular silver (Ag) nanoparticles (NP's) preparation. The AgNP's so obtained reported to have different shapes (triangular, circular, hexagonal) with size range from 10.0 to 50.0 nm. They reported 6.0 as optimal pH and 25 °C as optimal temperature for process. The synthesized nanoparticles were tested for their antibacterial activity against *Staphylococcus aureus, Escherichia coli* and *Pseudomonas* aeruginosa [64]. Molnar et al. carried out ecofriendly preparation of gold (Au) nanoparticles (NP's) by using various thermophilic fungi strains. They applied and compared three different techniques the extracellular fraction, the autolysate of the fungi or the intracellular fraction of 29 thermophilic fungi stains. The AuNP's so obtained were reported to have size range from 6.0 nm to 40.0 nm and the size distribution was found to be dependent on fungi strain and the conditions of experiment

[65]. Li et al. investigated fungus mediated green synthesis of silver (Ag) nanoparticles (NP's) by using *Aspergillus terreus* culture supernatants. The precursor Ag(I) aqueous solution was bioreduced by fungus to Ag(0) at ambient conditions in few hours. The AgNP's so obtained were reported as spherical in shape with size from 1.0 to 20.0 nm. The nicotinamide adenine dinucleotide (NADH) was identified probable candidate for bioreduction of metal ions and formation of AgNP's with enzyme-mediated extracellular mechanism. They investigated the synthesized Ag NP's for their antimicrobial activity against bacteria and fungi [66].

*Synthesis of nanoparticles using Algae:*

Algae are often referred as bionanofactories for synthesis of inorganic (metal & metal oxide) and organic (poly-ε-lysine, chitosan & quaternary ammonium compounds) nanoparticles. The living and dead algal biomass can be utilized to produce nanoparticles. Sathiyaraj et al. studied green biosynthesis of platinum (Pt) nanoparticles (NP's) using aqueous extracts of red algae Halymenia dilatata (Hd). The synthesized Hd-PtNP's were found to be stable and well dispersed having pure crystalline structure, spherical shape, mean size of 15 ± 1.7 nm and the zeta potential of -19.9 mV. The phytochemical constituents present in aqueous extracts of Hd were identified as capping and reducing agents for formation of Hd-PtNP's. They evaluated antibacterial, antioxidant, and anticancer activity of Hd-PtNP's with notable activity and suggested these NP's as for potential pharmaceutical and biomedical applications [67]. Colin et al. investigated algae mediated ecofriendly synthesis of gold (Au) nanoparticles (NP's) by replacing conventional reducing reagents (alkyitrimethylammonium halides) with biocompatibles aqueous *extracts of Egregia sp*. The algal extracts which acts reducing as well as stabilizing agent forms a shell around the Au NP' s which provides biocompatibility. They optimized various parameters affecting particle size distribution to obtain narrow Au NP's. The Au NP's so obtained were recommended for biomedical applications [68]. Gonzalez et al. synthesized gold (Au) nanoparticles (NP's) via algae mediated green synthesis method. The aqueous extracts of brown algae macroalgae *Cystoseira baccata* (CB) were used to produce gold nanoparticles (AuNP's) in a fast, green and one-pot synthetic route. The AuNP's so obtained were reported to be polycrystalline, spherical with mean diameter of 8.4 ± 2.2 nm. They investigated cytotocity of AuNP's with mechanism elucidation and reported to have strong cytotoxicity against cancerous cells (Caco-2 & HT-29) without affecting healthy cells via extrinsic and mitochondrial pathways. The AuNP's were recommended for treatment of colon rectal cancer [69].

*Synthesis of nanoparticles using Plants:*

The plant extracts can be extensively used for synthesis of metal nanoparticles of gold and silver by reduction of aqueous solution of metal ions. The metal NP's are synthesized by rapid extracellular reduction process. Tellez-de-Jesus et al. synthesized encapsulated Gold (Au) and silver (Ag) nanoparticles (NP's) by using *Argemone mexicana L*. extracts. The Au and silver Ag NP's so obtained were investigated for antibacterial activity and reported to show excellent results against antibiotic resistant bacteria under practical environment (hospital). They evaluated that this methodology can be used to produce highly efficient antibiotics in future [70]. Patil et al. investigated green synthesis of silver (Ag) nanoparticles (NP's) using aqueous seeds extracts of *Cuscuta japonica*. The Ag NP's so obtained were reported spherical in shape with MPS (mean particle size) 73.22 ± 3.55 nm. The synthesized Ag NP's Ag NPs wer reported to show excellent antioxidant activity (scavenging free radical) and antibacterial activity against *Bacillus subtilis, Staphylococcus aureus* and *Escherichia* coli [71]. Mallig Arjuna Rao et al. synthesized zinc oxide (ZnO) nanoparticles (NP's) using hot plat combustion method (HPCM) from *Camellia sinensis extract.* The ZnO NP's so obtained were reported to have spherical rod shape with size range of 10-20 nm. They investigated the synthesized ZnO NP's for photocatalytic degradation of MO (methyl orange) dye with 80 %

efficiency and antibacterial activity. Better antibacterial efficiency was reported for *Escherichia coli as compared to Staphylococcus* aureus [72].

*Synthesis of nanoparticles using Viruses:*

Viruses can be used for bottom-up preparation of metal sulphide nanoparticles (PbS and CdS NP's) due to their inorganic material nucleation and assembling ability. The peptides in viruses were identified as probable agents for nucleation and assembling. Shenton et al. investigated Tobacco Mosaic Virus (TMV) mediated synthesis of silica (Si), iron oxide ($Fe^{2+}$ & $Fe^{3+}$), lead sulphide (PbS) and cadmium sulphide (CdS) nanoparticles. They evaluated the internal and external surfaces of the protein with repeated patterns of charged amino acid residues which serve as sites for nucleation of nanoparticles [73]. Mao et al. investigated M13 bacteriphase virus mediated green biological synthesis of zinc sulphide (ZnS), cadmium sulphide (CdS) single crystals and chemically ordered CoPt and FePt freestanding nanowires. The capsid peptides selected by an evolutionary screening process were reported to provide control on size, composition and phase during nucleation. The desired peptide template can be prepared for specific semiconducting and magnetic materials by incorporating specific nucleating peptide in to generic scaffold [74]. Scibilia et al. synthesized silver nanoparticles (AgNP's) by self-assembly approach using M13 P9b phage clone, specific for Pseudomonas aeruginosa. They investigated various parameters like influence of pH of medium and different ions on self-assembly of nanoparticles. The electrostatic interactions of phage pVIII major capsid proteins and the silver particles was reported as probable mechanism of nucleation. The AgNP's so obtained were reported useful for advanced biosensing and targeted gene delivery (TGD) and drug delivery (TDD) [75].

Table 3. Biological methods for NP's synthesis with applications.

| S.No. | Technique | Type | Applications |
|---|---|---|---|
| 1 | Bacteria Mediated Synthesis | Bottom-up | Metal NP's |
| 2 | Yeast Mediated Synthesis | Bottom-up | Metal NP's |
| 3 | Fungi Mediated Synthesis | Bottom-up | Metal NP's |
| 4 | Algae Mediated Synthesis | Bottom-up | Metal NP's |
| 5 | Plant Mediated Synthesis | Bottom-up | Metal and metal oxide oxide NP's |
| 6 | Virus Mediated Synthesis | Bottom-up | Metal and Metal sulphide NP's |

**Conclusion:**

The nanoparticles research has become a multidisciplinary area of interest due to their diverse applications and unique physico-chemical properties. Many techniques have been developed in last couple of decades for synthesis of nanoparticles having different morphology shape and size. The nanoparticles Synthesis mainly includes: chemical, physical and biological method. The chemical methods can be used for synthesis of pure nanoparticles at large scale but high cost is the only limitation. The physical methods are low cost but yields non uniform nanoparticles while biological methods provide green synthetic routes.

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