AN OVERVIEW OF CLASSIFICATION, SYNTHESIS AND CHARACTERIZATION OF NANOMATERIALS

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ABSTRACT: Nanotechnology is the branch of science that deals with particles ranging from 1 to 100nm. Nanoparticles become technically advance over their parent material due to their adaptable characteristics and enhanced performance. They are mostly synthesized by reducing metal ion into uncharged nanoparticles by using hazardous reducing agents. In recent years, green technology for nanoparticles synthesis has received great attention due to its efficiency, less toxicity, safety and high productivity. Green synthesized particles at their nanoscale range are commercially and economically very beneficial for the environment. Moreover, this paper will discuss the nanoparticles, its types, synthesis methods, applications and prospects.

Keywords: nanoparticles, classification, synthesis, characterization, metals.

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INTRODUCTION

Nanotechnology evolved as an achievement in the field of science in the 21st century. Nanoparticles possesses promising applications in various fields like environment, biotechnology, agriculture, medicines, food, biomedical, etc. The most important function is the interaction of atoms with complex molecules by advanced methods may be in vitro or in vivo efficacy (McNeil, 2005). Nanotechnology is a new and advanced level of the invention whose need is increasing globally, socially, and economically (Adlakha-Hutcheon et al., 2009).

The nanoparticles are becoming more prevalent in industrialized procedures, and medical goods, so it is critical that the employees and end-users should be safe from harmful NPs Sharifi et al., 2012). Green synthesis of nanoparticles is easy, cost-effective, environment friendly, more repeatable, and has well-defined physicochemical characteristics. Maximum macro-molecules and cellular structures can generate NPs in response to environmental circumstances and sample treatment. As recently reviewed, the capacity of biological structures (yeast, fungus, plants, algae, and bacteria) to generate NPs has been observed in an extensive range (Duran & Marcato, 2012, Durán & Seabra, 2012, Devatha and Thalla, 2018). Nanoparticles oftenly exhibit distinguished size-dependent characteristics, might be due to their small size and colossal surface area (Guo et al., 2013).

Types of nanomaterials

Nano-cage: Nanomaterials were first described in 2002 which comprised of resonating central and spongy wall with the metallic nanoparticles in them. Their size range from 15 to 150 nanometers (Sun et al., 2002). They can be utilized as multi-photon luminescence tracers, contrast mediators for photoacoustic imaging and multimodal imaging, photothermal mediators for target killing of malignant matter, and drug distribution automobiles for intelligent discharge in reaction to peripheral incentives that are near-infrared waves (Xia et al., 2011).

Nano-crystals: Nano- crystal are pure crystals with sizes in the nanometer range surrounded by a thin coating of surfactant (Park et al., 2007). The synthesis strategy has been developed for fabricating singly dispersed nanocrystals with numerous interactions and possessions using a wide range of materials such as noble metals, Semi-conductors, electrically charged magnets, scarce fluorescent, therapeutic, organic photoelectric conductive polymers (Suryanarayana and Koch, 2000).

Nano-belt: A nano-belt is a thin, flat sheet of ribbon-like structures with a dimension of 30–300 nm. The production of ultra-long nano belts can be distinguished by a trapezoidal cross-section and very well crystallographic surfaces (Chen et al., 2016). Lanthanum hydroxide nano-belts can be made with a composite-hydroxide-mediated synthesis technique. Nano-belts have a significant impact on self-powered nano gadgets and systems. They can be used in FETs, high-sensitivity gas and bioelectronics, attenuators, and linear actuators with nanoscale resolution, among other things (Wang et al.,2007).

Nanofibers: These are two-dimensional fiber constructed with a diameter of less than 100 nanometers. Electrospinning is the most widely used process for producing NFs from a large range of polymeric materials, transition metals and oxides of metal, organic compounds based on carbon, highly ordered heterodimers, and other materials (Teo and Ramakrishna, 2006). Other approaches for manufacturing Nanocomposites include emulsified polymers, assembly, molten blasting, and phase's segregation. They can be used in submersible pumps, implanted devices, biomaterials, medication delivery mechanisms, and electrical equipment (Ellison et al., 2007).

Nanoparticles (NP): Nanoparticles are defined by IUPAC as particles of any shape with diameters between 1.109 and 1.107 nm. (NP). They cab be synthesized by various methods (Luo et al.,2006). NPs can be utilized for several purposes, encompassing biological applications (nanosensors, synthetic biology, pharmaceutical administration, biological controllers, micro/nano technologies, and so forth), electrical and optical devices, the food service industry (Joye and McClements, 2014).

Nanotubes (NT) and Nanorods (NR): A nanotube (NT) is a tiny tube with a diameter measured in nanometers (often 100 nm). The majority of the nanotubes are vacuous. Nano Rods are rigid in structure having parameters ranging from 1 to 100 nm (Dai, 2002). Electric discharging, plasma treatment, and physical vapor deposition methods are some of the processes utilized to manufacture conductive nanostructures and nanorods (CVD). Another chemical technique for creating soft nanotube structures is emulsion polymerization (Volder et al.,2013). Nanotubes have been identified as potential superconductors and are being used in medicinal fields (Qi et al., 2016).

Nanowires: Nanowires can be made in two ways as by top-down and bottom-up approaches (Schmidt et al., 2009). Nanowires can be made by optoelectronic chemical vapor deposition, and physical vapor deposition methods. As nanowires have excellent electrical and optoelectronic properties, so they may be utilized to make p–n junctions, semiconductors, photovoltaic cells, and detectors (Majumdar et al.,2013).

Quantum dots (QD): Quantum dots are semiconducting nanocrystals that are small in size and exhibit fundamental mechanical characteristics. The optical and electrical properties of these are strongly size-dependent. Within the quantum dot volume, there can be as few as 100 to 1000 [narrow space (1/6-em) 1000 atoms, with a diameter of 10 to 50 atoms (Dhand et al., 2015). Quantum dots can be designed by organometallic technique, which consists of 3 main parts: antecedents, natural lubricants, and fluids (Seth et al., 2016). They can be used in optics, light-emitting semiconductors, assays, biosensors, and medical diagnostics because of their excellent optical and electronic features (Jamieson et al., 2007).

Synthesis of nanomaterials: Nanomaterials have gotten a lot of interest in the last decade because of their wide range of uses. The subject of fabrication of nanoparticles has shown to be highly active. Numerous procedures are used, which include gas convection, chemical vapor deposition synthesis, structural demoralization, chemical coagulation, Sol-Gel methodology, electrochemical methods, transmission electron microscopy, ionized cluster beam, solvent metal - ligand source, single unit consolidation, vapor deposition, and gas agglomeration, coagulation / flocculation in the existence of targeting ligands, reaction in surfactant micelles, and others. The method produces nanomaterials are different for different materials (Rajput, 2015).

The top-down strategy is overwhelmingly popular in the business world for the manufacturing of many synthetic or artificial materials, with the electronics industry representing an improved version, in which characteristics of metal-oxide-semiconductor transistors (MOSFETs) are engrained onto a silicon dioxide substrate using selective laser sintering, a fiber lasers methodology (Wang et al.,2016).

Nanoparticles of acceptable and regulated composition are sought for industrialization in a variety of sectors. There are two fundamental techniques that are typically used to manufacture nanoparticles:

(1) A top-down method in which production is started with the corresponding bulk materials, which are released gradually, resulting in the production of microscopic nanoparticles. Top-down approaches for commercial processing of nanoparticles include selective laser sintering, electromagnetic lithography, grinding processes, electrodeposition, ion and laser ablation, and others.

(2) A bottom-up approach where the particles of matter combine or integrate to form a diverse spectrum of nanoparticles. Bottom-up methods include self-assembly of copolymerization of monomers/subunits and pharmacological or electromechanical processes. Nanostructuring condensation, hydrate polymerization, beam decomposition, chemical vapor deposition (CVD), fusion or flare jet production are all examples of bio-assisted manufacture (Daraio and Jin, 2012).

In general, nano Particles manufacturing procedures can be distributed in three groups

- physical techniques
- chemical techniques
- Bio-assisted techniques

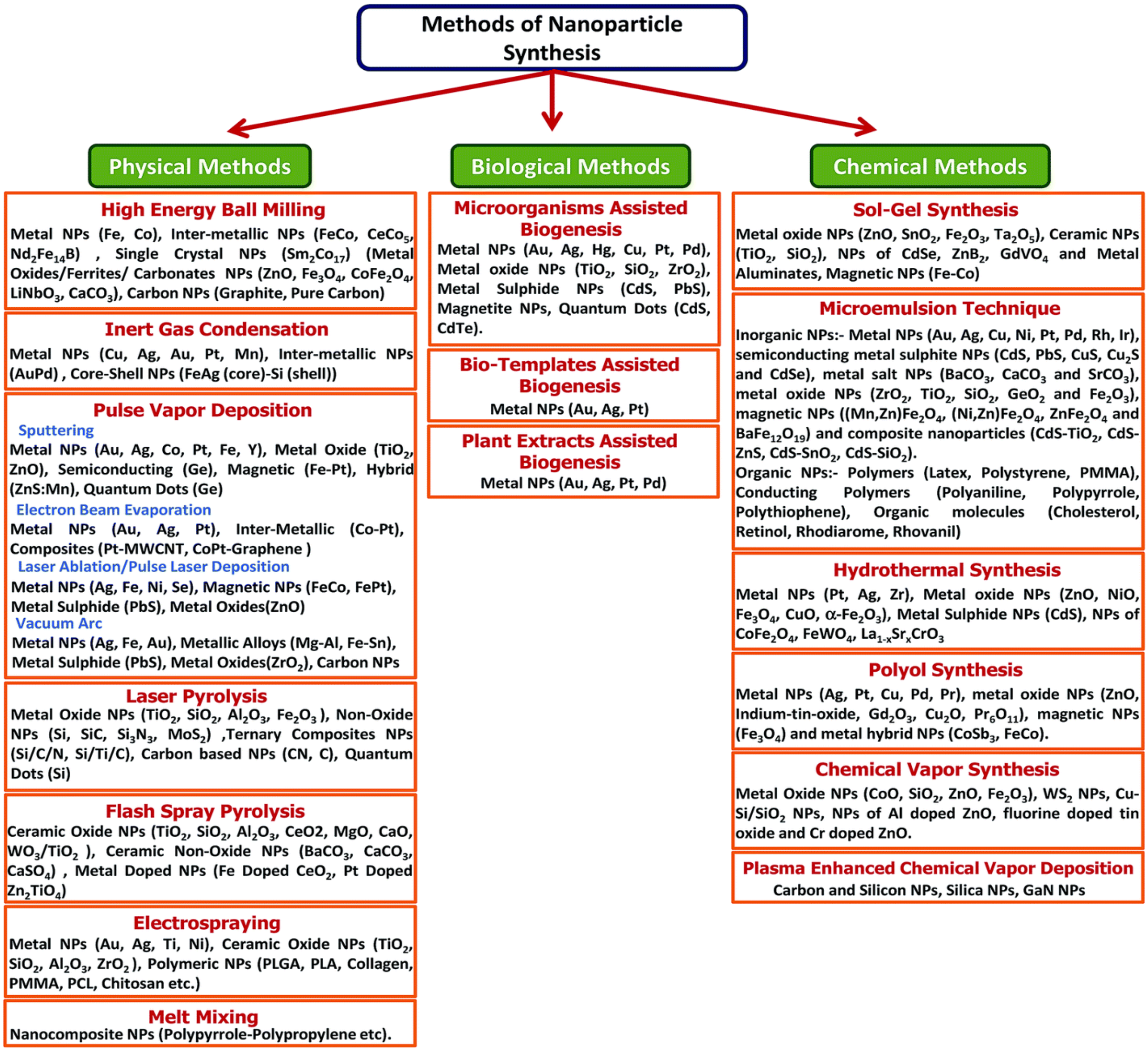

Figure: 2.1 Methods of nanoparticles synthesis

Synthesis of nanomaterials using physical techniques: Mechanical pressure, increased radioactive isotopes, heat energy, or electrical potential are used in physical techniques to cause substance erosion, melting, vaporization, or precipitation. Some techniques, which predominantly employ a top-down method, are advantageous even though they are liquid and produce homogenous narrow size distribution nanoparticles. Similarly, the high quantities of waste created during synthesizing reduces the cost-effectiveness of physical techniques. Physical processes for creating nanoparticles include high-energy ball milling, laser ablation, electro spraying, shielding gas precipitation, mechanical physical vapour deposition, laser decomposition, lightning magnetron sputtering, and molten mixing.

High energy ball milling (HEBM): John Benjamin in 1970, introduced High energy ball milling, to produce oxides scattering enhanced composites that are strong enough to withstand high heat and compression, is a dependable and fuel synthesis technique for generating Nanoparticles of diverse lengths and diameters (Xing et al., 2013).

In the HEBM process, the acceleration of the moveable rollers is transmitted to the ground substance. As a consequence, one‘s chemical properties are severed, and the ground components are prolapsed into finer molecules with recently established substrates. Rotary media, milling speed, ball-to-powder relative density, milling type (dry or wet), high energy ball mill type (vibrator mill, celestial mill, etc.), milling ambiance, and milling timeframe all influence the extent of radiant energy seen between balls and the substance during the procedure, influencing the external and surface morphology of the end product. The HEBM mechanism is characterized as a thermomechanical fabrication technique since it may incorporate exceptionally high local heating (>1000 °C) and increased pressure e. g. multiple G Pa (Salah et al., 2011).

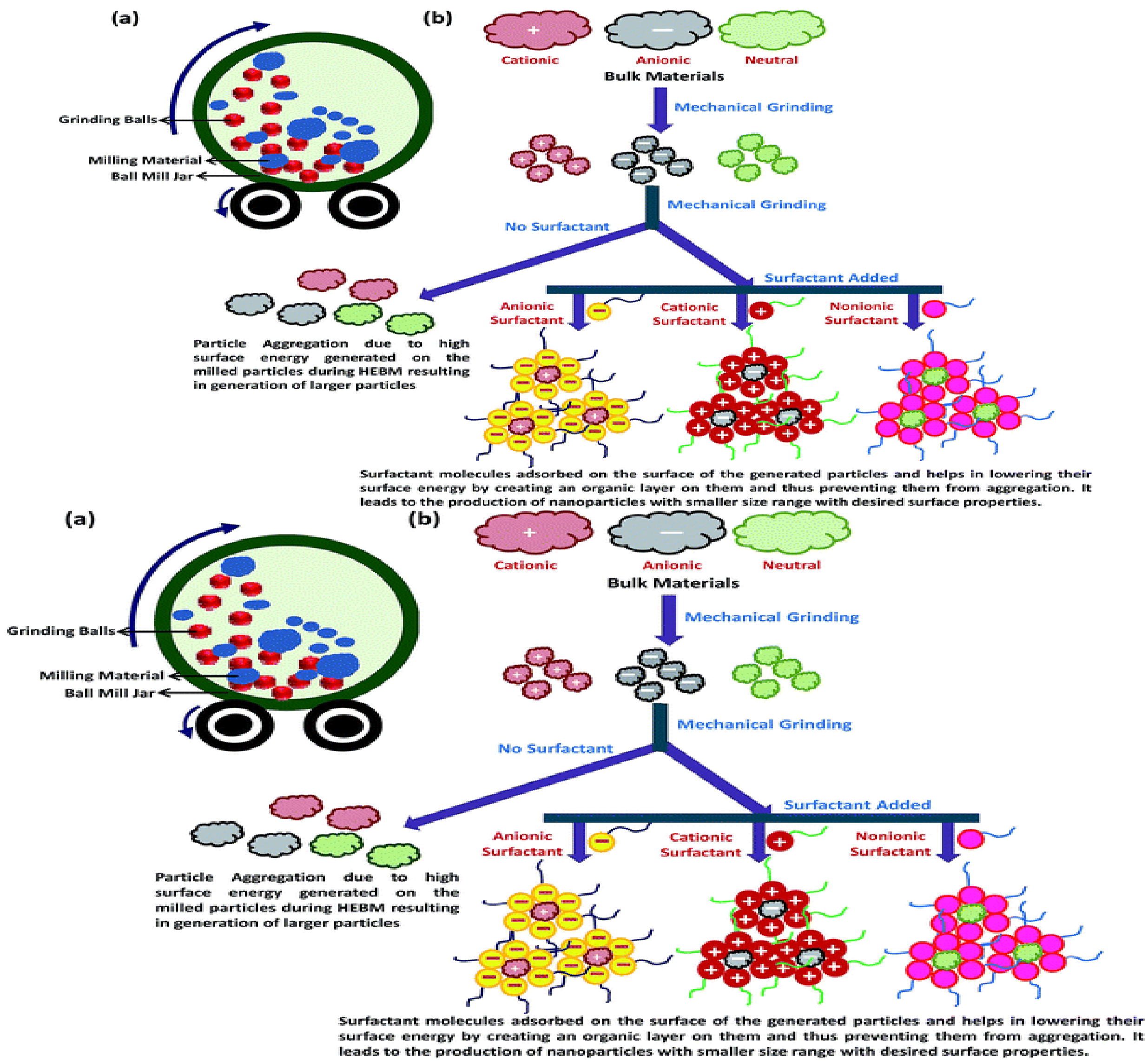

Figure: 2.2 High Energy Ball Milling Techniques

Inert gas condensation (IGC): Inert gas condensation is a simple process for producing nanoparticles that employ inert gases (such as He or Ar) and a compressed N_2 sample container. The vaporized ingredients are transported employing inert gases and condensed onto a compressed nitrogen surface. We used this approach to manufacture manganese nanoparticles and used the IGC (inert gas condensation) process to analyze the effect of tempering on the morphology of the implanted nanoparticles. (Ward et al.,2006).

IGC was revealed to be an extremely effective method of generating elevated silver and platinum nanoparticles. A sputtering depositing technique was used to generate those nanoparticles and also described the formation of Ag nanoparticles that used the IGC technique, and observed also that evaporated temperatures (ranging from 1123 K and 1423 K), as well as inert gas (He) pressure, had a substantial impact on the morphological, crystalline structure, and dimension variation i.e. varying between 0.5 and 100 Torr (Raffi et al.,2007).

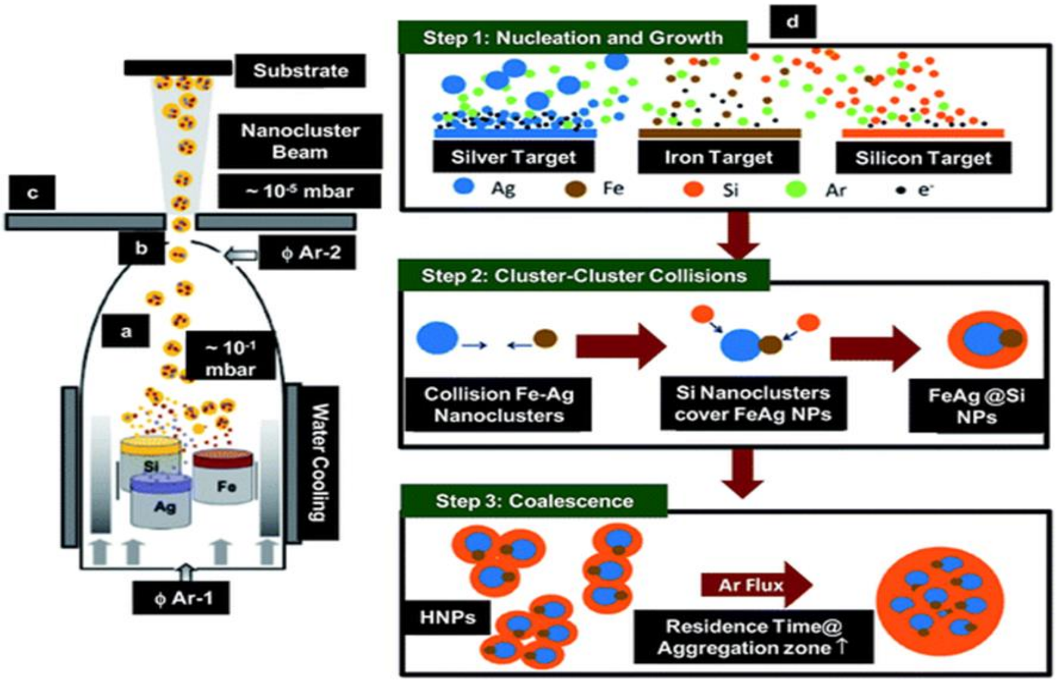

Figure: 2.3 Inert Gas Condensation

Physical vapor deposition (PVD): Physical vapor deposition is a phrase used to describe a collection of methods to produce nanoparticles and implant thin films that are generally a few nm to many micrometers wide. PVD is a sustainable arc discharge technique that consists of three fundamental aspects:

(1) Solid source vaporization

(2) Vaporized material conveyance

(3) Nucleation and growth to produce thin films and NPs.

For the synthesis of NPs, the most often utilized PVD methods include (a) plasma sputtering, (b) electron beam evaporation, (c) pulsed laser deposition, and (d) vacuum arc technique. The schematic figure is given below:

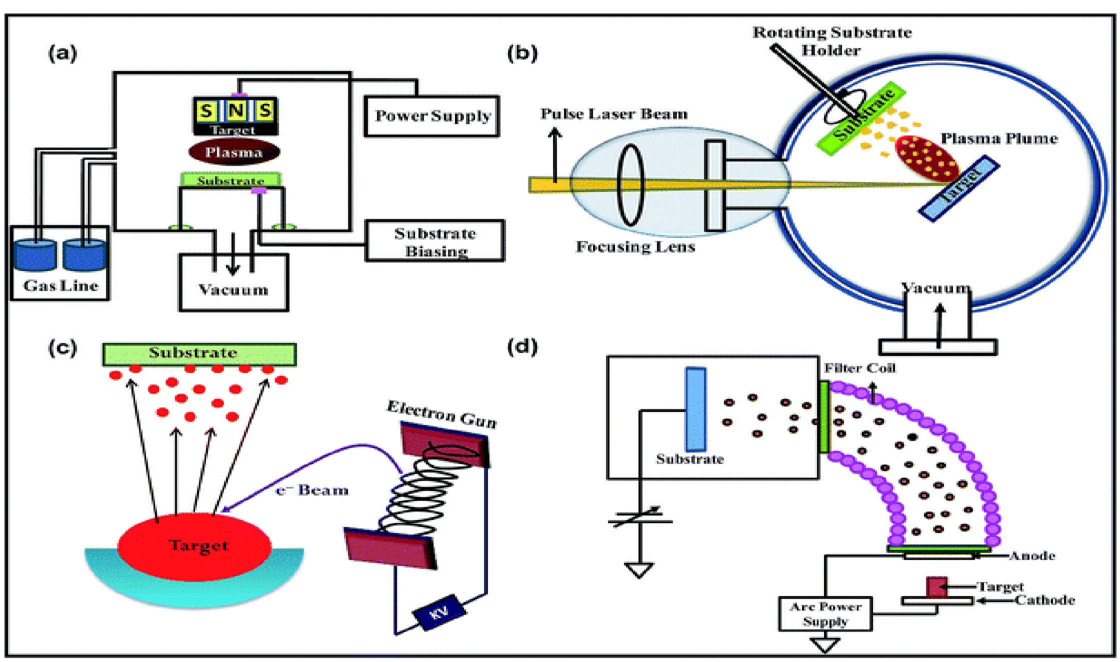

Figure: 2.4 Schematic Diagram of Physical Vapour Deposition

Chemical methods for the synthesis of nanomaterials

Sol-gel method: The sol–gel manufacturing approach has 2 kinds of components 'sol,' which is a colloidal suspension of granular suspended particles, and 'gel,' which seems to be polymeric materials comprising liquid. As a consequence, such a mechanism generates 'sols' in the liquids, which subsequently combine to create a network of suspended particles or web polymerization. The fundamental phases of the sol–gel procedure are hydrolyzed and condensed, whereby the latter consumption of water separates the molecules of the originator, while the latter stage is the first stage in the development of the gel state. This one is proceeded via condensing, which leads to the development of nanostructures, whereupon the additional water is evaporated to predict the overall strength of the sample (Zha and Roggendorf, 1991).

Microemulsion technique: Microemulsions are stable chemical, morphologically homogenous, optically transparent, and isolated system colloidal carriers consisting of at least three aspects: polar phase (often water), non-polar phase (typically hydrocarbon liquid or oil), and stabilizer. Emulsifiers produce an interfacial layer between both the organic and aqueous phases, lower surface tension between emulsification and the additional phase, and serve as an electrostatic obstruction to protect particles in aggregating (Solanki & Murthy, 2011).

A microemulsion framework comprises narrow-size distribution circular drops (diameters stretching from 600 nm to 8000 nm) with water-in-oil (w/o) or oil-in-water (o/w) based on the emulsifier used. The w/o reverse micellar technique provides an efficient relevant to the target for the nanoparticle synthesis. As shown in the picture following, a reverse micelle is a water-in-oil microemulsion whereby the surfactant's hydrophilic head of the groups creates the wet core and stays within, while the organic tails of the solvent molecules are pointed outwards:

(a) Typical reverse micelle system,

(b) Different steps take part in one microemulsion process and

(c) Sequence of reactions involved in the 2 micro emulsion nanoparticles synthesis.

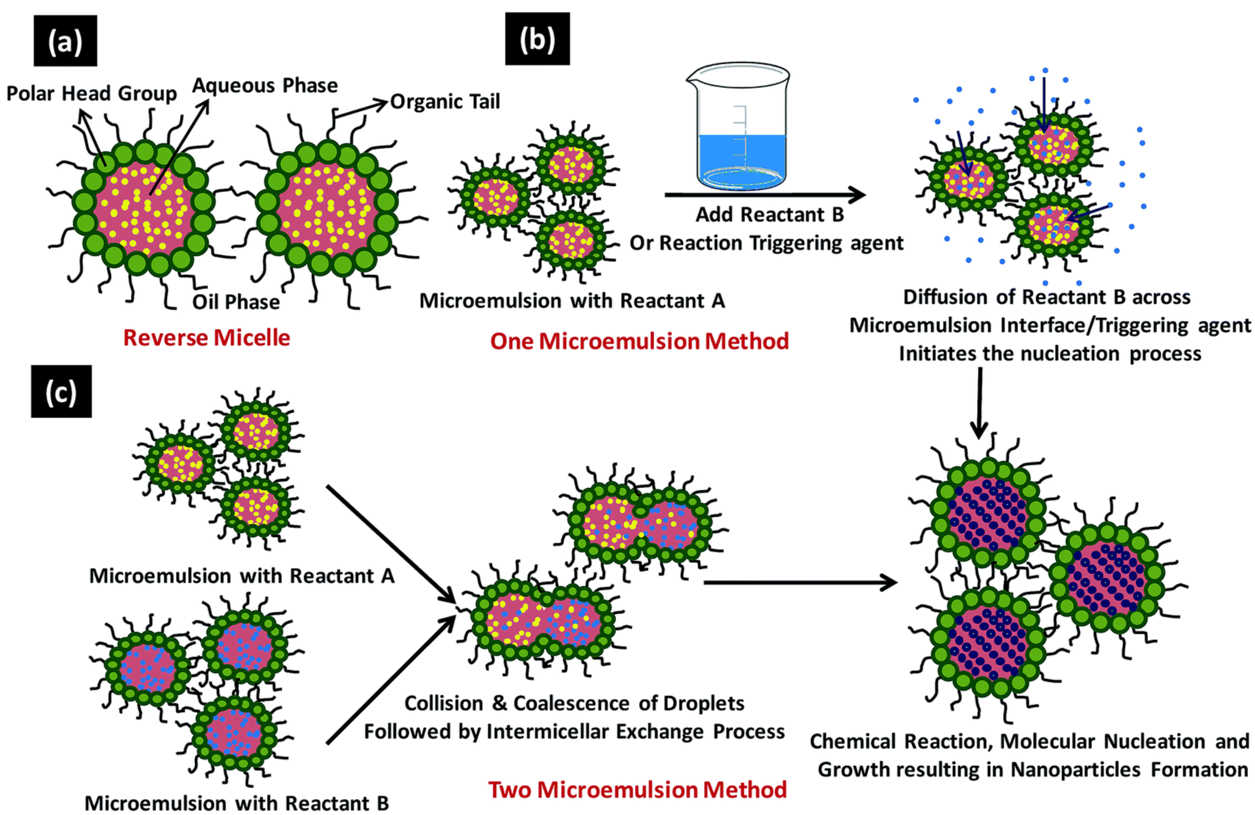

Figure: 2.5 Two Micro emulsion Method

Hydrothermal synthesis: This process is used to create oxide of metals, ferric oxide, and lithium iron phosphate nanoparticles by maintaining hold across particle characteristics by changing the properties of relatively close or extreme water at different temperatures and high settings. It is possible to accomplish it in two ways: batching hydrothermal and continuously hydrothermal. The latter can carry out a system with the required proportion stages, but the latter can generate a quicker rate of response in less time (Hayashi & Hakuta, 2010).

Polyol synthesis: The polyol procedure is a metal-containing molecule synthesis technique that employs poly (ethylene glycol) as that of the reaction mixture, acting as a solvent, capping agent, and interfacial operator simultaneously, with soluble stabilizing or protective agents (Rahman & Green, 2009). This electrochemical method was used to create a diverse range of metallic-based Nanoparticles (Ag, Pt, Pd, Pr, Cu), metallic oxides nanoparticles (ZnO, indium-tin-oxide; ITO, Gd_2O_3, Cu_2O), magnetically nanoparticles, and metal hybrid NPs. Researchers utilized the polyol method to create Pt nanoparticles with varying $NaNO_3$ and H_2PtCl_6 molar ratios. They also reported tailoring the dimensions, surface characteristics, and crystalline structure of nanomaterials by decelerating the reduction of Pt(II) and Pt(IV) species (attained by ethylene glycol) facilitated by variable concentrations ranging of nitrate ionic species, that have a high binding affinity stable compounds with Pt(II) and Pt(IV) (Herricks, Chen, & Xia, 2004). The morphology advancement of oxides of nanoparticles in polyol processes using two different polyols.

Chemical vapor deposition (CVD) & chemical vapor synthesis (CVS): Chemical vapor deposition (CVD) and chemical vapor synthesis (CVS) are two processes for depositing solid coatings from the vapor stage using chemical reactions that occur at enormously elevated temperatures. Ultra-minute particles can be discovered in thin films made by the CVD process under specific conditions.

As a consequence, whenever the conventional methods are adjusted such that the CVD technique requires nanoparticles instead of thin metallic sheets, the altered technique is known as CVS. The phrases chemically vapor reactivity (CVR), chemical vapor precipitate (CVP), and chemical vapor condensation (CVC) all refer to the same phenomenon (CVC). The three types of predecessors (solid, liquid, and gas) are produced in the furnace as vapors under circumstances that need molecules would go through the crystallization processes.

CVS additionally employs a variety of substrates to produce multi-component or hybridized Nanoparticles. Erbium (Er) has just been merged into Si nanoparticles by the use of substrates such as organometallic Erbium and di-silane compounds, and composites Nanoparticles are created by enclosing one material inside another (e.g. silicon tetrachloride in reaction with sodium chloride that consequence in the production of sodium chloride-enclosed silicon particles (Vallejos et al., 2011).

Bio-assisted methods for the synthesis of nanomaterials: To manufacture and create NPs, bio-assisted methods, also called biosynthesized or green synthesis, provide environmentally approachable, low-toxicity, low-cost, and economical approaches. In the manufacture of metal and metallic oxide nanoparticle synthesis, these approaches use natural systems such as bacteria, fungus, viruses, yeast, actinomycetes, plant extracts, and so on. Three types of bio-assisted approaches can be distinguished:
(i) Microorganism-based biogenic production
(ii) Biomolecules as templates for biogenic production
(iii) Herbal extract-based biogenic synthesis

Biogenic Synthesis Using Microorganisms: Prokaryotic microbes, actinomycetes, fungi, phytoplankton, and yeast are among the bio-reactors used to manufacture nanoparticles. This technique for generating a diverse spectrum of NPs required significant scientific effort (Ag, Au, Pd, TiO2, CdS, etc.). Microscopic creatures gather target ions in their atmosphere and employ enzymes generated by cellular processes to transform the metallic ions into the constituent of metal.

Nanoparticles can be distinguished as intracellular or extracellular based on where it is produced. In the intracellular method, metal ions are carried inside the microbiological cell to produce nanoparticles in the presence of catalysts. Extracellular nanoparticle synthesis necessitates the capture of metallic ions in the extracellular environment as well as ion reduction in the presence of protein (Zhang et al., 2011).

Biomolecules as templates to design nanoparticles: To make NPs, several biomolecules such as nucleic acids, membranes, viruses, and diatoms were employed as templates. DNA is recognized as a good biomolecular template with a great affinity for transition metal ions. It was demonstrated that a DNA hydrogel could be produced and crosslinked before transition metal ions (e.g. gold, Al (III) metal ions) were merged into DNA macromolecules, consequential in the manufacture of Au nanoparticles. A reduction of Al(III) occurs, resulting in the creation of Au atoms and metal clusters, which evolve into Al nanoparticles on the DNA chain (Zinchenko, Miwa, Lopatina, Sergeyev, & Murata, 2014).

Characterization of nanoparticles: Characterization is the study of many facts like the structure of nanoparticles, the material from which nanoparticles can be formed, and many other properties. There are different techniques for the characterization of nanoparticles. These techniques are atomic force microscopy, particle size analysis, scanning electron microscopy, X-ray diffraction, transmission, electron microscopy Fourier transform infrared spectroscopy, X-ray photoelectron spectroscopy Raman spectra, etc. SEM and TEM are the essential techniques. SEM depends upon the electron scanning method. TEM depends on electron transmittance properties and XRD is based on the structural properties.

The characterized biosynthesized nanoparticles by SEM and XRD exposed the crystalline structure and spherical geometry. XRD and SEM analysis indicates the magnitude of the particles was found to be about 60-70 nm in range. The size of the particle is approximately 100 nm measured by the PSA of synthesized Aluminum oxide powders. The triumphant structure of Aluminum oxide nanoparticles was confirmed by retaining SEM-EDX, XRD, and PSA analysis.

The characterization of synthesized Al_2O_3 nanoparticles UV-Vis spectroscopy was used. The crystalline nature of the biosynthesized nanoparticle was exposed by X-ray Diffraction (XRD) and confirms the size of the nanoparticle as 25.7nm. EDX with SEM was used to confirm the shape, composition, and size of green-manufactured Al_2O_3 nanoparticles.

Different techniques are used for characterization. For crystalline size X-ray diffractometer (XRD), for geometrical studies Field Emission Scanning Electron Microscope (FESEM), to analyses the absorption patterns UV-Visible spectroscopy (UV-Vis), for analyzing the functional groups Fourier Transform Infrared (FTIR) spectroscopy is used which take part in the reaction. The development of Aluminum oxide nano-particles was checked by comprehensive characterization techniques. Fourier transform Infrared (FT-IR) spectral data analysis is used for the confirmation of the presence of metal oxides and biomolecules. The surface geometry that is of Al_2O_3 particles observed by Scanning electron microscope (SEM) showed the hexagonal-shaped Al_2O_3 crystallites. The formation of pure cubic Al_2O_3 crystalline nanoparticles is shown by X-ray diffraction (XRD). The surface geometry of Al_2O_3 nanoparticles observed by Scanning electron microscope (SEM) showed the hexagonal-shaped Al_2O_3 crystallites.

Conclusion: Nanoparticles possess potential applications in various fields like energy storage, catalysis, medicine and electronics. However, major challenge is the synthesis method. As the size and shape of nanoparticles have high impact on its properties. The chemical methods can cause toxicity, so green method is becoming popular because of its harmless nature. There is ongoing research on metal NPs in medicine, including cancer therapy and drug delivery.

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