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Preparation of Nanoparticles

Takalani Cele

Abstract

Innovative developments of science and engineering have progressed very fast toward the synthesis of nanomaterials to achieve unique properties that are not the same as the properties of the bulk materials. The particle reveals interesting properties at the dimension below 100 nm, mostly from two physical effects. The two physical effects are the quantization of electronic states apparent leading to very sensitive size-dependent effects such as optical and magnetic properties and the high surface-to-volume ratio modifies the thermal, mechanical, and chemical properties of materials. The nanoparticles' unique physical and chemical properties render them most appropriate for a number of specialist applications.

Keywords: nanoparticles, physical methods, chemical methods, synthesis, metal nanoparticles

1. Introduction

Several methods have been developed to produce metal nanoparticles. Two synthesis approaches have been identified that is top-down and bottom-up approach. Top-down methods comprise of milling, lithography, and repeated quenching. This approach does not have good control of the particle size and structure. Bottom-up method is the approach that is mostly used by scientists in the synthesis of nanoparticles as it involves building up a material from bottom: atom-by-atom, molecule-by-molecule, and cluster-by-cluster [1, 2]. Several chemical routes have been identified to synthesize the colloidal metal nanoparticles from different precursors using chemical reductants in solvents (aqueous and nonaqueous). The chemical routes that have been studied for various applications include electrochemical method [3], sonochemical method [4], radiolytic [5] and photochemical [6] method.

2. Methods of synthesis of metal nanoparticles

2.1 Chemical methods

2.1.1 *The polyol method*

The Polyol method is a chemical method for the synthesis of nanoparticles. This method uses nonaqueous liquid (polyol) as a solvent and reducing agent. The nonaqueous solvents that are used in this method have an advantage of minimizing surface oxidation and agglomeration. This method allows flexibility on controlling of size, texture, and shape of nanoparticles. Polyol method can also be used in producing nanoparticles in large scale [7].

The polyol process can be taken as a sol-gel method in the synthesis of oxide, if the synthesis is conducted at moderately increased temperature with accurate particle growth control [8]. There are several reports that have studied the synthesis of oxide sub-micrometer particles and these include Y_2O_3 , V_xO_y , Mn_3O_4 , ZnO , CoTiO_3 , SnO_2 , PbO , and TiO_2 [9–16].

The solvent that is mostly used in polyol method in metal oxide nanoparticles synthesis is ethylene glycol because of its strong reducing capability, high dielectric constant, and high boiling point. Ethylene glycol is also used as a crosslinking reagent to link with metal ion to form metal glycolate leading to oligomerization [17]. It has been reported that as-synthesized glycolate precursors can be converted to their more common metal oxide derivatives when calcined in air, while maintaining the original precursor morphology [8].

The polyol synthesis process has also been used for the synthesis of bimetallic alloys and core-shell nanoparticles [18–20]. Yang and co-workers used polyol method to produce icosahedral and cubic gold particles on the order of 100–300 nm by careful regulation of the growth rate for each crystallographic direction [21]. Xia and co-workers reported the production of controlled morphologies such as nanocubes and nanowires by controlling the molar ratio between silver nitrate and PVP [22].

2.1.2 Microemulsions

An emulsion is a liquid in liquid dispersion. A solution of polymers can produce emulsions as it is liquid. Emulsions are divided according to the size of droplet, i.e., macro-emulsions, mini-emulsions, and micro-emulsions [23].

Micro-emulsion synthesis method is widely used for the production of inorganic nanoparticles [24]. When oil and water are mixed, they separate into two phases as they are immiscible [25]. The energy input is required to mix the two phases to create water-oil.

An attempt to combine the two phases requires energy input that would establish water-oil connection replacing the water-water/oil-oil contacts. The interfacial tension between bulk oil and water can be as high as 30–50 dynes/cm and this can be avoided by using surfactants (surface-active molecules). Surfactants contain hydrophilic (water-loving) and lipophilic (oil-loving) groups [26]. The interface can be aligned and established between oil and water by reducing the interfacial tension if there are enough surfactant molecules.

The preparation procedure of metallic nanoparticles in water in oil microemulsion commonly consists of mixing of two microemulsions containing metal salt and a reducing agent, respectively as shown in **Figure 1**.

Brownian motion is formed after the exchange of reactants (collision) between micelles that happens after mixing two microemulsions. Good collisions result into coalescence, fusion, and mixing well of the reactants. Metal nuclei are formed from the reaction between solubilizates. Bönemann et al. reported the formation of zerovalent metal atoms at nucleation stage from reducing a metal salt, which can collide with additional metal ions, metal atoms, or clusters to form an irreversible seed of stable metal nuclei [28].

The growth stage happens around the nucleation point, where successful collision occurs between a reverse micelle moving a nucleus and another one moving the product monomers with the arrival of more reactants due to intermicellar exchange. The morphology and size of nanoparticles are based on the size and shape of the nanodroplets and the type of the surfactant. The surfactant is usually used to stabilize the particle and protect them from proceeding to grow [28].

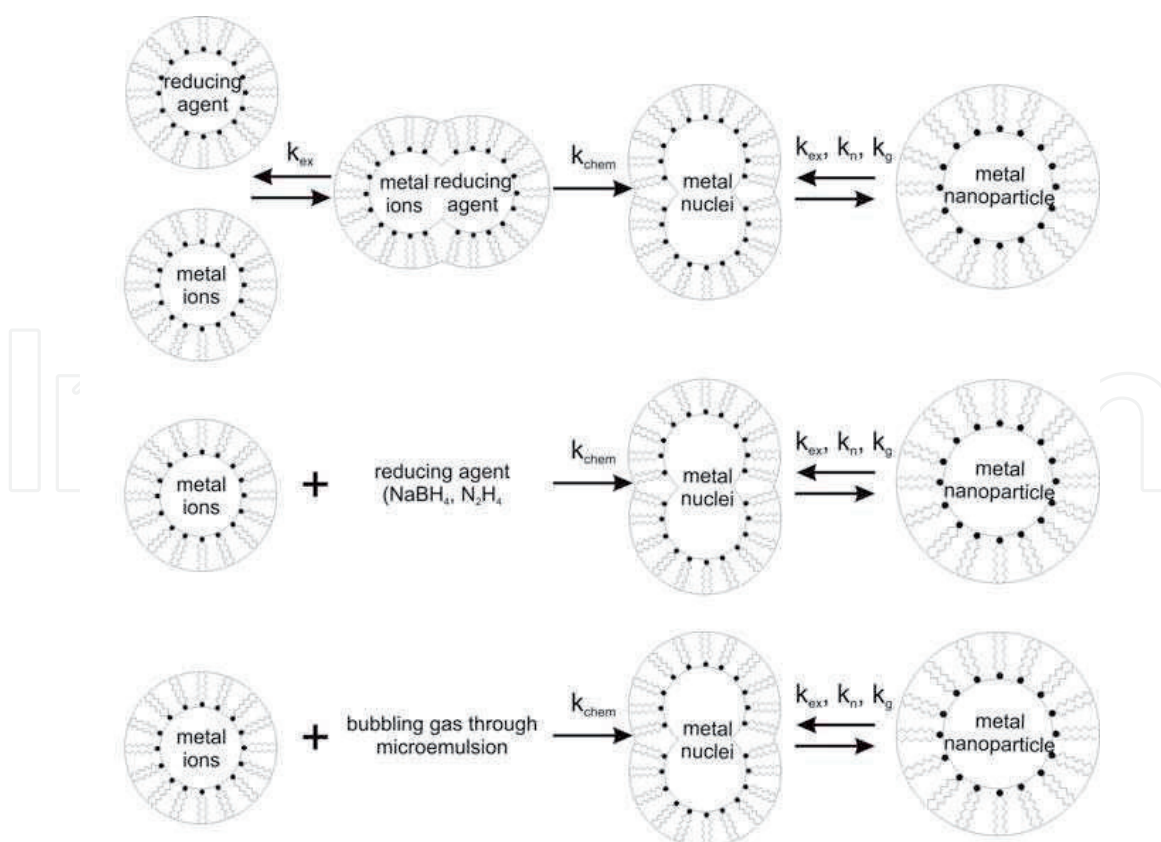


Figure 1. Schematic illustration of nanoparticles preparation using microemulsion techniques: Particle formation steps. K_{chem} is the rate constant for chemical reaction, k_{ex} is the rate constant for intermicellar exchange dynamics, k_n is the rate constant for nucleation, and k_g is the rate constant for particle growth [27].

Wongwailikhit et al. reported the formation of iron (III) oxide, Fe_2O_3 using water in oil microemulsion by combining the required amount of H_2O in a stock solution of Sodium Bis (2-Ethylhexyl) Sulfosuccinate (AOT) in *n*-heptane. The solution was left overnight, then the concentrated Hydroxylamine (NH_2OH) and $FeCl_3$ were mixed into the water in oil microemulsion. Suspension of Fe_2O_3 was filtered and washed with 95% ethanol and dried at $300^\circ C$ for 3 h. The product was spherical, monodisperse nanoparticles with diameter of about 50 nm. The size of particles depended on the water content in microemulsion system. The increase of particles size was achieved with increasing the water fraction in water in oil microemulsion [29].

Sarkar et al. reported the formation of pure monodispersed zinc oxide nanoparticles of different shapes. Microemulsion was composed of cyclohexane, Triton X-100 as surfactant, hexanol as cosurfactant and aqueous solution of zinc nitrate or ammonium hydroxide/sodium hydroxide complex. The molar ratio of TX-100 to hexanol was maintained at 1:4. The microemulsion containing ammonium hydroxide/sodium hydroxide was added to microemulsion containing zinc nitrate and stirred. The nanoparticles were then separated by centrifuging at 15,000 rpm for 1 h. The particles were washed with distilled water and alcohol and dried at $50^\circ C$ for 12 h [30].

Maitra was the first to establish Chitosan nanoparticles by microemulsion technique. Chitosan nanoparticles were prepared in the aqueous core of reverse micellar droplets and crosslinked through glutaraldehyde. Surfactant dissolved in *N*-hexane was also used with chitosan in acetic acid and glutaraldehyde was added in the surfactant at room temperature. The mixture was stirred continuously and nanoparticles were produced [31].

2.1.3 Thermal decomposition

Thermal decomposition also known as thermolysis is a chemical decomposition that is caused by heat. In this method, the heat is required to break chemical bonds in the compound undergoing decomposition and the reaction is endothermic. If decomposition is sufficiently exothermic, a positive feedback loop is created producing thermal runaway [32].

Arshad et al. reported on thermal decomposition of metal complexes of type MLX_2 [$M = \text{Co (II)}, \text{Cu (II)}, \text{Zn (II)}, \text{and Cd (II)}$; $L = \text{DIE}$; $X = \text{NO}_3^{1-}$] by TG-DTA-DTG techniques in air atmosphere. They synthesized nitrate complexes of transition metals with 1,2-diimidazoloethane (DIE) of the general formula $M(\text{DIE})(\text{NO}_3)_2$. The study was conducted by thermoanalytical techniques in static air atmosphere to study the thermal behavior of these complexes and to determine their mode of decomposition. The complexes and ligands decomposed in a two-step process when heated to 740° . Above 740° , the residue was found to correspond with metal oxide. The thermal stability of the complexes increases in the following series: $\text{Co(II)} < \text{Cu(II)} < \text{Zn(II)} < \text{Cd(II)}$ [33].

Patil et al. studied infrared spectra and thermal decompositions of metal acetates and dicarboxylates. The study was done to determine the metal-acetate bonding and the thermal decomposition of lead, copper, and rare earth acetates was studied by means of thermogravimetric analysis and differential thermal analysis. The investigations on decomposition products yielded good results [34].

George et al. reported on the mechanism of thermal decomposition of *n*-Butyl 1 (tri-*n*-butylphosphine) copper (I). This study provided the first easily interpretable example in which succeeding reaction of a metal hydride and its parent metal alkyl was found to be vital in determining the products of a thermal decomposition [35].

Thermal decomposition of bismuth and silver carboxylates was investigated by means of TG, DSC, mass spectrometry, X-ray analysis, and electron microscopy Logvinenko et al. [36]. Non-isothermal thermogravimetric data were used for kinetic studies. All decomposition processes had multi-step character [36].

Ewell et al. investigated nearly pure talc both unheated and after heating at various temperatures ranging up to $1,435^\circ\text{C}$. The research included the measurement of heat effects, weight losses, and changes in true specific gravity occurring on heating talc. There was no change in the crystal structure of the talc heated up to 800°C . At the temperature between 800 and 8400°C , the talc decomposed to enstatite, amorphous silica, and water vapor. At the temperature approximately $1,200^\circ\text{C}$, the enstatite steadily changed to clinoenstatite and the amorphous silica changed to cristobalite approximately $1,300^\circ\text{C}$, giving clinoenstatite and cristobalite as end products [37].

2.1.4 Electrochemical synthesis

Electrochemical synthesis is the synthesis of chemical compounds in an electrochemical cell. The main advantage of electrochemical synthesis over an ordinary chemical reaction is rejection of the potential wasteful alternative half-reaction and the ability to accurately tune the preferred potential [38].

Electrochemical synthesis of silver nanoparticles has been extensively studied in the previous years. The method of electrochemical that was used was based on the dissolution of a metallic anode in an aprotic solvent. The silver nanoparticles that were produced by electroreduction of anodically solved silver ions in acetonitrile containing tetrabutylammonium ranged from 2 to 7 nm. The particle size was obtained by varying the current density. Different types of counter electrodes were used to study the effect of the different electrochemical parameters on the

end particle size. The UV-Vis spectra showed the presence of two different silver clusters [39].

Dobre et al. also reported on the electrochemical synthesis of colloidal silver solutions using “sacrificial anode” technique conducted with a home-built current pulse generator with alternating polarity and a stirrer. Poly (N-vinyl-2-pyrrolidone) (PVP) and sodium lauryl sulfate (Na-LS) were used as a stabilizer and co-stabilizer, correspondingly. Spherical Ag particles with the size approximately 10–55 nm were synthesized. The UV/Vis spectra showed the absorption band at 420 nm, which is the evidence of the presence of Ag nanoparticles. The zeta potential values between –17 and –35 mV suggested a presence of particles covered by stabilizer with a slight agglomeration [40].

More research was done on the electrochemical synthesis of silver nanoparticles in aqueous poly (vinyl alcohol) solution (PVA). PVA is a low price widely used synthetic polymer with properties such as nontoxicity, water solubility, biocompatibility, biodegradability, and excellent mechanical properties. The experiment was conducted at a constant current density of 25 mA cm^{-2} for a synthesis time of 10 min. Silver nanoparticles with an average diameter of $15 \pm 9 \text{ nm}$ were obtained [41].

The electrochemical synthesis of red fluorescent Silicon (Si) nanoparticles stabilized with styrene. Si nanoparticles emit fluorescence under UV excitation, which is great for optics applications, etc. It was found that the liberated silicon particles in ethanol solution interact with styrene, which resulted in the substitution of Si-H bonds with those of Si-C. The developed styrene-coated Si nanoparticles exhibited a stable, bright, red fluorescence under excitation with a 365 nm UV light, and resulted into approximately 100 mg per Si wafer with a synthesis time of 2 h [42].

More investigations were done on the preparation of long-lived silver nanoparticles in aqueous solutions and silver powders using electrochemical method. The produced silver nanoparticles had a size distribution ranging from 2 to 20 nm and the nanoparticles remained stable for more than 7 years. Silver crystals containing agglomerated silver nanoparticles with sizes below 40 nm was found growing on the surface of the cathode [43].

The research was conducted on using electrochemical method to synthesize highly pure silver nanoparticles. This method was used as it is one-step less expensive procedure and easy to control at room temperature and it does not use dangerous chemicals. The experimental setup brought up the oxidation of the anode and reduction of the cathode. The silver nanoparticles synthesized were spherical and had a particle size below 50 nm [44].

Islam et al. explored on the synthesis of platinum nanoparticles by electrochemical deposition method. The particle size was controlled by varying electrolysis parameters and homogeneity of platinum particles was improved by varying the composition of electrolytic solutions. Platinum nanoparticles were deposited on electrode surfaces and the particle sizes were found to be larger than 10 nm and had wide particle size distribution [45].

2.2 Physical methods

2.2.1 Plasma

Plasma method is another method that is used to produce nanoparticles. The plasma is generated by radio frequency (RF) heating coils. The initial metal is enclosed in a pestle and the pestle is enclosed in an evacuated chamber. The metal is then heated above its evaporation point by high voltage RF coils wrapped around the evacuated chamber. The gas that is used in the procedure is Helium (He), which

forms a high-temperature plasma in the region of the coils after flowing into the system. The metal vapor nucleates on the helium gas atoms and diffuses up to a cold collector rod, this is where nanoparticles are collected and they are passivated by oxygen gas (**Figures 2 and 3**) [46].

Classification of plasma methods based on the feeding materials to reactor and also the heating source (electrodeless/ electrode containing), see (**Figures 2 and 3**).

2.2.2 Chemical vapor deposition

The chemical vapor deposition method (CVD) involves a chemical reaction. CVD procedure is mostly used in semiconductor manufacturing for depositing thin films of different materials. The method involves one or more volatile precursors, the substrate is exposed to those precursors that decompose on it and form the desired deposit. The vaporized precursors are inserted into a CVD reactor and adsorb onto a substance being placed at high temperature. The molecules that get adsorbed react with other molecules or decompose to form crystals. The three steps in CVD method are:

1. Reactants are transported on the growth surface by a boundary layer.
2. Chemical reactions occur on the growth surface.
3. By products produced by the gas-phase reaction has to be removed from the surface. Homogeneous nucleation occurs in gas phase and heterogeneous nucleation happens in a substrate.

The CVD method can synthesize ultrafine particles of less than 1 μm by the chemical reaction taking place in the gaseous phase. The reaction can be controlled to produce nanoparticles of size ranging from 10 to 100 nm [46, 47].

2.2.3 Microwave irradiation

Microwave irradiation is a synthesis method that has been widely used in the synthesis of organic, inorganic, and inorganic-organic hybrid materials because of its well-known advantages over conventional synthetic routes [48].

A research was conducted on a rapid and efficient oxidation of organic compounds in microwave condition with new phase transfer oxidative agent: CTMABC. CTMABC (1 mmole) was suspended in acetonitrile (2 ml) and an alcohol (1 mmole in 0.5–1.5 ml of acetonitrile) was quickly added at room temperature and the resulting mixture was stirred vigorously. The mixture was then irradiated by microwave radiation (3.67 GHz, 300 W). The solution became homogeneous for a short time before the black-brown reduced reagent precipitated. Thin layer chromatography (TLC) and UV/VIS spectrophotometer (at 352 nm) were used to monitor the progress of reactions [49].

In another experiment conducted by Sahoo Biswa Mohan et al., o-Phenylenediamine (1.08 g, 0.01 mole) and anthranillic acid (1.37 g, 0.01mole) were dissolved in ethanol (15 ml). And K_2CO_3 was added to a mixture and the reaction mixture was put in microwave oven and refluxed at power (140 Watt) for 10 min. TLC was used to monitor the reaction. After the reaction was complete, ethanol was removed by distillation process and the residue was poured into crushed ice. Then the reaction was made alkaline by using 10% NaOH to get the solid product. The product was filtered, dried, and recrystallized from ethanol [50].

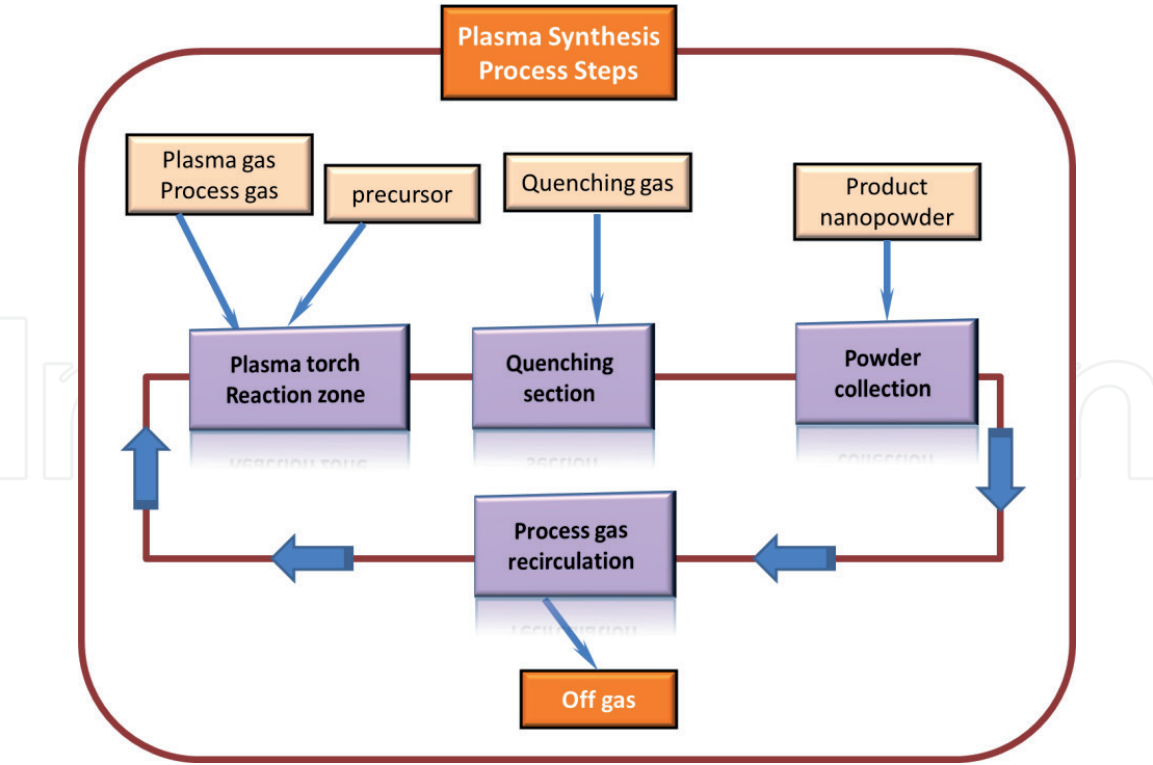


Figure 2.
Flow diagram for production plant based on plasma burners. The recirculation system is of special importance in the case of expensive reaction or carrier gases.

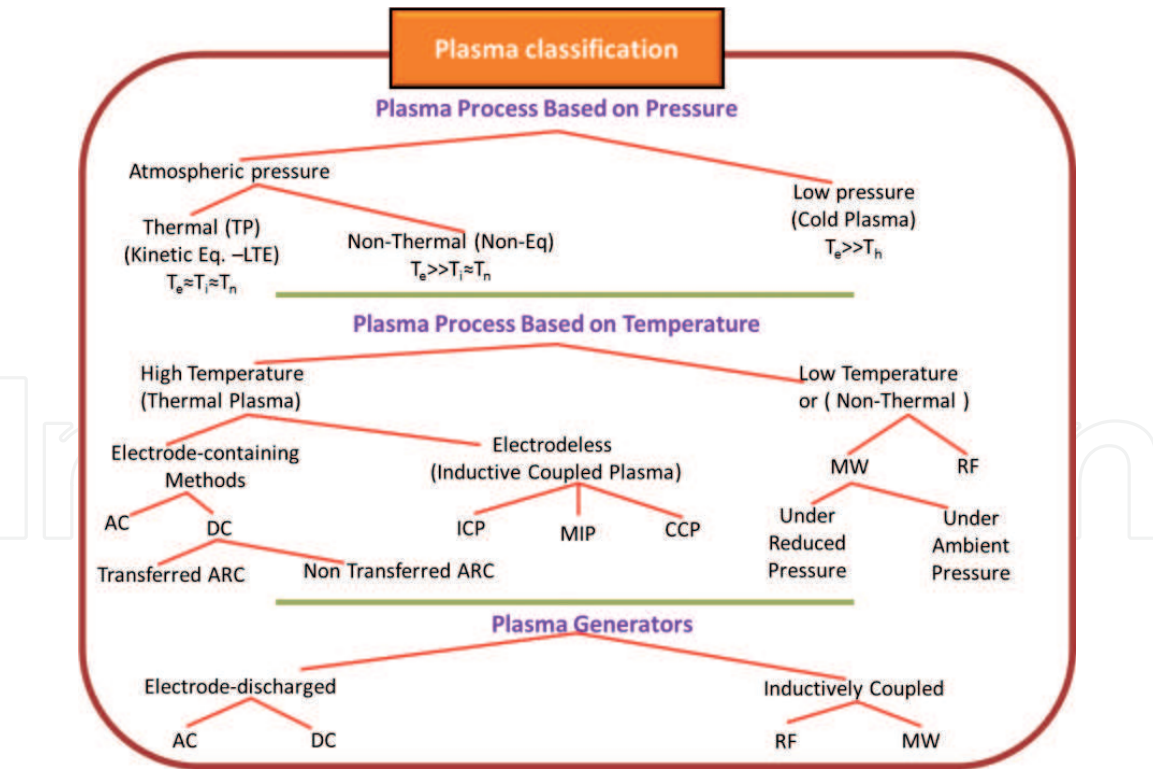


Figure 3.
Different plasma classification.

Sahoo Biswa Mohan et al. conducted another experiment where N-(2-(1H-benzo[d]imidazol-2-yl) phenyl) acetamide (2.51 g, 0.01 mole) was dissolved in ethanol (30 ml) and various aromatic aldehydes (0.01 mole) were taken and then an aqueous solution of KOH (2%, 5 ml) added to it. The reaction was then put in

a microwave oven and refluxed at power (210 Watt) for 10–20 min. The excess solvent was removed by vacuum distillation and then poured into crushed ice and acidified with dilute HCl. The product was filtered, dried, and recrystallized from ethanol [51].

Microwave-assisted organic synthesis has been widely used due to enhanced reaction rates, higher yields, improved purity, ease of work up after the reaction and eco-friendly reaction conditions compared to the conventional methods. In above experiments, microwave irradiated synthesis of chalcone was carried out to get higher yield with less reaction time period as compared to conventional method.

The synthesized benzimidazolyl chalcone produces yield around 60% (conventional) and 80% (microwave) [52].

Another study was conducted to synthesize silver nanoparticles (AgNPs) in aqueous medium by a simple, efficient, and economic microwave-assisted synthetic route using hexamine as the reducing agent and the biopolymer pectin as stabilizer. The synthesized AgNPs were characterized by UV-VIS, Spectroscopy, Energy dispersive X-ray (EDX), X-ray diffraction (XRD), and Transmission electron microscopy (TEM) techniques. The nanoparticles were found to be spherical shape with an average diameter of 18.84 nm. The rate of reaction was found to increase with increasing temperature and the activation energy was found to be 47.3 kJ mol^{-1} [53].

ZnS nanoparticles were synthesized by microwave-assisted irradiation method. The produced ZnS nanoparticles were characterized by XRD, SEM, and UV-Vis spectroscopy. The average size of the nanocrystallites was measured by Debye-Scherrer formula as per the XRD spectrum, and there were found to be approximately 6 nm [54].

2.2.4 Pulsed laser method

Pulsed laser method is a method that is mostly used in the synthesis of silver nanoparticles, at a high rate of production of 3 gm/min. Silver nitrate solution and a reducing agent are poured into a blender-like device. The device is composed of a solid disc that rotates with the solution. The disc is exposed to pulses from a laser beam to create hot spots on the surface of the disc. Hot spots are where the silver nitrate reacts with reducing agent to produce silver particles that can be separated by centrifuge. The particle size is controlled by the energy of the laser and angular velocity of the disc [46] (Figures 4 and 5).

2.2.5 Sonochemical reduction

Sonochemical method has been studied in the synthesis of metal nanoparticles. The synthesis of different types of metal nanoparticles has been studied by use of the sonochemical reduction of the corresponding metal ions. The sonochemical reduction of MnO_4^{4-} , Au^{3+} , Au^+ , and Pd^{2+} in the absence and presence of organic additives were investigated in relation to the synthesis of size and shape controlled metal nanoparticles. The rates of reduction were controlled to control the size and shape of metal nanoparticles. The size of the Au nanoparticles formed from the sonochemical reduction of Au^{3+} was controlled in the presence of an organic stabilizer citric acid [55].

Obreja et al. conducted a study on alcoholic reduction platinum nanoparticles synthesis by sonochemical reduction. H_2PtCl_6 was reduced with methanol, ethanol, and propanol working as solvents and reducing agents, in the presence of capping polymers such as chitosan, polyethylene glycol, and poly (amidehydroxyurethane). The produced nanoparticles size was found to be approximately 3 nm [56].

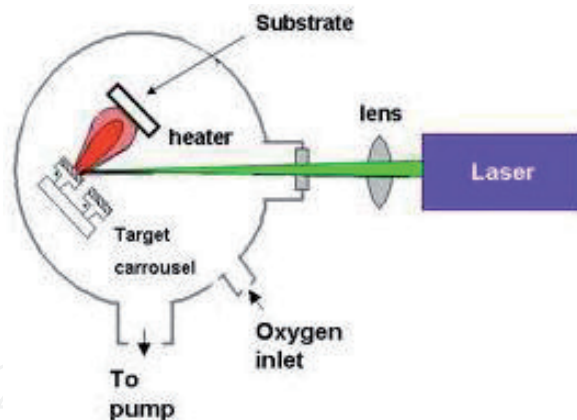


Figure 4.
Synthesis of nanoparticles using a pulsed laser method [46].

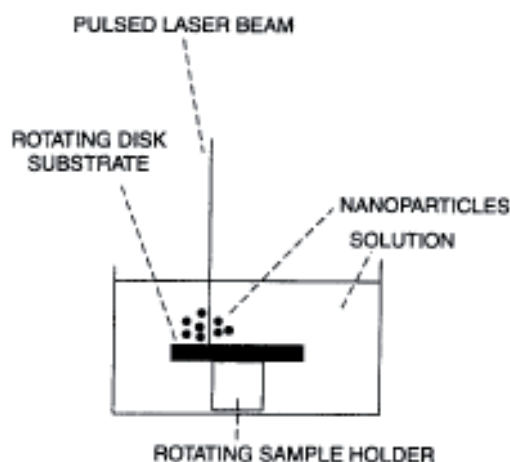
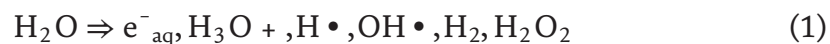


Figure 5.
Apparatus to produce silver nanoparticles using a pulsed laser beam that makes hot spots on the surface of a rotating disk [54].

2.2.6 Gamma radiation

Gamma radiation is the preferred method for metallic nanoparticles synthesis because it is reproducible, may control the shape of the particles yields monodisperse metallic nanoparticles, is easy, cheap, and use less toxins precursors: in water or solvents such as ethanol, it uses the least number of reagents, it uses a reaction temperature close to room temperature with as few synthetic steps as possible (one-pot reaction) and minimizing the quantities of generated by-products and waste [57].

The radiolytic reduction has been proven to be a powerful tool to fabricate monosized and highly dispersed metallic clusters [58]. The primary effects of the interaction of high-energy gamma photons with a solution of metal ions are the excitation and the ionization of the solvent [59]. The different reactions that are observed are well explained in the paper by Abidi and Remita. In particular, water can be produce upon irradiation of a series of reducing and oxidizing agents as shown in the following equation.



For the production of metallic nanoparticles from metallic salt solutions, the reducing agents e^-_{aq} and H^\bullet are the cornerstones of the process. Unfortunately, the production of hydroxyl radicals OH^\bullet hampers the efficiency unless some

specific hydroxyl scavengers are used. Among them, isopropanol is frequently used [60].

This technique has been widely used so far to produce solutions of MNP primarily gold and silver that were further investigated by UV-Visible spectroscopy with the aim to analyze their plasmonic absorption band. A wealth of literature can be found on this topic [61, 62]. Additionally, γ rays irradiation was also used to trap MNP inside polymers or inside porous frameworks like mesoporous silica for instance [63–65].

3. Conclusions

Nanoparticles have gained significant interest due to their unique chemical and physical properties and are applicable to diverse areas. Various methods of preparation of nanoparticles have been developed and they are suitable for synthesis of nanoparticles in different sizes and shapes. The methods that were discussed include gamma irradiation, chemical reduction photochemical method, thermal decomposition, and microwave irradiation among others.

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