

## PLASMA SYNTHESIS: A NEXT-GENERATION APPROACH TO METAL NANOPARTICLE ENGINEERING

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### Abstract

Owing to distinct properties and diverse applications, improvement in synthesis strategies of metal nanoparticles emerged as a field of prime interest in material science. There are a number of potential synthesis techniques for the purpose but synthesis of metal nanoparticles using plasma processes has been a highly resourceful route. The synthesis parameters such as discharge current, discharge voltage, discharge time, precursor concentration, presence and absence of surfactant material, rate of feeding gas, temperature and stirring etc. provide liberty to control the shape and size as well as quality of metal nanoparticles. This review is dedicated to highlight the technological and process development, issues involved and future challenges involved in plasma synthesis along with survey of reported literature on preparation of metal nanoparticles. The description on the most commonly used techniques and set-up for the synthesis of metal nanoparticles and mechanism of particle formation is given.

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### INTRODUCTION

anomaterials are of great interest owing to their abundant applications in various fields of life and utilization in devices [1]. The materials having one or more dimensions in range of less than 100 nm have unique physical, chemical and biological properties which cannot be obtained from otherwise bulk materials [2]. Though the majority of applications are served well with the particle size less than 100 nm however the particles having size less than 10nm are even more appealing for a variety of energy devices and

future appliances [3]. Metals, ceramics, polymers and organic nanoparticles have been synthesized by varying the size and shape such as Nano-sphere, nanorods and Nano-prisms. Among all these the metals nanoparticles earned extensive attention due to their unique optical, electrical and more specifically biomedical applications [4-6]. In a study the researchers presented a comprehensive first-principles investigation of the structural, electronic, and magnetic properties of the novel layered material MoGeTe<sub>3</sub> using Density

Functional Theory (DFT). Here the optimized bulk structure exhibited hexagonal symmetry with distinct layered morphology, confirmed through layer-specific formation energy calculations indicating intrinsic stability and van der Waals layering [7].

The quantum confinement effects play important role in chemical and physical properties of the nanomaterials which deviate them from the bulk counterparts. For instance, catalytic performance of metal nanoparticle is greatly linked to their shape and size [8]. The morphology and size should be such that the atoms of the nanoparticles are easily available and accessible to reactant molecules to initiate reactions. Hence, nanoparticles having smaller size and flatten shape are usually preferred because a larger fraction of the metal atoms is present at the surface [8]. In order to prepare the metal nanomaterials, variety of techniques have been used which include solution phase chemical reduction, metal vapor deposition, thermal deposition, UV photolysis, sono-chemical decomposition, electrochemical, physical and plasma processes etc [9].

The surface properties depend on the synthesis process which affects the final product drastically and may cause post-synthesis phenomenon such as agglomeration and resistance to surface oxidation [10, 11]. Different level of purity of the final product is achieved by different synthesis technique. It is believed that the gas phase plasma synthesis leads to pure products rather than the liquid phase plasma synthesis [12, 13]. The plasma is an ionized gas and considered to be the fourth fundamental state of matter besides the liquids, solids and gases. Modern industry and even humane life are greatly linked to plasma. Plasma is generated through the ionization of the neutral gases at higher temperatures. Besides the equal number of positive ions as well as negative electrons it also contains the neutrals, reactive radicles, ultraviolet light, metastable excited atoms or molecules and strong electric and magnetic fields [14]. Due to presence of the above-mentioned species plasma has unique properties sometimes called the "dusty plasma" which is used for the particle synthesis. The plasma is utilized in manufacturing industries like surface modification of materials and the surface process in integrated circuits processing [15]. The products of plasma synthesis have proved to have a great impact on capital investment economy, trade and other aspects of life [16]. The plasma can be broadly specified into different categories mentioned below which are based on the temperature and pressure of the species.

Thermal plasmas are specified by a dynamic equilibrium in which all the reactive species have the same temperature [17]. This temperature might be around 10,000 K. Arc plasma is an example for the thermal plasma. Non-thermal plasmas are defined by a thermal non-equilibrium it means the reactive species have different temperatures. Electrons have the temperature of several electron volts and positive ions and neutral elements have a rather colder temperature equal to the room temperature. Non-thermal plasma is suitable for the preparation of the nanoparticles because its overall

temperature is quite low which gives the benefit to produce nanoparticles at the low temperatures, could be ignited by even microwaves and RF. Non-thermal plasmas, also termed as cold plasma, its temperature ranges around 300-1000 K [18, 19]. Thirdly, atmospheric pressure plasmas are plasmas operated at atmospheric pressures and makes the process economical. Further, it needs smaller experimental efforts to ignite the plasma so technique is quite suitable for the industrial process. Both thermal and non-thermal can be operated at atmospheric pressures. Power sources are equally possible by RF, DC and microwaves. Low pressure plasmas are only operated at highly vacuum conditions usually ranges from 100Pa to 10,000Pa. This makes the technique expensive because it needs more sophisticated equipment and expensive vacuum components. Cold plasmas are generally the low pressure plasmas [17, 20, 21].

Plasma assisted fabrication is continuously increasing in importance to synthesize nanomaterials of different assemblies and hierarchies. This is right in accordance to meet the requirements in the applications for existing and future technological applications. The major funding agencies, industries and research units are working to improve the plasma fabrication strategies owing to its potential. It is believed that the performance of plasma-based nanomaterials and resulting devices have superior quality over the other competing nanofabrication methods. There is a need to further improve the plasma synthesis which ensures to prepare the metal nanoparticles with tailored size, shape and properties [22]. However, owing to properties of plasma processes, there is still possibility to further develop the technique for the synthesis of required size as well as morphology of nanoparticles which would be cheap, easy to handle and friendly to

environment. This review is dedicated to highlight the technological improvement in synthesis setup and processes involved along with survey of reported literature to explore the further ways to improve the strategy.

## 1. Experimental Details

The experimental setup to prepare the nanomaterials using plasma methods are based on processes which basically involves liquid phase and gas phase synthesis. In what follows, the description of these synthesis processes and involved setup are given.

### 1.1 Liquid phase synthesis

Plasma liquid interactions (PLIs) is an important branch of plasma research for the nanomaterial synthesis over the past decades and is rapidly rising because of its potential to operate from low to the atmospheric pressures. There exists a novel plasma liquid interface at which the physical and chemical reaction takes place and actually these interface reactions are responsible for the synthesis of the nanomaterial ranging from metal nanoparticles to graphene sheets. It may be categorized into the two simple classifications (i) plasma over the liquid and (ii) plasma inside the liquid. In both these types plasma liquid interface exists besides the liquid phase as well as the gas phase plasma. The plasma liquid interface is the important for the nanomaterial fabrication more specifically the

synthesis of metal nanoparticles [15]. Hoffman electrolysis apparatus which is often called dual plasma electrolysis is usually employed for the metal nanoparticle fabrication. Glow discharge plasmas at atmospheric pressures act as electrodes rather than the metal electrodes which immersed in the liquid solution. Plasma cathode irradiates the electrons in the solution surface and plasma anode generates the cations in the solution surface. The experimental setup which allows the simultaneous investigation of impact of electron and positive ion over the preparation of metal nanoparticles is shown in the figure 1. In order to prepare silver and gold nanomaterials, the aqueous precursor  $\text{AgNO}_3$  and  $\text{HAuCl}_4$  are used respectively. In case of  $\text{AgNO}_3$  the Ag nanoparticles are formed on the plasma cathode side whereas in the case of  $\text{HAuCl}_4$  gold nanoparticles are formed at both plasma anode and plasma cathode sides. This is obvious that the silver nanoparticles are reduced by electrons and gold nanoparticles are reduced through ion irradiation instead of the electron irradiation. This concludes that the dissociation reaction of  $\text{HAuCl}_4$  is triggered by the positive ions at the liquid interface. When both these materials are used as mixture e.g.  $\text{AgNO}_3 - \text{HAuCl}_4$  the nanoparticles are formed in such a way that there is gold core and a silver surface. Surfactant free electrostatically stabilized gold nanoparticles have been prepared through plasma induced liquid chemistry [23-24].

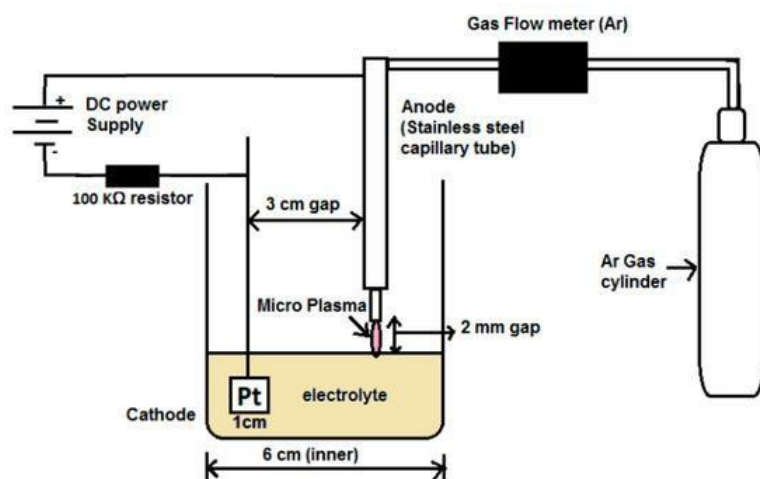


Figure 1: Experimental set-up of plasma electrolysis for the synthesis of metal nanoparticles. Reprinted from permission through open access article [26]

Without the use of any capping agent the gold nanoparticles is usually prepared by non-equilibrium plasma induced electrochemistry. It is believed that the size, shape and rate of synthesis are greatly

affected by the plasma processing parameters. In the aqueous media different shaped particles have been prepared. The size of the particles is greatly linked by the initial concentration of the precursor which is

$\text{HAuCl}_4$ . The required sized particles can be prepared by varying the concentration. The PH value, conductivity and concentration of  $\text{H}_2\text{O}_2$  is needed to be addressed. The role played by the reducing agent  $\text{H}_2\text{O}_2$  is important to reduce the gold cations and particle produced are electrostatically stabilized. The impact of the concentration of precursor as well as the discharge current on particle growth is very important. Lower the concentrations, smaller will be the resulting

particles whereas the rate of reaction can be greatly enhanced by increasing the discharge current. The mechanism is initiated by the reactions at the plasma liquid interface.

Figure 2 illustrating the setup for generation of micro-plasma at atmospheric pressure to prepare gold nanoparticles [27]. Helium plasma has been generated inside the vessel where a hollow capillary containing the micro-plasma acted as the cathode and the carbon rod acted as the anode.

The vessel has a metal salt solution  $\text{HAuCl}_4$  in deionized (DI) water. The surface characterization of the prepared materials performed by using TEM analysis shows that different shaped particles spherical, triangular, hexagonal, pentagonal and cubical have been formed.

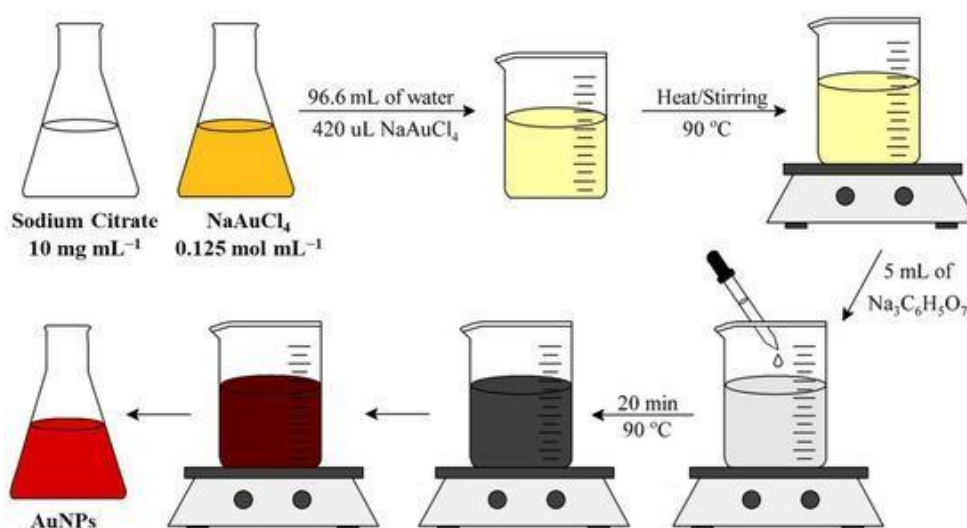


Figure 2: Schematic of micro-plasma setup utilized for preparation of gold nanoparticles. Reprinted from permission through open access article [28].

Platinum nanoparticles were synthesized through the plasma chemical reduction techniques. This method was more specifically adopted to focus the reaction zone at the interface. Right above the solution surface hydrogen plasma was produced and platinum nanoparticles were produced by reducing  $\text{H}_2$   $\text{Pt Cl}_6$ . At  $10^\circ\text{C}$  temperature of the solution the particle size was 2nm.

This method is simple and fast for the production of Pt nanoparticles and does not necessitate the use of Stabilizer[29]. Two critical steps involve in this process

one is to prepare of the salt solution and other is the reduction of that metal salt solution by reducing agents. They generated the hydrogen plasma for reduction of metal salts to neutral metal atoms. They reduced the  $\text{PdCl}_2$  to metallic Pd when  $\text{PdCl}_2$  is treated with hydrogen plasma. Simplest chemical reaction exists in the system. This chemical process produced  $3\mu\text{m}$  thick  $\text{PdCl}_2$  film having coating of Alumina substrate and the reacting system without heating takes only 30 minutes to complete the reaction. The experimental strategy is shown in figure 3.

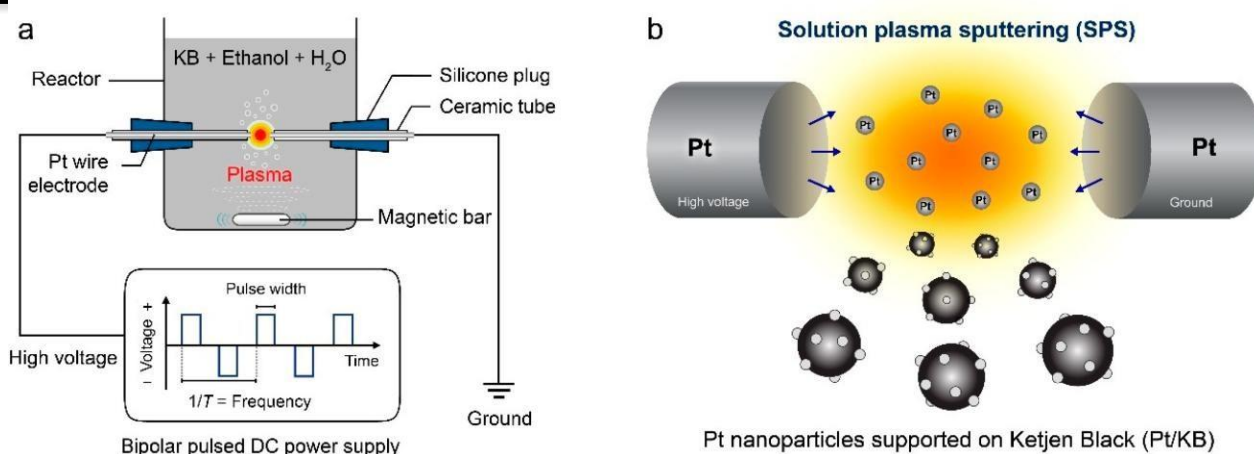


Figure 03: (a) Experimental setup of solution plasma sputtering (SPS) to prepare Pt/KB catalysts (b) enlarge view of electrode gap, revealing the production of nanoparticles from the electrode and their deposition on support. Reprinted from permission through open access article [30].

The generated hydrogen plasma has been powered by radio frequency discharge [30, 31]. The atmospheric pressure conditions and H-He have been used as the working gas. The technique for the reaction to occur at the plasma liquid interface has been improved in such a way that the plasma becomes directly in contact with the ionic salt and precursor hexa-chloro-paltinic acid  $H_2 PtCl_6$  has been utilized. As far as the method of solution phase is concerned using the low power hydrogen plasma this technique in solution phase is novel for noble metal nanoparticles production. No use of surfactant, low temperature reaction, atmospheric pressure and fast kinetics are its novel aspects. The hydrogen plasma technique is useful for the archaeological plasma reduction.

Richmonds et. al. synthesized metal nanoparticles by employing microplasma reduction of colloidal cations [32]. The synthesis of Ag and Au nanoparticles has been carried out by employing the processes of plasma liquid electrochemistry. The atmospheric micro-plasma acted as cathode and a solid metal anode in an electrochemical cell. The micropalsma operated at high pressures about 1 atm non-thermally which given the best substitute of thermal low pressures plasmas. The experimental set up is shown in figure 4.

The sustainable redox battery system is shown in figure 4 which shows that electricity derives cathod reaction producing OH<sup>-</sup> ions. There is chemical reactor which use  $[Fe(CN)_6]^{4-}$ , which cause the reduction of O<sub>2</sub> from seawater. It has been found that the micro-plasma contains a highly energetic

electrons which reduce the metal cations in the aqueous solution [33]. Due to presence of these electrons, no external reducing agent is required. This is the benefit offered by the strategy over the other conventional methods for nanoparticle synthesis[34]. Fructose has been used as the surfactant material.

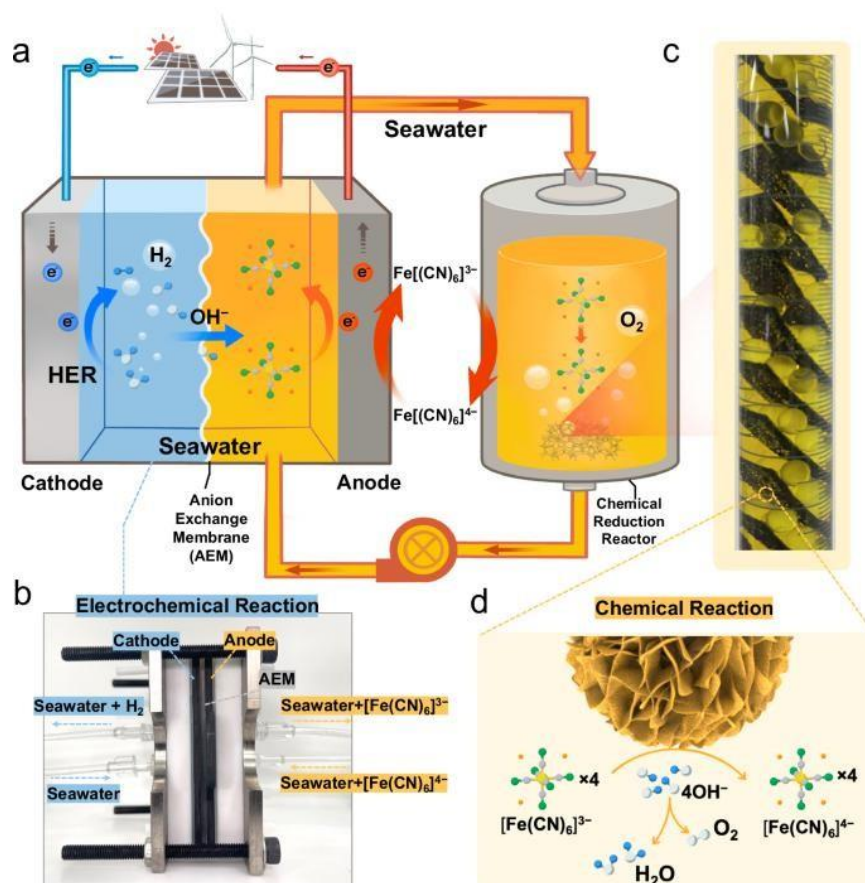


Figure 04: The illustration of redox battery system showing renewable energy using seawater. (a) the overall system using electricity which derive cathod reactions (b) the electrochemical reactor cell which used use [Fe(CN)<sub>6</sub>]<sup>4-</sup> cause the reduction of O<sub>2</sub> from seawater. Reprinted from permission through open access article [35].

The findings obtained from UV-visible spectrometry revealed that the synthesis parameters are directly related to the redox reactions initiated by micro-plasma at the interface in the generated electrochemical cell. The experiment has been performed for the two different cases and particle growth kinetics was compared. One for using metal foil immersing in the solution containing an acid as electrolyte and other is using metal salt solution with no use of acid. It has been found that the in the case of metal foil the anodic dissolution occurs due to which metal dissolve in the presence of electrolyte to generate cations which are then reduced by the electrons. On the other hand, in the case of metal salt solution no dissolution of the anode occurred. The redox reactions initiated the process in this case. The findings for the both cases shown that for metal foil the particle growth starts slowly, increase rapidly and continued to increase. Whereas for the metal salt solution the particle growth increased rapidly in the initial and saturate after the further increased in the reaction time. The growth parameters involved the

current density, acid or metal salt concentration and surfactant concentration [36-38].

The exploitation of plasma liquid interaction to fabricate the metal nanoparticles is fascinating. Zou et. al. has reported an alternate method of reduction of metal cations from Ar glow discharge plasma instead of conventional reducing chemicals [16]. The glow discharge set up used by this group was similar to that described in previous sections[39-44]. The plasma has been energized by high voltage DC generator. Two stainless steel electrodes having 170 mm distance between them was positioned to conduct electricity. The vacuum of the order of approximately 100 Pa has been created inside the chamber. The plasma forming gases were Ar and O<sub>2</sub> with flow rate of 10 mL/min with current in a range of 1-2 mA. The reduction time has been specified as 60 minutes. The plasma reduced metal particles have been found in the form of amorphous clusters which were found transformed into crystals upon thermal treatment. Further, oxygen and hydrogen glow discharge

plasma can act as reducing agents for noble metal ions and clusters with mechanism as given in figure 5 [16].

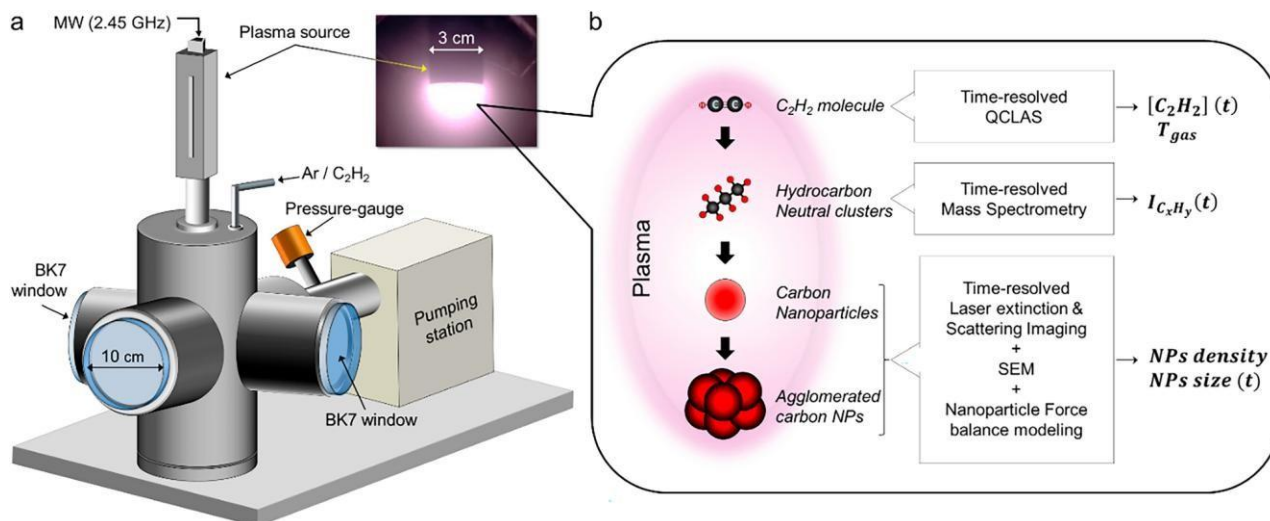


Figure 5: Mechanism of plasma reduction of metal nanoparticles. Reprinted from permission through open access article [45]

The preparation of metal nanoparticles by reduction from glow plasma has been routinely carried. Chou and Jonathan tested the preparation of iron nanoparticles by utilizing the microwave plasma [11]. The findings of their work pointed out that, the nanoparticles were of pure iron in case of hydrogen plasma but were in the form of iron oxide in case of oxygen and argon plasma. The growth parameters include injection point as well as composition of the plasma. An experiment to carry out plasma reduction of metals Ag, Au, Pt, Ir, and Ru cations to prepare their nanoparticles has been reported [46]. This approach appeared to pattern the metal nanoparticles as low cost, transparent and flexible outcome in a non-lithographic manner in polymeric systems. Later in another attempt, reduction of metal nanoparticles has been carried out by Ar glow discharge method to prepare Au-Pd mixed nanoparticles [47]. The structures were obtained via hydrogen thermal reduction which facilitated the growth of dispersed nanoparticles in the form of Pd shell-Au core which exhibited excellent catalytic properties. In another attempt, reduction by using liquid plasma has been carried out to synthesize silver nanoparticles [27]. It has been observed that, the time of plasma treatment as well as starting concentration of  $\text{AgNO}_3$  precursor put effects to increase the size of prepared silver nanoparticles.

## 1.2 Gas phase synthesis

The gas phase methods involve vaporization of the chemical compounds as precursors where chemical

reaction takes place in the gaseous environment. The formation of nanoparticles begins by the homogeneous nucleation process after the collision of evaporated atoms or molecules [48-50]. The condensation of vaporized molecules takes place and nuclei start growing on the already existing particles [50]. The collisions then facilitate the growth and coagulation of the particles further. The ignition of gas phase plasma has been made by radio-frequency, microwave, dielectric barrier discharge (DBD), alternating current (AC), direct current (DC), electric arc (EA) methods [16]. The energy required for starting the reactions can be given by thermal plasma to vaporize the precursors for super saturation followed by particle nucleation. The energy obtained from plasma fully decomposes the precursors into atoms from the nanoparticles on condensation [17, 51].

The gaseous plasma-based method is specifically used to produce uniform and spherical shaped metallic nanoparticles. An aerosol of solid metallic microparticles is produced at first. Non-oxidizing plasma generation is basic requirement in this method. In order to vaporize the microparticles the temperature of this non-oxidizing plasma must be sufficiently high. The metal microparticles aerosols are then directed towards plasma zone which vaporize the microparticles to metal vapor. These metal vapors are pulled away from the hot plasma zone to enter in the afterglow region in order to cool and condense to form metallic nanoparticles. In this method the growth parameters include plasma driven power, heating rate of precursor gases, cooling rate of molten

particles and total gas pressure. The variation of these parameters can provide metal nanoparticles of desired size and morphology. It is energy efficient and cost-effective method which produces metallic nanoparticles from inexpensive precursors. Aluminum nanoparticles have been produced as a practice. All metals including alkali metals Li, Cs, Na, Rb, K and Fr alkaline earth metals Be, Mg, Ca, Sr, Ba and Ra transition metals Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Nb, Mo, Tc, Ru, Rh, Pd, Ag, Qd, La, Hf, Ta, W, Re, Os, Ir, Pt and Au metals of the lanthanide series, metals of the actinide series and post transition metals Al, Ga, In, Si, Ge, Pb, Sb, Te and Bi in the periodic table can be obtain in the form of nanoparticles [53].

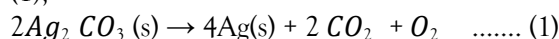
Highly irregular shaped nanoparticles can be converted to smaller, uniform and spherical particles by this method. Microwave, Radio frequency and DC source is suitable for generating plasma for this method. The precursor gases are chosen on the basis of the non-oxidizing property with all metals. He, Ne, Ar, Xe, nitrogen, halogen gases and even oxygen gas are the appropriate choices for generating non-oxidizing plasmas. The metals highly resistant to oxidation can be treated with the

oxygen plasma like noble metal Pt. It is concluded that highly pure metallic nanoparticles are produced by this method than PVD, evaporation, laser ablation and other conventional methods.

Gas phase synthesis yields the clean products. This technique has been highly resourceful to produce silver nano-powders by using microwave as the plasma driving source [22]. In this study micro-plasma also leads the benefit of good stability, uniform temperature field and it operates at atmospheric pressure. Besides this benefit it also provides lower reaction temperature than DC and RF plasma. It is possible to control the temperature during the reaction in the range of 300- 900°C which depends on the gas pressure, chemical species and field strength. In this study the silver carbonate has been used as the precursor material which is 99.5% pure for the production of silver nano powders. The experimental set up includes microwave source, resonance chamber, and reaction chamber having a quartz tube inside, heat exchange and powder collector. Nitrogen has been used to ignite the plasma and frequency of the microwave used was 2.45 GHz. Feeding of the raw material

$Ag_2CO_3$  has been done by a specially made device called powder dosing device. Feeding rate of the precursor is controlled by the pistol in the dosing device. Raw material  $Ag_2CO_3$  is then injected in the reaction

chamber having the nitrogen gas. The plasma is ignited by highly energetic microwave as energy source, the heat evolved also decomposes the raw material  $Ag_2CO_3$  and silver monomers and clusters are formed by the condensation. By the collision of these monomers and clusters the homogenous nucleation begins to start. Particle growth and coagulation occurs. The silver particles are obtained as per reaction given in equation (1);

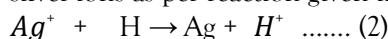


The usage of heat exchanger helps immediate quenching of the powders formed at the low temperature wall and are separated from gas by filter near the powder collector.

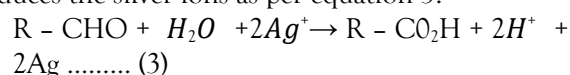
## 2. Mechanism

### 2.1 Mechanism of liquid phase synthesis

The mechanism of preparation of metal nanoparticles by utilizing liquid phase plasma is explained by describing the preparation of silver nanoparticles. The fabrication of silver nanoparticles could be carried out through RF driven atmospheric pressure plasma jet. The impact of long-lived plasma species such as UVV, fructose as well as short-lived plasma species such as H, OH as well as the impact of electrons on the fabrication of silver nanoparticles is needed to be carefully addressed. It is known that short-lived reacting species dominantly initiate the reaction mechanism for the preparation of silver nanoparticles. However, in case of gold nanoparticles  $H_2O_2$  reduces the gold nanoparticles. Both materials are usually treated with Ar and Ar + 0.64%  $H_2$  at few millimeters plasma touching the liquid surface. The process may be initiated by using 1mM of  $AgNO_3$  and 10 mM of fructose in 100  $\mu$ l volume of solution as a result of which H atoms could reduce the silver ions as per reaction given in equation 2;

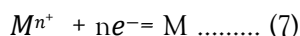
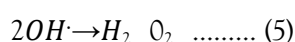
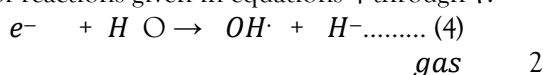


OH is an oxidizing agent it could not reduce the silver ions however it reacts with the fructose molecules. Although fructose is certainly not a reducing agent and in normal conditions it cannot reduce silver ions but plasma treated fructose behaves differently and it can reduce the silver cations. An aldehyde is expected to be formed by this plasma interaction which act as strong reducing agent when oxidized to acid. This aldehyde reduces the silver ions as per equation 3.

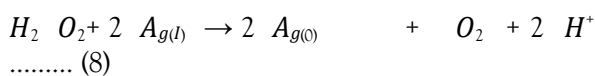


Fructose is a ketone and it don't have this hydrogen atom in normal conditions so it is unable to oxidize and reduce the silver cations. Tautoisomerism is a tollens type reaction which converts fructose to glucose and these basic conditions this ketonis fructose to tautoisomerize to aldehyde glucose and this usually requires an inceased temperatue of 70-75 °C and basic PH which it contain in the plasma environment [53]. The electrostatically stabilized gold nanoparticles free from surfactant have been prepared by plasma induced liquid chemistry [27]. The reaction

mechanism is governed by the initial reaction taking place at the interface. Plasma water interface generates the electron which actually initiate the reaction and ultimately form  $H_2O_2$ . The cause to reduce the gold cations as shown through a number of reactions given in equations 4 through 7.



At the interface the energetic ions strike the liquid surface and create secondary electrons which decompose the liquid constituents. In the bulk plasma the hydrated electrons (the secondary electrons immersed in the water to form hydrated electrons) have great reducing ability. Water decomposes to form H and OH radicles their presence is verified by optical emission spectra.  $H_2$  also produces close to the liquid surface by combining the atomic hydrogen by itself which also act as the reducing agent.  $H_2O_2$  also produces through different pathways which shows different reducing abilities depending upon the PH values for different metals ions. Silver ion reducing follows the reaction by  $H_2O_2$  given in equation 8.



Hydride is  $H^\cdot$  also produces by absorbing the dissociative electron by the water molecule which

acts as a strong reducing agent as given in equation 9.

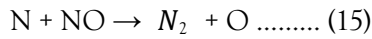
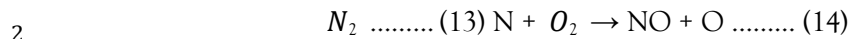
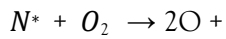
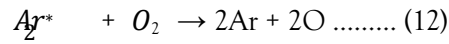
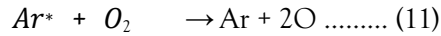


The UV photons transfer energy to water molecules which also creates the active species causing oxidation and reduction [14].

## 2.2 Mechanism of gas phase synthesis

The vapor phase or gas phase synthesis is different from liquid phase synthesis in the sense that the gas phase synthesis does not involve the presence of interface which is predominantly important in the liquid phase synthesis. The energy obtained from plasma fully decomposes the precursors in the gaseous form into atoms which then form nanoparticles upon condensation and cooling. The processes of evaporation-condensation produce nanoparticles from a supersaturated vapor by homogenous nucleation. Nuclei are formed during nucleation when the precursor is got ionized. Nucleus in the vapor growth depends upon the growth condition along with environment. The more the temperature the more the nuclei are formed. primary particles through aggregation and growth mechanism. The nuclei formation earlier produce chemical reaction kinetics is greatly enhanced by plasma because of the ionization as well as dissociation of the molecules to form nanoparticles. It also supports the injection of the precursors into the plasma chamber which decomposes the atoms. These atoms are cooled with a cool gas and particles condense to form the nanoparticles. At this stage both heat and mass transfer occur when plasma is in contact with the condensed matter. Formation of nanoparticles is a strong function of plasma temperature and velocity. Temperature distribution is the tool to access the evolution of the nanoparticles and it depends upon the mass flow density of nanoparticles, cooling surface temperature and density of the flow [15].

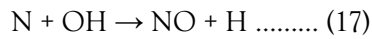
In the vapor phase synthesis, the reacting species undergo a number of gaseous reactions in the plasma plume. A detail of the general mechanism for the vapor phase synthesis is presented here. The reactions shown here are only involving the generation and destruction processes. Atomic oxygen O is generated in the dissociation reactions of  $O_2$  in the different excited species.



This further reacts with the atomic nitrogen to form nitrogen oxide. Here M is the arbitrary reaction partner.

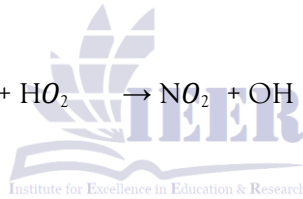


Atomic nitrogen may react to form nitrogen oxide generating nascent hydrogen.



Also additional O is generated from reactions of N with NO.

$N + NO \rightarrow N_2 + O \dots\dots\dots (18)$  Another reaction is also feasible for the production of  $NO_2$



NO is also generated from  $N_2$  and O but in the lesser extent. Reverse of this is also favorable on the high density of Argon gas.

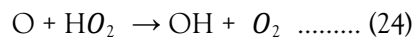


$N_2^*$  is the excited molecule of nitrogen.

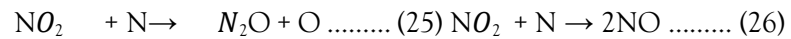
For the formation of  $O_3$ , O could also be lost through reactions with OH and  $HO_2$

$O + O_2 + M \rightarrow O_3 + M \dots\dots\dots (22)$  Oxygen gas is produced through the reaction of atomic oxygen with the OH.

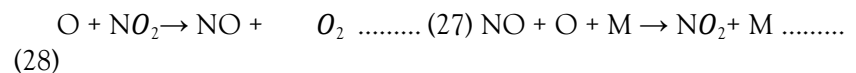
$O + OH \rightarrow H + O_2 \dots\dots\dots (23)$  Following reaction is favorable in this regard for the generation of  $O_2$ .



Nitrogen dioxide combines with N in two different routes to form nitrogen monoxide and dinitrogen oxide.

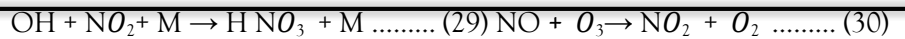


This nascent oxygen produced further combines with the  $NO_2$  for the following products.



$HNO_3$  is produced in the following reaction.

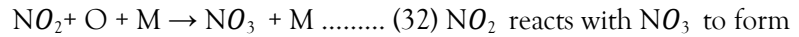




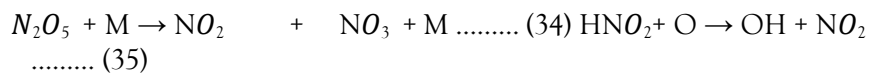
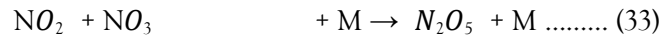
Further oxidation of NO yields NO<sub>2</sub>.



This NO<sub>2</sub> produced further combines with the O and M (the arbitrary partner may be the metal) to form NO<sub>3</sub>



N<sub>2</sub>O<sub>5</sub>.



For the generation s of N<sub>2</sub>O following two processes are found to be important. [54]



**2.2.1 Effect of discharge voltage**

The impacts of discharge voltage on synthesis of gold nanoparticles has been reported [25]. The study has been conducted at 1600V and 3200V with discharge time of 45 minutes. The concentration has been changed to verify the simultaneous effect of both the parameters on synthesized gold nanoparticles. Two samples have been prepared at 0.65mM at 1600V and

0.3mM at 3200V. Smaller and exotic shaped particles such as triangular, hexagonal and pentagonal formed at 3200 V and 0.63 mM. It has been concluded that at higher voltage and small concentration small and different shaped particles are formed. Whereas lower voltage and higher concentration yielded large sized and spherical particles. The experimental setup of it is shown in figure 6.

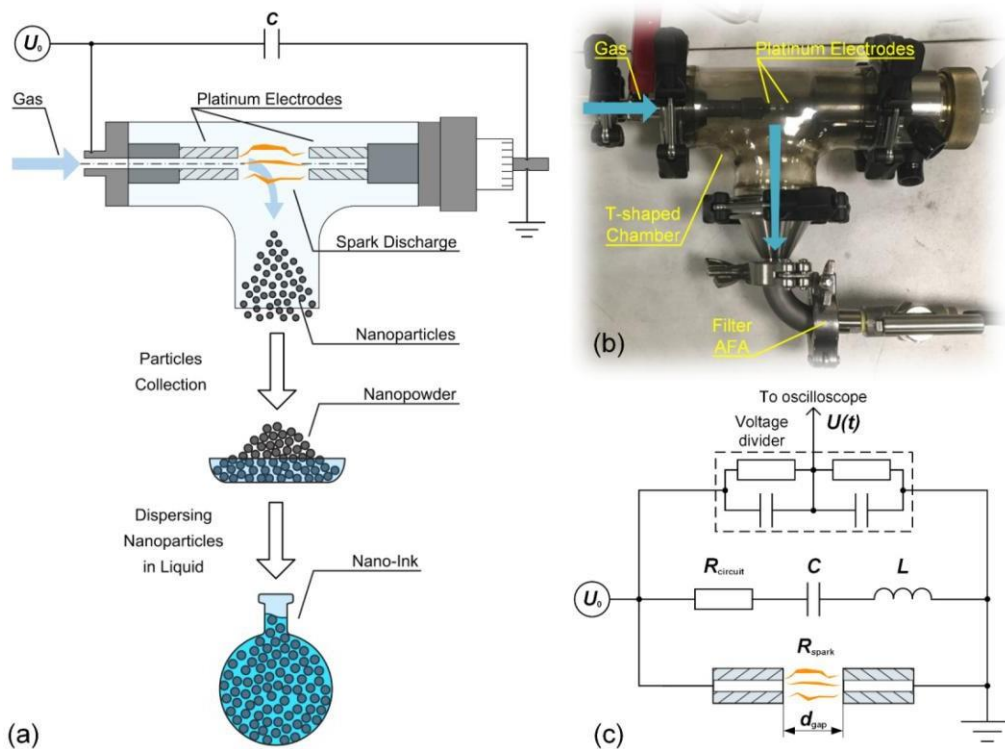


Figure 06: the experimental setup of spark discharge synthesis of nanoparticles. (a) the introduction of gas flow between electrodes (b) the apparatus highlighting the platinum electrodes

(c) the electric circuit model for discharge, used to monitor energy. Reprinted from permission through open access article [55]

### 2.2.2 Effect of discharge time

The exposure time is an important parameter in the nanoparticles formation by all plasma methods. A number of studies has been performed to check the effect of this parameter. Wang et. al. reported the synthesis of silver colloidal nanoparticle by using microplasma at different discharge times [56]. They synthesized the particles at 5, 10, 15, 20, 25 min the reaction time and their UV-visible spectrometry results show that the SPR absorption band was around 396nm. Increase in the reaction time appeared to intensify the absorbance peak which is due to the increment in the AgNPs concentration and narrow size distribution. As the reaction time has been increased from 5 to 20 min the concentration of AgNPs increases at high rate at first and it get saturated at 25 min. Because initially more silver cations were present so they appeared to rapidly reduced by the reducing elements leaving the lesser amount of silver cations in the solution. That's why the growth rate is high in the start and it gets saturated at 25min when all the available cations are consumed.

The similar studies to investigate the effects of discharge time have been reported by other groups (Hieda et. al. 2008). The values of discharge voltage and concentration have been fixed at 3200V and 0.3mM respectively. The evaluation has been carried out by TEM images which revealed that after the discharge of 5 minutes nanoparticles in hexagonal and dendrite shapes have been formed. The particles size appeared to reduce from 50 to 30 nm and very small particles size in the range of 20 nm formed at the discharge time of 15 minutes. At the discharge time of 25 minutes a significant reduction in the size of the prepared gold nanoparticles has been observed after which triangular and hexagonal shapes appeared dominant. At the discharge time of 45 different shaped particles (triangular, pentagon, hexagon) formed whereas the particle size has been approximately 20 nm [56].

### 2.2.3 Effect of Concentration

The effects of precursor concentration on silver nano particles by atmospheric pressure microplasma has been reported in literature [26]. The effects of the concentration of precursor and discharge current on particle growth have been analyzed by this group. It has been concluded that the lower concentrations produce

smaller particles and rate of reaction formation is greatly enhanced by increasing the discharge current. The reaction mechanism is initiated by the reactions at the plasma liquid interface. UV visible spectroscopy resulted a peak at 544nm which is a characteristic plasmon resonance peak for the gold nanoparticles which also confirms the gold nanoparticle formation. Plasmon resonance wavelength (PRW) is a function of particle size as the average particles size increase PRW also increases. It has been concluded that smaller concentrations lead to narrow size distributions and same shaped particles. Whereas higher concentrations leads to wider size distributions and different shaped particles in a single sample [27].

The same kind of results has been obtained for Ag nanoparticles when variation in the concentration taken place [56]. The salt and surfactant ratio has been set to 0.02 and samples have been prepared at 0.1 mM, 0.2 mM, 0.5 mM and 1mM. UV visible spectrometer results shows that

with the increase in the concentration the peak shifts towards the red from 402nm to 420nm. This result is in the right accordance with the expectation that the absorbance peak is red shifted when the particles are larger in size. Hence, more silver particles form when concentration raises. The result also exhibit that the increased concentration broadens the size distribution from 71nm to 133nm.

A similar work has been reported to investigate the consequence of varying the concentration parameter [25]. The concentration of  $[\text{AuCl}_4]^-$  has been changed by addition of 100  $\mu\text{l}$  of NaBr to the solution after the discharged. The absorption peak shifted towards the shorter wavelength

due to the reduction in the concentration as the peak was observed at 382 nm rather than the characteristic of 544nm. Due to the addition of NaBr to solution also leads to the reduction of the PH of the solution because of the formation of  $\text{H}_2$   $\text{O}_2$  during the discharge. This happens due to the electron donation of H radical to the solution and consumption of OH radical during the formation of  $\text{H}_2$  and  $\text{O}_2$ .

### 2.2.4 Effect of rate of feeding gas

In order to check the effect of rate feeding precursor raw material on particle size distribution a different type of

study has been performed to prepare silver Nano powders by microwave plasma synthesis. Two different types of operating conditions were used in the study termed as run 1 and run 2. The particle size was smaller than 10 nm has been found with both the conditions. When rate of feeding of the precursor of  $Ag_2NO_3$  has been slightly enhanced from 0.50 to 0.70g/min the particle size appeared least disturbed by the increment. Whereas when the rate of feeding gas has been increased twice as before the particles' size increases up to 50 nm and the average size of the particles was 15.7nm. the plasma sputtering method shows the stability of Au nanoparticles, low NaCl concentration leads to narrow distribution and long-term stability as shown in figure 7. By plasma synthesis using microwave source the maximum synthesis rate of the silver nano powders which can be achieved has been 0.6g/min. By controlling plasma current and flow rate of plasma forming gas reaction temperature can be controlled. Higher feeding rate also increase the concentration which increase the nucleation and growth rate of silver particles. By this method broad size distributions are there as compared to other chemical reduction method [22].



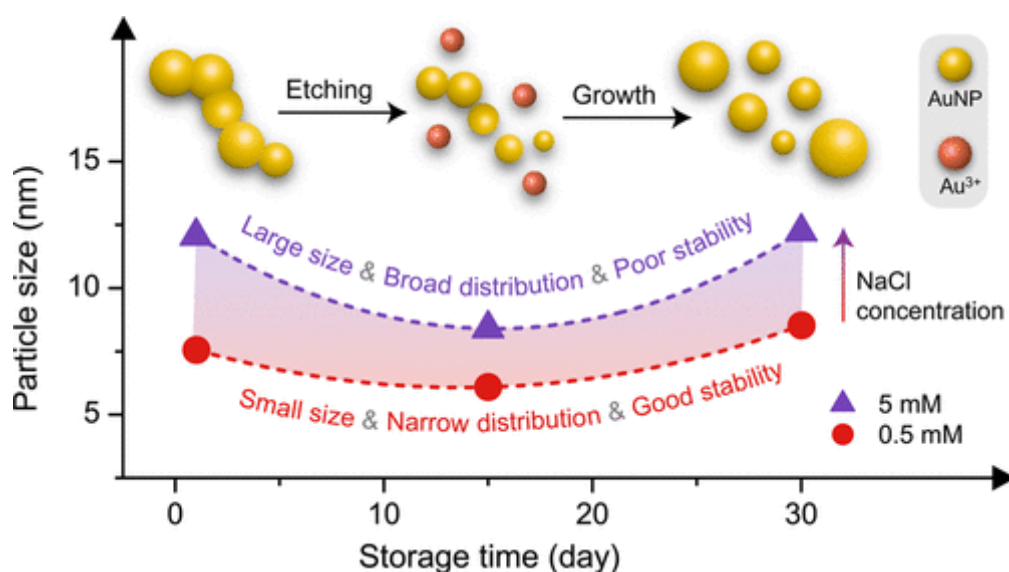


Figure 07: The stability of AuNP synthesized by plasma sputtering method, the two step evolution shows the etching of nanoparticles followed by aggregation and growth. Reprinted with permission from open access article [58].

### 2.2.5 Effect of temperature

The effects of temperature on the synthesized metal nanoparticles have been studied in detail (Huang, et. al. 2013). In order to study the silver nano particles, the electrolyte has been maintained at room temperature 25 °C and high temperature 70 °C. UV-visible spectroscopy results shown that at high temperature larger absorbance is possible and production efficiency also increases at higher temperature. Slightly narrower FWHM has been achieved at 70 °C case which revealed that more uniform sized nanoparticles is obtained for 70 °C in comparison to that of the room temperature case. Because of the peak positions at both the temperatures were nearly the same the average size of particles prepared at both the temperatures was also approximately the same as per TEM results shown in figure 6. The images show that approximately all the particles have spherical shape and average particles size is 15nm and the particles range from 10 to 20nm. However, the particles for 70°C case have been larger in disparity [57].

### 2.2.6 Effect of stirring

The effect of stirring on the synthesized Ag NPs has been investigated on the room temperature with and without stirring the electrolyte without the surfactant [57]. It has been concluded that the non-uniform, agglomerated, undefined and irregular shaped particles have been produced without

stirring in the stabilizer free case. Whereas with stirring spherical, disperse and uniform sized particles have been produced.

### 2.2.7 Effect of surfactant

Wang et. al. prepared silver colloidal nanoparticle by microplasma and studied the effects of surfactant [56]. They used dextran as the surfactant as well as the reducing agent. Dextran contain the benefit of high-level biocompatibility for the biological applications. The ratios which have been considered were dextran/Ag cations of 5:1, 10:1, and 50:1. The synthesized silver particles have been characterized by TEM and AFM as shown in the figure 7. The TEM images obtained the particles synthesized exhibited spherical shape with average diameter of  $16.46 \pm 3.01$  at 5:1 (Dextran/Ag cations),  $11.15 \pm 2.27$  at 10:1 and  $5.37 \pm 1.23$  at 50:1.

Theoretically, the higher surfactant to silver cation ratio should produce ultra-fine particles but practically we can't increase this ratio beyond a certain limit because electrolyte become more viscous which affects the plasma discharge. It has been concluded that at Dextran/ Ag cation 10:1 optimum results have been obtained for reaction time 25 minutes [56].

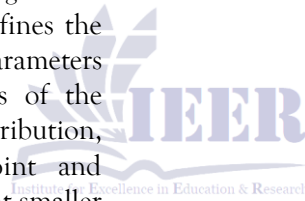
### 2.2.8 Effect of discharge current

The variation in discharge current is one of important plasma parameters which effects the particle growth. The

study to investigate the effects of change in discharge current during plasma growth of metal nanoparticles has been reported. The results indicated that the growth rate of particles is a strong function of discharge current. At longer wavelengths around 600nm and larger discharge currents of 7.5mA it has been observed that spectrum of the particles show a tail. This tail might be due to high concentration present in the solution which causes the particle agglomeration. These results were obtained for Ag nanoparticles whereas Au was also treated with the similar conditions of these parameters. SPR band has been observed at 530nm and at the same value of the discharge current an intense peak for Au may be possible with increase in process time as compared to that of Ag which has a low particle growth rate [59].

### 2.2.9 Effect of injection point and plasma composition

Chou and Phillip in 1992 prepared iron and iron oxide nanoparticles by using microwave generated low pressure and low power plasma by injecting ferrocene into the afterglow region. The injection point and plasma composition have been the parameters investigated in this study. The location of the carrier gas defines the injection point where it is inserted. These parameters appeared to greatly influence the properties of the synthesized particles. The particle size distribution, phase and yield depend on injection point and composition of plasma. The results showed that smaller yields of small particles have been produced from the injection of the ferrocene into the afterglow region. However, in case if ferrocene has been injected through the coupler very higher yields of large particles appeared to be produced. Pure metallic iron nanoparticles have been produced in the hydrogen plasma composition in contrast to the argon and oxygen plasma composition where iron oxide particles have been formed. In all cases particles have been formed in graphitic carbon. The TEM images of both hydrogen and oxygen plasma are shown in figure 8.



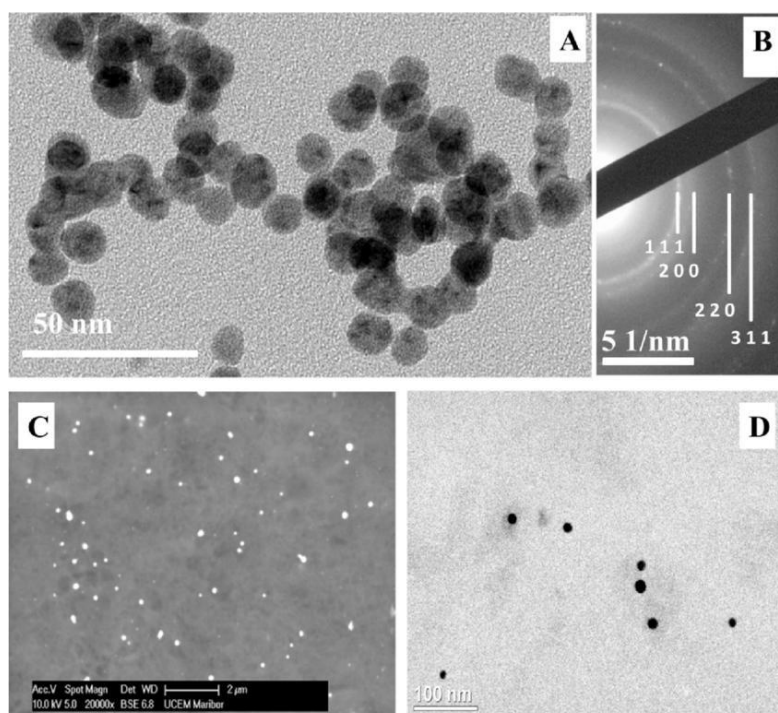


Figure 8: TEM images (a) Dextran/ Ag cation 5:1 (b) 10:1 (c) 50:1 (d) 3D AFM image red arrows shows silver nanoparticles well dispersed and spherically shaped. Blue arrows are the dextran molecules surrounding the silver nanoparticles

The reduction and hence synthesis of Ag NPs has been performed by plasma jet at atmospheric pressure driven by radio frequency. The study of synthesis of the silver nanoparticles involves investigations at two different conditions by checking the effects of non-touching plasma and touching plasma. These two conditions (plasma in contact and without contact to the liquid) leave fluxes of different species to the liquid. It has been observed that for a constant discharge voltage the plasma power increases for the touching case as compared to the non-touching case.

### 2.2.10 Non-touching case

In the absence of liquid the H density for Ar and Ar + 0.64%  $H_2$  has been investigated [53]. Hydrogen has been present in water for Ar as feed gas because of the dissociation of the impurities present in the water. When 0.64%  $H_2$  has been mixed to the feed gas atomic H appeared to increase in magnitude of the order of 1 as compared to the H produced for the Ar case. As the distance from the jet nozzle increases the density of H both for Ar and Ar + 0.64%  $H_2$  decreases as similar to the trend observed for other species like OH in case of non-touching case. The UV continuum emission spectra of the material is

given in figure 10. It is clear that the inverse relation is observed in the emission intensity and the distance from the jet nozzle. The results indicated that the emission intensity reduces with increase in the distance from the jet plasma nozzle. Intense emission has been observed for Ar at 124nm.

### 2.2.11 Touching case

The scenario for Ar and Ar + 0.64%  $H_2$  for the touching case has been complex [53]. It has been reported that the flux of the reacting species is larger in magnitude as compared to the non-touching plasma conditions in case of touching of liquid with the plasma. In the case of Ar the absorbance as a function of the distance relates with the plasma dissipated power. For the case of Ar + 0.64%  $H_2$  the power variation is nearly 12% and for Ar case the power variation is significant by a factor of 1.5.

### 3. Conclusion

To shed light on mechanism of plasma production of metal nanoparticles, an over view of the commonly used techniques and set-up for the synthesis and challenges are discussed in detail. The effects of the plasma

parameters such as discharge current, discharge voltage, discharge time, precursor concentration, presence and absence of surfactant material, rate of feeding gas, temperature and stirring on the properties of synthesized Nano materials are found important. The plasma synthesis techniques are found very effective to tune the properties of metal nanoparticles on the basis of size, shape and yield which put strong impact on utilization of the prepared particles for different applications. The example of growth of silver nanoparticles is used to describe the growth of metal nanoparticles in liquid phase.

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