



Solution Plasma Process (SPP) for Nanoparticle Production: Its Role, Opportunities, and Limitations

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Abstract:

One of the many methods used for nanoparticle production, the Solution Plasma Process (SPP) is a technique that enables nanoparticle synthesis using non-equilibrium cold plasma generated in the liquid phase, typically at room temperature and atmospheric pressure. The Solution Plasma Process offers a broad range of possibilities, including the conversion of metal ions into metal nanoparticles through plasma-induced reduction, the “bottom-up” production of heteroatom-doped carbon structures from carbonaceous precursors via plasma-induced decomposition, and the direct synthesis of oxide/nitride/carbide compounds through the controlled generation of plasma-activated species. Comprehensive reviews and application studies published in recent years have highlighted the potential of the Solution Plasma Process in catalysis, energy materials, biomedical applications, and environmental applications, as well as its advantages in terms of particle size/shape control and surface functionalization. Nevertheless, they also point to challenges that remain to be addressed, particularly regarding scalability, reproducibility, and energy efficiency.

1. Introduction

Throughout the historical development of the field, the term “plasma” was defined by Langmuir in 1928 as an ionized gas containing approximately equal numbers of free electrons and ions.[1] Based on this definition, the concept of plasma has evolved to be simply described as an ionized gas. Today, because of its distinctive properties, plasma is regarded as the fourth state of matter, distinct from the solid, liquid, and gaseous states. Although plasma is considered the fourth state of matter, the solid, liquid, and gaseous states are more common under atmospheric conditions on Earth. The plasma state is predominantly found in the universe, including in the Sun, stars, and nebulae. Accordingly, plasma is characterized by an almost negligible net space charge, which is why it is referred to as a quasi-neutral gas.[2] Nevertheless, artificial plasma can be generated on Earth by supplying sufficient energy to ionize neutral gases.

Plasmas consist of positive and negative ions, electrons, free radicals, photons, metastable species, as well as excited and neutral atoms and molecules.[3]

Plasmas can be classified according to gas temperature, electron and ion temperatures and densities, pressure, current magnitude, power mode, thermodynamic equilibrium, and degree of ionization. The degree of ionization of plasma can range from partially ionized to fully ionized. Fully ionized plasmas can be found in much of the visible matter in the universe, including stars and visible interstellar matter. They occur in many forms, ranging from low-pressure plasmas in the interstellar medium to high-pressure, high-energy fusion processes in the core of the Sun.

2. Materials and methods

The aim and methodology of this review are to provide a general overview of plasma research,

which is one of the important research areas today, and subsequently focus on the solution plasma method used in nanoparticle synthesis, while also comparing the data obtained through our own applications of the method with information reported in the literature. From the recent past to the present, a search of the Web of Science database reveals tens of thousands of publications related to plasma. These studies include gas plasma, solid plasma, and liquid plasma. Although the term plasma generally evokes gas-phase plasmas, which are primarily ionized gases, plasma can also exist in solid and liquid forms. Gas-phase plasma is widely used in numerous industrial applications, such as electronic device manufacturing, hard-coating processes, and surface treatment. Solid-phase plasma is used in surface plasmon resonance spectroscopy and nanoparticle research and is developing as a new research area in plasmonics. On the other hand, although liquid-phase plasma is used in water treatment and electrical discharge processes, it is not yet as well understood. The fundamental characteristics of liquid-phase plasma, including its generation techniques, physical state, and the active chemical species present within it, have not yet been fully determined. Since the molecular density of the liquid phase is much higher than that of the gas phase, greater chemical diversity and a wider range of chemical reactions can be expected under lower-temperature conditions. Therefore, advancing research on liquid-phase plasma is important for engineers and scientists.[4]

The formation of plasmons, which are quanta of plasma oscillations, can be triggered within a solid through the collective oscillation of free electrons moving around a lattice site. In contrast, plasma in the liquid phase is generally generated by an electrical discharge within the liquid. Similar to gas-phase plasma, it can lead to molecular dissociation and the formation of reactive species. These reactive species have attracted considerable interest in both industrial and academic fields.

Considering the large number of studies conducted on plasma, these studies can be broadly categorized into areas such as plasma applications in nanoparticle processing, plasma studies in chemical analysis, plasma applications in organic wastewater treatment, plasma-based sterilization and disinfection, plasma studies in biomedicine—including wound healing, blood coagulation, dermatology, and dentistry—plasma applications in oncology and cancer treatment, plasma studies in agriculture and food safety, plasma in dielectric media, and plasma studies in polymeric media.

Plasma-based approaches are of great importance in nanoparticle synthesis. Various methods are

currently available for nanoparticle synthesis, among which the reduction of metal ions in solution is a powerful approach for producing nanoparticles with different sizes, shapes, and compositions. Aqueous chemical methods based on chemical reduction, in which a suitable reducing agent is mixed into the solution, remain an effective approach. However, the relatively long processing times, which can extend to several hours, are a significant limitation of these methods. To overcome this problem, many research groups have attempted to develop alternative reduction methods in which energetic electrons act as reducing agents. Owing to the unique properties of plasma and the solubility of metal ions in liquids, plasmas generated in or in contact with liquids have attracted significant attention for nanoparticle synthesis. Nanoparticle synthesis using plasma–liquid interfaces offers several important advantages, including the elimination of the need for reducing agents, simplicity of experimental design, and continuous synthesis during plasma irradiation.[5–9] In the past, liquid-phase plasma research focused primarily on wastewater treatment because it can effectively generate reactive species such as hydroxyl radicals ($\bullet\text{OH}$), superoxide anions (O_2^-), and hydrogen peroxide (H_2O_2).[10–13]

Liquid-phase plasma is referred to as “solution plasma” because different plasmas can be generated by selecting different combinations of solvents and solutes in solution. By controlling various parameters associated with discharge ignition, a glow discharge can be generated in a pulsed power system. The generation of a glow discharge in solutions is important because it enables the formation of a new reaction field at nearly room temperature. Solution plasma has potential applications in nanomaterial synthesis, surface modification, water treatment, sterilization, and the decomposition of cellulose and toxic compounds. Depending on the parameters associated with discharge ignition, both glow and arc discharges can be generated using a pulsed power generator.[4]

The experimental setup used in our studies of the solution plasma process, together with relevant images of the process, is presented in figure 2.

Figure 3 presents the FSEM images obtained from our synthesis of Au, Ag, and CuO nanoparticles using the solution plasma process.

The plasma is located at the center and is surrounded by a gas phase enclosed by the liquid phase. There are two interfaces in the system: the plasma/gas interface and the gas/liquid interface. The plasma is confined by a condensed phase, which generates the unique characteristics of solution plasma and enables rapid reactions.

Compared with conventional chemical reactions, arc discharge in liquids, and plasma electrolysis, solution plasma offers several advantages in terms of the reaction zone. Two important effects are associated with solution plasma: the “**cage effect**” and the “**trapping effect**.” Radicals generated by the discharge are surrounded by solvent molecules, and recombination leads to the formation of gas molecules. These gas molecules form bubbles, which is referred to as the “cage effect.” Subsequently, radicals and ions within the bubbles undergo solvation with solvent molecules following collisions, and deactivation occurs through strong emission. This results in plasma quenching, which is referred to as the “trapping effect.” These two effects are associated with the plasma formation process.[4]

In addition to liquid-chemical and physical methods used for nanoparticle production, such as sol-gel processing, chemical reduction, chemical vapor deposition, thermal decomposition, and laser ablation, a new class of approaches based on plasma–liquid interactions has emerged in recent years. In this class of methods, plasma generated by an electrical discharge either within the solution or at the solution surface accelerates transformations that are difficult to achieve through conventional chemical routes by providing high local electric fields, short-lived radical species, and solvated electrons. These conditions can provide both reducing and oxidizing environments at low temperatures, often simultaneously. The solution plasma process can enable the synthesis of metal nanoparticles and functional carbon materials at room temperature and atmospheric pressure without the addition of external reducing agents or ligands. For this reason, current research positions the solution plasma process as a distinct approach within the broader literature on “plasma–liquid systems” and considers it a promising candidate for industrial applications and a relatively green synthesis method.[14]

The solution plasma process enables the production of noble- and transition-metal nanoparticles, particularly allowing the synthesis of noble metals such as Ag, Au, and Pt and transition metals such as Ni and Cu under stabilizer-free or low-ligand conditions. In particular, Ag nanoparticle preparation has demonstrated the rapid and clean reduction capability of plasma.[15] The solution plasma process also enables the production of oxide (CeO₂, TiO₂), nitride, and carbide nanoparticles under low-temperature conditions through oxygen- and nitrogen-derived reactive species as well as localized hot-spot and shock effects.

The solution plasma process is particularly prominent in the production of heteroatom-doped

carbon materials. N-doped carbon nanosheets and dual-heteroatom (N,S)-doped graphene-like structures can be synthesized in a single step at room temperature and atmospheric pressure through the plasma-induced decomposition of solvent–precursor systems. These structures offer sustainable alternatives to precious-metal catalysts for electrochemical applications.[16] The simultaneous reduction–deposition capability of the solution plasma process also enables the fabrication of supercapacitor/electrode materials, for example, by growing Fe₂O₃ nanoparticles on N-doped activated carbon supports. This approach is particularly important because it enables the **in situ** adjustment of surface and interfacial chemistry.[17] In addition, several advantages of the solution plasma process over other methods have been reported. These include operation at room temperature and atmospheric pressure; simultaneous generation of reducing and oxidizing species, which can reduce the need for chemical reducing agents and high temperatures [18]; formation of clean surfaces [19]; and rapid nucleation and finely controlled growth, enabling particle size and shape control through pulsed power and chemical parameters.[20] Studies have also shown that several parameters can be adjusted in the solution plasma process, including the power source and pulse parameters, ionic strength and conductivity, precursor type and solvent chemistry, and gas atmosphere.

Despite these advantages, several limitations have also been reported in studies employing the solution plasma process. These include:

- **Scalability:** Increasing the plasma volume can lead to challenges related to energy efficiency, plasma homogeneity, and heat management.[14]
- **Reproducibility:** Electrode erosion, bubble formation, and localized electric fields can affect product distribution.[21]
- **Process analytics:** The need for real-time process monitoring and diagnostics remains an important challenge.[22]

3. Results and Conclusion

Aqueous and non-aqueous solutions, liquid nitrogen, supercritical fluids, and other media can be used in the solution plasma process. The method has numerous potential applications, including nanoparticle synthesis, surface modification, water treatment, sterilization, recycling of rare metals, and decomposition of toxic compounds. In addition, solution plasma is effective for the hydrophilic treatment of particles. High-quality composites can be produced using this technique,

and it can also be applied to the processing of cellulose and toxic compounds.[4]

Solution plasma is a promising emerging type of plasma with a broad range of applications. Although its fundamental characteristics have not yet been fully elucidated, the physics and chemistry of this plasma remain open to further development. The method offers advantages in numerous fields for synthesizing and modifying materials for important applications, including energy and medical technologies. Carbon materials produced through solution plasma have also demonstrated that the method can provide an effective route for the synthesis and modification of advanced carbon materials with controllable performance for energy applications. Furthermore, metal and metal oxide materials can be modified using solution plasma to sufficiently improve their catalytic performance.

The method also enables a green process requiring extremely low amounts of chemicals, making it suitable for the preparation of biomaterials for medical applications.[18]

In summary, although the solution plasma process stands out due to its ability to produce highly surface-active nanostructures in a single step, at room temperature, and under ligand-free or low-ligand conditions, and offers significant advantages for electrocatalytic and energy applications requiring heteroatom-doped carbon and clean metal surfaces, its industrial competitiveness and future development will depend largely on energy efficiency and control over product distribution during large-scale nanoparticle synthesis. Therefore, research in this direction is likely to become an increasingly important and active area of study in the future.

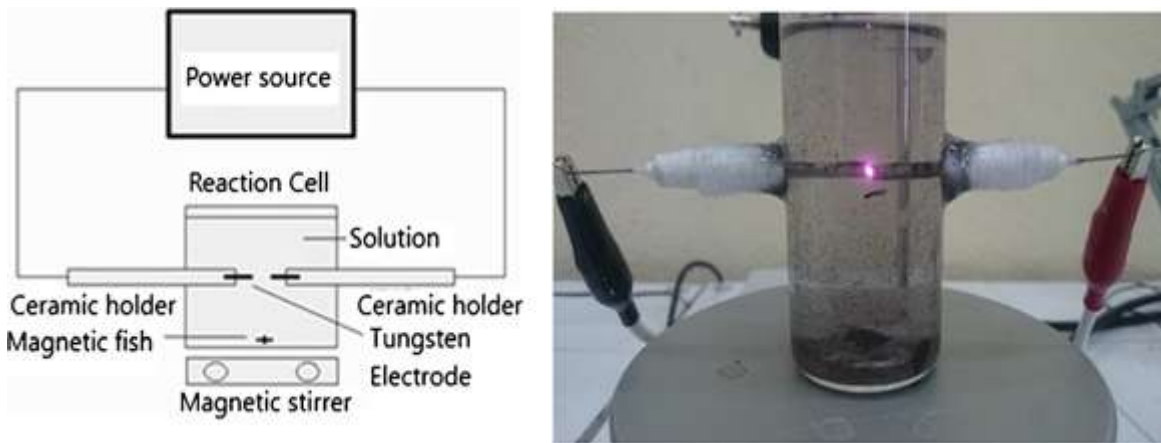


Figure 1: Experimental Setup of the Solution Plasma Process

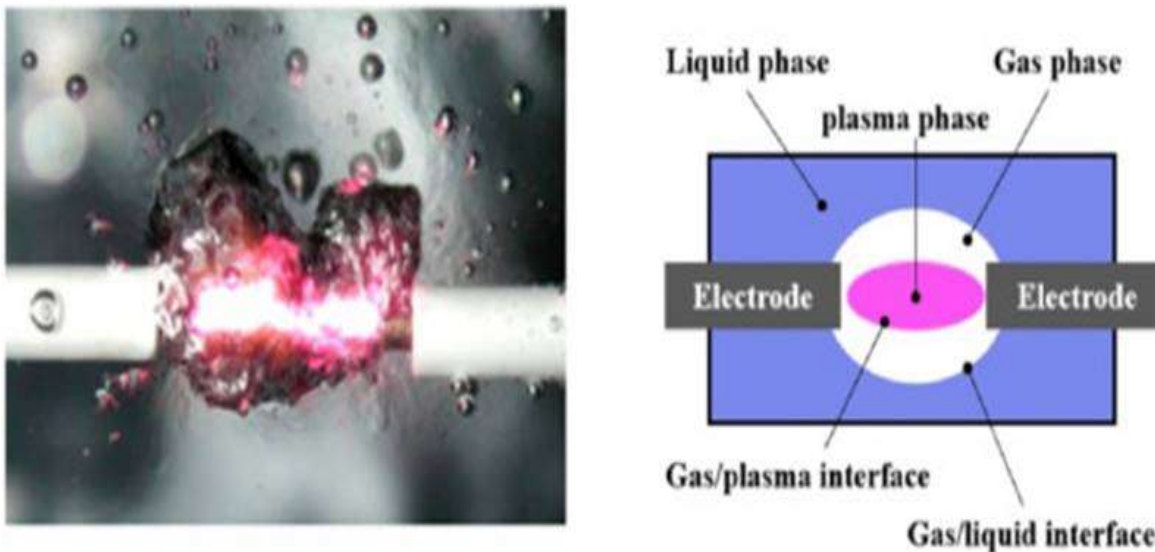


Figure 2: Plasma Formation Between the Electrodes

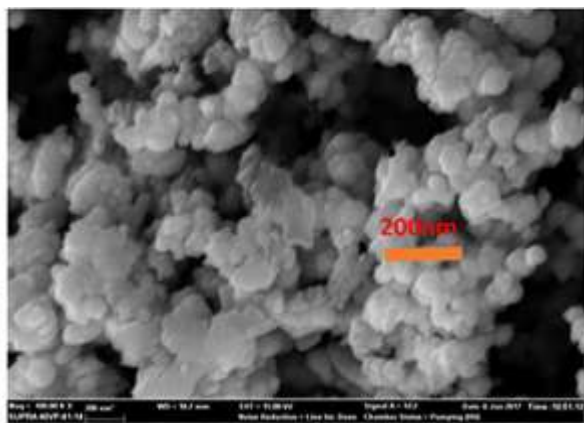


Figure 3: FSEM Image of the Au Sample

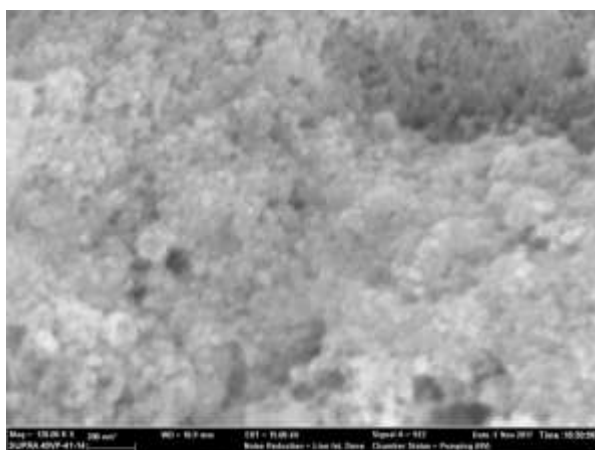


Figure 4: FSEM Image of the Ag Sample

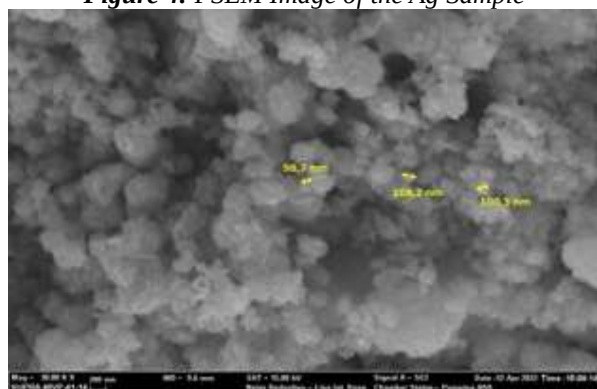


Figure 5: FSEM Image of the CuO Sample

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- **Ethical approval:** The conducted research is not related to either human or animal use.
- **Conflict of interest:** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper
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