

Plasma Technology: A New Remediation for Water Purification with or without Nanoparticles

Pankaj Attri^{*1}, Bharti Arora², Rohit Bhatia³, P. Venkatesu³ and Eun Ha Choi¹

¹Plasma Bioscience Research Center / Department of Electrical and Biological Physics, Kwangwoon University, Seoul, Korea

²Department of Applied Science, ITM University, Gurgaon, Haryana, India.

³Department of Chemistry, University of Delhi, Delhi, India

Abstract

One of the most serious problems facing billions of people today is the availability of fresh water. The conventional water recycling methods like chemical oxidation processes and advanced oxidation processes (AOP) are effective but have problems such as high cost, secondary pollution and production of chlorinated species. To overcome these problems nanoparticles are increasingly used. The conventional methods of nanoparticle production usually requires a long time and have a high cost, which render them ineffective for large-scale production of nanoparticles. Nanoparticle production using the plasma technique overcomes these shortcomings. Additionally, the plasma itself acts as a perfect method for water purification due to the formation of various reactive chemical species such as radicals ($\text{OH}\cdot$, $\text{H}\cdot$, $\text{O}\cdot$, $\text{HO}_2\cdot$) and molecular species (H_2O_2 , H_2 , O_2). Hence, the use of the combined action of nanoparticles and plasma is a new method for solving water treatment problems.

Keywords: Water purification, plasma technique, nanoparticles, reactive chemical species, molecular species, chemical oxidation processes

4.1 Introduction

Water is an indispensable requirement for life as well as industries. Water is a resource that is becoming increasingly scarce and needs to be sustained,

^{*}Corresponding author: chem.pankaj@gmail.com

globally and locally. Water pollution added to the scarcity of water has become an acute problem in recent times. Groundwater, river, and lakes are polluted by human activities and industrial wastes. One of the most serious problems faced by billions of people today is the availability of fresh water. It has been estimated that 1.2 billion people have no water within 400 m of their dwelling [1]. Purifying and reusing wastewater has been thought of as a solution due to the adverse effects of the discharge pollutants on human and aquatic life. For the treatment of wastewater, traditional methods such as flocculation, carbon adsorption, reverse osmosis and activated sludge process have been used. But these methods cannot be successfully employed in the complete destruction of pollutants [2], and have the further disadvantage of potential secondary pollution [3]. A water treatment process has been developed using chemical oxidation processes (COPs). The most commonly used oxidants are chlorine, chlorine dioxide, chloramines, ozone, and potassium permanganate, which have high oxidation potential and hence prove to be an effective oxidant for water treatment. However, their use for destructive treatment of wastes has been limited because such reagents have usually been expensive, and the addition of large quantities of oxidizing agents to a waste solution may result in a new waste treatment problem involving the reduced products of the oxidizing agents. For example, halogenated organic by-products can form if chlorine or ozone (in the presence of bromide ion) are used, which can have possible health effects [4, 5].

4.2 Water Purification Using Advanced Oxidation Processes (AOP)

There has been a continuing need for the development of effective, cheap and environmentally friendly processes for the disinfection and degradation of organic pollutants in water. Great attention has been focused on the so-called advanced oxidation processes (AOP) that are based on generation of highly reactive species, especially hydroxyl radicals. This is because the hydroxyl radical is a very powerful, non-selective oxidant that has the oxidation potential to completely oxidize organics to carbon dioxide and water [5]. There are a number of methods for generating hydroxyl radicals that may be applied in AOP, such as photochemical and electrochemical oxidation, photolysis of hydrogen peroxide and ozone, Fenton-type reactions, TiO_2 photocatalysis, wet oxidation, sonolysis, and irradiation of water by high energy electron beams or γ -rays. Further, work with the combination of other catalysts, including alumina [6] and zeolites [7], has

also been conducted. However, a common feature of AOP is that the radical production involves a significant expense of energy (either chemical, electrical or radiative). The need for an energy efficient method for production of highly reactive transient species has motivated research on the application of high voltage electrical discharges for water purification. In general, strong electric fields applied to water initiate both chemical and physical processes as ultraviolet radiation, overpressure shock waves and, especially, formation of various reactive chemical species such as radicals ($\text{OH}\cdot$, $\text{H}\cdot$, $\text{O}\cdot$, $\text{HO}_2\cdot$) and molecular species (H_2O_2 , H_2 , O_2).

4.3 Nanoparticle Synthesis Using Plasma and Its Application towards Water Purification

Water purification using nanoparticles is currently an area of intense scientific research. Traditionally, nanoparticles have been produced only by physical and chemical methods. Some of the commonly used physical and chemical methods include ion sputtering, solvothermal synthesis, reduction and sol-gel technique. Iron nanoparticles have attracted considerable interest because of their potential applications in water purification, special magnetic properties in micro- and nanoelectronics, and so on. However, traditional processes usually need a long time or have a high cost. The new method for the preparation of nanoparticles is to use the plasma technique for nanoparticle production. Plasma methods effectively combine the contributions of ultraviolet (UV) radiation, active chemicals and high electric fields that contain free charges (electron, ions), free radicals, excited molecules and UV photon, and generate transient electric fields (Figure 4.1).

Plasma is obtained when gases are excited into energetic states by radio-frequency (RF), MW, or electrons from a hot filament discharge. Plasmas can be categorized into two main groups, i.e., high-temperature plasmas and low-temperature or gas discharge plasmas. A typical classification and parameters of different kinds of plasmas are given in Table 4.1. High-temperature plasma implies that all species (electrons, ions and neutral species) are in a thermal equilibrium state; while low-temperature plasma has been further subdivided into thermal plasma, also called quasi-equilibrium plasma, which is in a local thermal equilibrium (LTE) state, and non-thermal plasma (NTP), also called non-equilibrium plasma or cold plasma (8–11).

Dielectric barrier discharge (DBD) plasma may be a good method to produce iron nanoparticles, but the system needs to be heated. A simple method of producing Fe nanoparticles based on the dissociation of

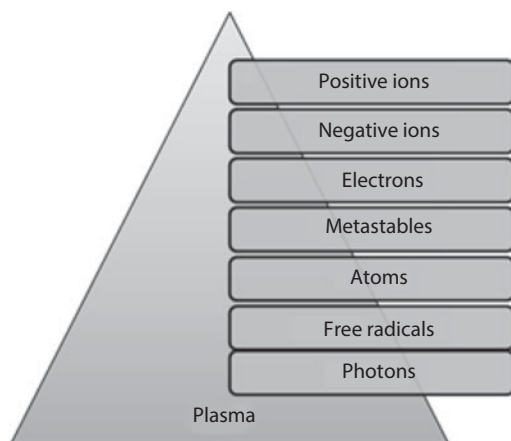


Figure 4.1 Constituents of plasma.

Table 4.1 Classification of plasma.

Plasma	State	Example
High-temperature plasma (Equilibrium plasma)	$T_e \approx T_i \approx T_g, T_p = 10^6\text{--}10^8\text{K}$ $n_e \geq 10^{20}\text{ m}^{-3}$	Laser fusion plasma
Low-temperature plasma		
Thermal plasma (Quasi-equilibrium plasma)	$T_e \approx T_i \approx T_g \leq 2 \times 10^4\text{ K}$ $n_e \geq 10^{20}\text{ m}^{-3}$	Arc plasma, plasma torches, RF inductively coupled discharges
Non-thermal plasma (Non-equilibrium plasma)	$T_e \gg T_i \approx T_g = 300\text{.....}$ 10^3 K $n_e \approx 10^{10}\text{ m}^{-3}$	Glow, corona, APPJ, DBD, MHCD, plasma needle, etc.

ferrocene by a simple single electrode atmospheric cold argon plasma jet has been described by Yu-Tao *et al.* [12]. The system has been driven by a sinusoidal ac-supply with a peak voltage of 0–30 kV and a frequency of 50 kHz. The average size of iron nanoparticles analyzed by scanning electron microscopy has been found to be about 10–30 nm for the gas phase samples, and 30–100 nm for the liquid phase samples. This method has been assumed to be competitive for metal nanoparticle synthesis due to its simplicity and low cost [12].

4.4 Application of Plasma for Water Purification

In addition to the nanoparticle preparation for water purification systems, plasma itself serves as an independent remediation for water purification. Plasma treatment of contaminated water appears to be a promising alternative method in this direction. It has a characteristic feature that all kinds of organic materials, including recalcitrant matter, are decomposed due to active species produced by plasma. Dyes have been one of the major pollutants in water. Due to their high solubility in water, they can be transported over large distances once they are discharged in streams and rivers. Most of the dyes have been found to be resistant to normal wastewater treatment processes. Biotreatment of effluents containing organic dyes has not been effective due to their resistance to aerobic degradation, while by anaerobic degradation carcinogenic aromatic amines have been formed as by-products [13]. An AOP based on the generation of plasma at the gas-water interface has been studied for the degradation of water-bound pollutants like dyes [14]. In a dielectric barrier discharge (DBD) reactor, the electrical discharges take place between electrodes, where at least one of the electrodes is covered with a thin layer of dielectric material such as glass or quartz [15]. Dielectric barrier discharge has been an effective way to produce low-temperature plasma. Media can cover the electrode surface or place between the two electrodes, two electrodes with a high enough voltage, electrode gap of air generated by the breakdown discharge. The discharge forms a large number of fine discharge fast pulse discharge channels, the performance being uniform, diffuse and stable. Dielectric also plays a role in energy storage during the discharge process to delay discharge stability and produce very short pulses, while it can inhibit the production of spark discharge. In the case of the water treatment application of DBD reactors, a layer of water around one of the electrodes acts as a dielectric [16]. Decomposition of the azo dye methyl red in aqueous solution has been performed using a pulsed dielectric barrier discharge in coaxial configuration. The solution has been made to flow as a thin layer on the surface of the inner electrode of the DBD reactor, so that the oxidizing species formed in the discharge can easily penetrate the solution, react with the dye molecules and decompose them. After plasma treatment for 10 minutes, 93% removal of the dye has been found to reach 50 mg/L initial concentration of methyl red in solution and the corresponding removal yield has been found to be 52.5 g/kWh. Lower concentrations of the dye could be removed faster, however, the efficiency has been found to be lower in that case [17]. A DBD reactor has also been designed and tested for the degradation of a model aqueous pollutant crystal violet (CV).

It has been observed that the major reactive species involved in the degradation process include hydroxyl radical, hydrogen peroxide, and ultraviolet, which might cause strong oxidizing effects in the degradation of CV. The effect of various parameters like applied voltage, gas flow rates, concentrations of dye, addition of Fe^{2+} or H_2O_2 , and change in pH has been studied on the synergistic effect during degradation. Mineralization of the dye has been confirmed by total organic carbon (TOC) analyzer and infrared gas analyzer. It has been observed that both degradation efficiency and TOC removal increases with increasing the input energy; however, the energy yield decreases. Formation of H_2O_2 has been confirmed, and it increases with treatment time. The degradation process of the model dye, CV, has been found to obey first-order kinetics, and the rate of degradation strongly increases with the addition of Fe^{2+} due to the catalytic formation of hydroxyl radicals via Fenton-type reactions. The specific advantage of the present process is found to be the mineralization of CV, the highest energy yield up to 86.3 g/kWh [18].

Another method for the removal of dye is contact glow discharge electrolysis (CGDE). Contact glow discharge electrolysis is a novel type of electrochemical process in which plasma is sustained by dc glow discharges between an electrode and the surface of the electrolyte. In CGDE, a continuous dc voltage of around 0.5 kV is applied to a thin wire anode in contact with the water surface while the cathode is dipped in water and isolated from anode through porous glass [19, 20]. Also, H_2 is formed in the gas phase and H_2O_2 in the aqueous phase [20]. A sheath of vapor forms around the anode through which current flows as a glow discharge. Charged species in the plasma (present in the discharge gap or sheath of vapor around the anode) are accelerated due to the steep potential gradient and enter the liquid phase with an energy that may be as high as 100 eV. In the case of contact glow discharges, almost all the species in the discharge zone, i.e., anions, cations and neutrals, heat up, so the plasma generated in the reactors can be called a hot plasma [21]. In the case of both CGDE and DBD reactors, the electrical discharges take place in the gas phase in close proximity to the water surface. They require an intense electric field of the order of 1 MV cm^{-1} for electrical discharge to take place in water. Such a high electric field has been made possible by applying high-voltage pulses of 15–100 kV, usually of positive polarity, with a sharp rise time (a few nanoseconds) and short duration (nano- to microseconds) in a pulsed corona discharge reactor [22]. Oxidative degradation of eight kinds of dyes induced by plasma in aqueous solution has been investigated with contact glow discharge electrolysis (CGDE). It has been demonstrated that these eight dyes (Acridine Orange [AO], Methyl Blue [MB], Rhodamine B [RhB],

Table 4.2 The decolorization rate of different dyes after different discharge times.

Dyes solution	MB	FG	RB	MO	AO	CAS	RhB	GTL
Decolorization 2 min	97.49	92.84	93.63	99.11	97.99	93.86	95.13	94.93
Rate (%) 5 min	99.46	94.41	94.11	99.34	98.47	93.94	97.20	96.55

weak Acid Flavine G [FG], Weak Acid brilliant Red B [RB], Cationic Red [GTL], Chrome Azurol S [CAS], Methyl Orange [MO]) underwent degradation in CGDE, where Fe^{+2} could be utilized to raise the efficiency of the degradation of the dyes. The concentration of Fe^{+2} has been taken as $1\text{ }\mu\text{M}$ and the value of pH has been controlled to 2.14. The decolorization rates of single dyes after degradation are listed in Table 4.2 [23].

Further, another important waste to be purified was the wastewater from the paracetamol factory. Atmospheric pressure plasma in gaseous media was utilized to decompose organic materials in environmental water and kill bacteria in wastewater. Non-thermal plasma combined with activated carbon has been developed to purify wastewater from the paracetamol factory. The effect of discharge time, discharge voltage, initial pH value of wastewater, velocity of the air flow and whether adding activated carbon on the chemical oxygen demand (COD) degradation and decolorization ratio have been investigated. It has been seen that as discharge time was prolonged, the decolorization ratio decreased but the COD degradation ratio increased at first and then reached to a constant value. The ratios of COD degradation and decolorization decreased when the initial pH value increased. With the increase of inputting air flow, it has been found that the COD degradation ratio went up and had an optimal peak value, and then the decolorization ratio declined. The ratio of decolorization and the COD degradation could be improved significantly with the addition of activated carbon [24]. Non-thermal plasma generated in a pulsed dielectric barrier discharge in coaxial configuration has been investigated for the removal of three β -lactam antibiotics (amoxicillin, oxacillin and ampicillin) from water. The discharge was generated at the gas-liquid interface at room temperature and atmospheric pressure, in oxygen. Solutions of concentration 100 mg/L were made to flow as a film over the surface of the inner electrode of the plasma reactor, so the discharge was generated at the gas-liquid interface. The electrical discharge was operated in pulsed regime, at room temperature and atmospheric pressure, in oxygen. Amoxicillin has been found to degrade after 10 min plasma treatment, while the other two antibiotics required about 30 min for decomposition. The evolution of the

degradation process was continuously followed using liquid chromatography-mass spectrometry (LCeMS), total organic carbon (TOC) and COD analysis [25].

In addition to this, the effect of these discharges on bacteria has been studied using water contaminated with *Escherichia coli* or *Bacillus subtilis*, the latter in both the vegetative and spore state. The strongest effect was obtained on *E. coli*. The concentration of *E. coli* was reduced by three orders of magnitude after applying eight corona discharges to the water. The corresponding energy expenditure has been found to be 10 J/cm^3 . The decontamination rate had the largest values at the beginning, and decreased considerably after 15 electrical discharges, reaching a constant residual concentration value of 10^{-4} of the initial concentration. For *B. subtilis* in the vegetative state, it took almost 30 discharges to reach the same result, corresponding to an energy expenditure of 40 J/cm^3 . It has been found that there was no effect on *B. subtilis* spores. Comparisons with the pulsed-electric field (PEF) method indicated that the decontamination efficiency of the PWC method has been found to be slightly higher than that of the PEF method [26]. Atmospheric non-thermal plasma of the gliding-arc type (Glidarc) [27, 28] has been found to be efficient against microorganisms for treatments performed under burning discharge [29, 30], and the inactivation of cells in water continued after the discharge had been switched off [29]. Microbial cells have been found to be killed by contact with water that had first been activated by electric discharges (and so-called plasma-activated water [PAW]) without themselves being subjected to the plasma plume [31]. It has been reported that water treated by various plasma discharges becomes acidic, which leads to antimicrobial effects [32, 33]. Oehmigen *et al.* [34] suggested that the acid created in water is nitric/nitrous acid and the antimicrobial properties observed were due to the result of the synergetic action between H_2O_2 and nitric/nitrous acid [34]. Similar results were obtained by Ikawa *et al.* [35]. The major role of nitrites has been evidenced together with a synergistic effect of nitrates and H_2O_2 and matching acidification. It has been found that reactive nitrogen- and oxygen-based species play an important role in the lethal effect of non-equilibrium atmospheric air-based plasma [36, 37]. The main radical species present in the Glidarc plasma plume were identified as $\text{OH}\cdot$ and $\text{NO}\cdot$ when humid air is the working gas [38]. These radicals are precursors of other active species in water, such as nitrates, nitrites, and hydrogen peroxide [39]. Acidified nitrites and H_2O_2 are known to be less efficient versus yeast than versus bacteria [40, 41], and PAW was found to be more efficient against *H. alvei*, *Staphylococcus epidermidis*, and *Leuconostoc mesenteroides* than against *Saccharomyces cerevisiae* [42].

Water purification by streamer discharge using a stacked Blumlein line pulsed power generator under the water containing pollutants has been investigated. The pulsed power generator consisted of six stacked Blumlein lines to generate high voltage with low charging voltage. The output voltage was applied to a wire electrode to generate streamer discharge in the water and the wire electrode was set on an array of small size holes of an air tube. The air bubbles were injected into the water through the holes to assist the discharge development. Acid Blue 64 solution was employed as a specimen. It was found that the number of discharges increased with the increasing number of holes. Energy efficiency for decolorization of the solution was found to improve by increasing the number of discharges produced simultaneously at the same input energy, whereas the degradation efficiency and energy transfer from the pulsed power generator to the reactor decreased when a streamer-to-arc transition occurred. The solution of 2000 $\mu\text{S}/\text{cm}$ conductivity has been successfully decolorized using the wire electrode placed in the air tube to reduce ohmic loss with high-conductive solution [43]. One single bubble whose average diameter was 2.4 mm was produced in water and the electrical discharge phenomena was observed by both electrical and optical measurements. The experiments were carried out using a pulse-shaped circuit by a Blumline-type pulse forming network (PFN). For the experiments, first the pure water whose conductivity was 2.33 $\mu\text{S}/\text{cm}$ was used [44]. A new type of discharge method (i.e., discharge inside bubbles in water operating in bubbled water) has been proposed in order to use not only ozone but also atomic oxygen, OH, and other radicals generated for effective water treatment [45]; this method was applied to decolor the indigo solution whose original color is blue. The discharge of this method has been thought to begin at the crossing points of mesh electrode, acrylic resin spacer, and bubbling gas (i.e., called a triple junction, where there is a weak point for the electrical breakdown in the electrical power devices). This weak point for the electrical breakdown has been very actively used for the surface discharge [46].

Recently, liquid-phase electrical discharge reactors have been investigated, and it has been found that strong electric fields applied to water (electrohydraulic discharge) initiate both chemical and physical processes. Three types of electrohydraulic discharge systems (pulsed corona electrohydraulic discharge [PCED], pulsed arc electrohydraulic discharge [PAED] and pulsed power electrohydraulic discharge [PPED]) have been employed in numerous environmental applications, including disinfection, chemical oxidation, and the decontamination of sludge [47]. The types of electrohydraulic discharge systems differ in several operational characteristics, as summarized in Table 4.3, due to their different configurations as well as the

Table 4.3 Characteristics of different electrohydraulic discharges¹.

Parameter	Pulsed Corona (PCED)	Pulsed Arc (PAED)	Pulsed Power (PPED)
Operating frequency [Hz]	$10^2\text{--}10^3$	$10^{-2}\text{--}10^2$	$10^{-3}\text{--}10^1$
Current (peak) [A]	$10^1\text{--}10^2$	$10^3\text{--}10^4$	$10^2\text{--}10^5$
Voltage (peak) [V]	$10^4\text{--}10^6$	$10^3\text{--}10^4$	$10^5\text{--}10^7$
Voltage rise [s]	$10^{-7}\text{--}10^{-9}$	$10^{-5}\text{--}10^{-6}$	$10^{-7}\text{--}10^{-9}$
Pressure wave generation	weak	strong	strong
UV generation	weak	strong	weak

¹Data taken from [48].

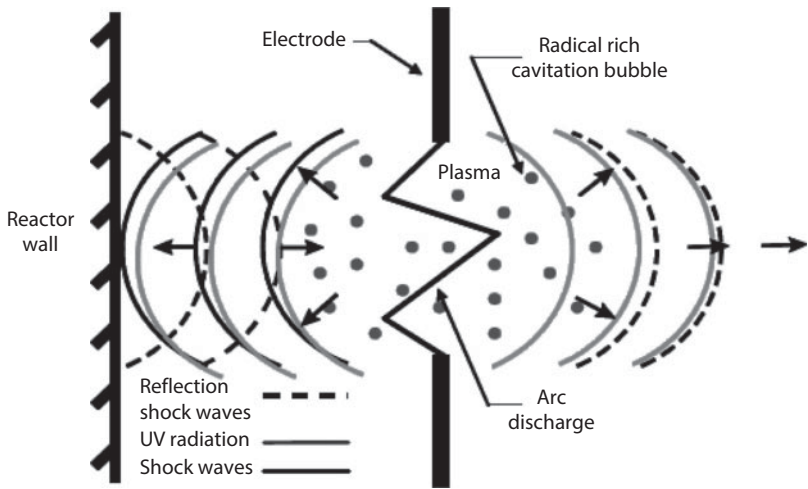


Figure 4.2 Water treatment mechanisms initiated by PAED [49, 58].

different amounts of energy injected into each type of system [48]. Direct plasma technologies (i.e., electrohydraulic discharge) have the potential to be more efficient than either indirect or remote plasma technologies as they capitalize, to some degree, on all of these mechanisms due to the direct application [49, 50].

Figure 4.2 shows the treatment mechanisms initiated by PAED. Preliminary research has indicated that PAED offers advantages over indirect plasma methods in that it can provide comparable or superior

Table 4.4 Comparison of Plasma and Conventional Water Treatment Processes [49, 50].

Target Compounds	Cl/ClO ₂	Ozone	Electron Beam	PCED	PAED	UV-C
Microorganisms	Adequate	Good	Adequate	Good	Good	Good
Algae	None	Partial	None	Partial	Good	Adequate
Urine Components	Adequate	Good	Good	Good	Good	None
VOCs	None	Adequate	Good	Good	Adequate	None
Inorganics	None	Partial	Partial	Adequate	Adequate	None

treatment of microorganisms, algae, volatile organics, nitrogenous municipal waste compounds, and some inorganics [51–57]; these observations are qualitatively summarized as in Table 4.4 [49, 50].

4.5 Combined Action of Nanoparticles and Plasma for Water Purification

The combination of plasma and nanoparticles also proves to be a methodology for water purification. For example, TiO₂ has been extensively used for advanced water treatment and water purification process due to its stable and non-toxic nature, insolubility in water, high photocatalytic activity and abundance. The synergistic effect of pulsed discharge plasma and TiO₂ nanoparticles, and the relation of decoloration efficiency of Acid Orange 7 (AO7) dye wastewater with an amount of TiO₂ particles added into the reaction system, as well as the electric parameters of the discharge system have been investigated. The results obtained further confirm that streamer produced by pulsed discharge plasma induced the photocatalysis of TiO₂ and there has been an optimal TiO₂ amount in the pulsed discharge system for achieving a higher decoloration efficiency of AO7. The synergistic decoloration efficiency of AO7 has been found to be higher in the case of higher input power [59]. Recently, isolated nanoparticles of TiO₂ were deposited over substrates providing larger surface area for the photocatalytic process. Nanoparticles of TiO₂ were deposited using microwave-assisted Electron Cyclotron Resonance (ECR) plasma. Hydrogen plasma has been used to deposit the nanotitanium with the help of chemical sputtering. This nanocrystalline film of TiO₂ has then been used to study the photocatalytic

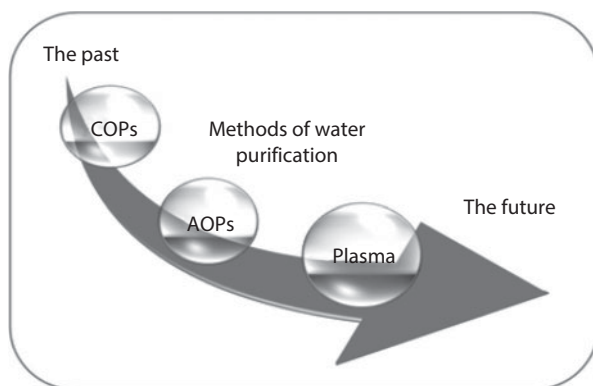


Figure 4.3 Plasma — a promising future approach for water purification.

degradation of methylene blue (MB) in aqueous solution. It has been found to be successful in immobilizing TiO_2 nanoparticles on glass substrate, shows strong adhesion consequently easy for repeated use for photocatalytic degradation, and thus is suitable for water purification [60].

All of these methods have been successfully employed for the complete decomposition of pollutants with the potential to stop further secondary pollution, but plasma has a characteristic feature that can decompose all kinds of organic materials, including recalcitrant matter, due to the active species produced by plasma. Compared to its fellow AOP approaches, non-thermal plasma can achieve greater output and more efficiency. However, applying non-thermal plasma technology in organic wastewater cleaning remains an interesting research area. To enhance the efficiency of bacteria inactivation in the wastewater various methods have been used, such as electrochemical and photocatalytic disinfections, chlorinations, ozonations, Fenton reaction, UV irradiation and electrohydraulics. The use of plasma in water purification attracts attention because of the possibility of obtaining gas jets with a high oxidizing power in compact devices at reasonably low energy inputs. New applications of plasma (Figure 4.3) and nanoparticles in the field of water purification systems have emerged as a promising future approach.

4.6 Conclusion

Many problems that have arisen due to water pollution seem to have been solved by using nanoparticles which have been synthesized at low cost using the plasma technique (such as Fe nanoparticles). Additionally, the

plasma itself acts as a perfect method for water purification due to the formation of various reactive chemical species such as radicals ($\text{OH}\cdot$, $\text{H}\cdot$, $\text{O}\cdot$, $\text{HO}_2\cdot$) and molecular species (H_2O_2 , H_2 , O_2). Further, advanced water treatment problems have also been solved by using a combination of the nanoparticle and plasma approach, where the synergistic effect of pulsed discharge plasma and TiO_2 nanoparticles leads to decoloration of Acid Orange 7 (AO7). Hence, we hereby conclude that all these parameters help in wastewater treatment and in preserving water, which has always been a valuable resource for the growing population.

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