Chapter 15
Application of Plasma-Assisted Advanced Oxidation Processes for Removal of Emerging Contaminants in Water

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Abstract The increasing occurrence of contaminants such as pharmaceutically active compounds, endocrine-disrupting compounds, nanomaterials, surfactants, personal care products is an emerging concern in the water sector. Some of these emerging contaminants are toxic to all life forms, are bio-resistant, and can sustain after primary and secondary wastewater treatment. Conventional water treatment processes are also ineffective in removing these compounds. Advanced oxidation is a potential technique and can degrade and mineralize complex organic molecules. Advanced oxidation processes (AOPs) rely on the in-situ generation of reactive chemical species (RCS) such as hydroxyl radicals for degradation. Most of the RCS with higher oxidizing potential is short-lived, and hence the effective production of these compounds is crucial for this technology's success. The plasma-mediated AOP is an emerging technology superior to other conventional AOPs due to its ability to generate RCS at a controlled rate without using chemical agents. Moreover, the nonthermal plasma can also produce the RCS at controlled temperature and ambient pressure and, therefore, very suitable for commercial-scale processing. The plasma produces a cocktail of reactive species whose collective effect enhances the efficacy of the process. However, before translating this technology to the commercial scale, it is essential to make it affordable and energy-efficient. In this regard, significant studies are being carried focusing on reactor design and its optimization. The chapter reviews the recent developments in plasma-based reactors and their application in the

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degradation of emerging contaminants. The chapter also highlights the current challenges and prospects of plasma-based technology in treating emerging contaminants and various operating parameters influencing the process.

Keywords Emerging contaminant · Plasma · Advanced oxidation · Reactors · Water treatment

15.1 Introduction

The decline in quality and quantity of fresh water is one of the most challenging problems faced by humanity in the twenty-first century. If adequate measures are not taken, these problems would aggravate in the coming decades and threaten the sustenance of human life and the ecosystem. According to a recent WHO report, at least 2 billion people lack access to clean water globally (WHO 2019). The United Nations World Water Development Report (UN WWDR) projected that by 2050, 6 billion people would face freshwater scarcity globally (Boretti and Rosa 2019). Further, the increasing use of chemicals in industrial processing, agriculture, and other daily needs has polluted many freshwater resources and are unavailable for direct use. The growing occurrence of new contaminants in the water is an emerging concern in the water sector. Some of these contaminants of emerging concern (ECs) are toxic to all life forms, are bio-resistant, flame retardants, and can sustain after primary and secondary wastewater treatment. In many cases, traces of these pollutants can pose serious health concerns (Rout et al. 2021). The widespread occurrence of ECs in water and their chemical toxicity demands the development of effective, efficient, and eco-friendly management and treatment strategies.

The NORMAN project, which enables the exchange of information and validation of ECs under the European Commission, has listed 1036 ECs by 2016 and is further labelled into 30 classes based on their origin and type (Dey et al. 2019). The ECs include pesticides, pharmaceutical active compounds (PhACs), disinfection by products, personal care products (PCPs), food additives, artificial sweeteners, flame retardants, endocrine-disrupting compounds (EDCs), nanomaterials, and surfactants (Lin et al. 2020). These contaminants exhibit very different physical and chemical properties, thus challenging to detect and remove from water. Many conventional treatment methods such as coagulation and flocculation, filtration, membrane treatment, and chlorination are ineffective in addressing ECs in water (Mondal et al. 2018). The high chemical usage and sludge production, biofouling, high energy requirements, and/or difficulty in operation and maintenance further limit their use. The frequent reports on the presence of ECs in treated water also support that the conventional treatment techniques are ineffective and cannot guarantee complete removal of ECs in water (Khetan and Collins 2007; Magureanu et al. 2015).

Advanced oxidation process (AOP) is a promising technique and can provide a potential route to degrade and mineralize complex organic molecules. The process relies on the in-situ generation of reactive chemical species (RCS) such as hydroxyl

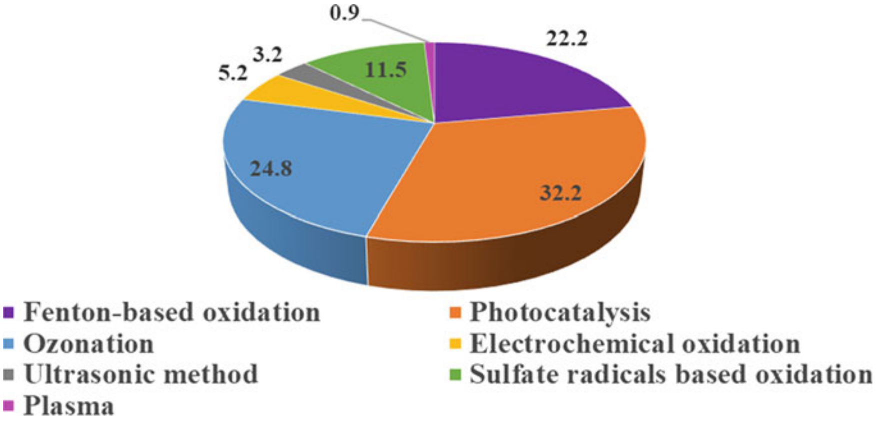

Fig. 15.1 The percentage number of articles published related to the application AOPs for the degradation of ECs in water and wastewater. This chart is made based on the number of articles published between 2000 and 2021 as per the scopus database

radicals, atomic oxygen, ozone, perhydroxyl radical, and hydrogen peroxide. These RCS can degrade or mineralize the ECs into less toxic, simple, and treatable compounds. Some existing AOPs include the Fenton and photo-Fenton processes, photocatalysis, sonolysis, catalytic wet air oxidation processes, and combinations thereof (Ribeiro et al. 2015). However, limitations like the addition of excess chemicals, catalyst regeneration, sludge generation, and low penetration efficiency of UV photons, formation of toxic byproducts, the negative influence of water quality parameters persist (Klamerth et al. 2010). Figure 15.1 summarizes the studies from 2000 to 2021 utilizing various AOPs to treat ECs in water.

Plasma-assisted AOP is an emerging technology that can simultaneously generate a wide variety of short and long-lived RCS at a controlled rate in an ambient environment. The technology also demonstrates good potential for commercial-scale processing (Pankaj et al. 2018). The significant attraction lies in the ability of plasma to produce a cocktail of reactive species without any additional chemical/catalyst (Gorbanev et al. 2018). The collective effect of plasma-generated RCS enhances the efficacy of the process. However, before translating this technology to the commercial scale, it is essential to make it affordable and energy-efficient. Significant efforts are being paid in this direction by developing novel reactors and hybridizing plasma technology with other approaches (Snoeckx and Bogaerts 2017). The following sections will outline the recent development in plasma-based reactors and their application in the degradation of ECs in water. The chapter will conclude by providing the current challenges and prospects of plasma-based technology in treating emerging contaminants.

15.2 Emerging Contaminants in the Aquatic Environment: Origin, Health Effects, Occurrence, Fate, and Transport

The significant levels of ECs in the aquatic system have become a serious global concern. They can enter the aquatic environment from the point or non-point sources, including wastewater treatment plants (WWTP), agricultural activities, surface runoff, air transportation and precipitation, adsorption onto soil, and sewage biosolids. Inadequate information on interaction mechanisms and toxic effects of ECs at sub-lethal concentrations pose a severe threat to environmental and public health (Brausch and Rand 2011). A few examples of ECs detected in surface and groundwater of different countries during 2010–2021 are shown in Table 15.1.

ECs like propranolol, ketoprofen, naproxen, E2, gemfibrozil, EE2, and ibuprofen are susceptible to photolysis (Khetan and Collins 2007). Recent studies report the occurrence of metabolites such as 3-hydroxycarbofuran, acephate, clethodim, acetochlor ethane sulfonic acid, and acetochlor oxanilic acid in water bodies. These metabolites are listed in contaminant candidate list 3 (Epa 2009). The human body cannot absorb drugs completely, and a good portion of the drug is often excreted with its metabolites. For instance, ibuprofen was found to be present as a parent chemical (1%) in surface water along with some of its metabolites like (+)-2–40-(2-carboxypropyl)-phenyl propionic acid (37%), (+)-2–4′-(2-hydroxy-2-methylpropyl)-phenyl propionic acid (25%) and conjugated ibuprofen (14%) (Kasprzyk-Hordern et al. 2008). Numerous metabolites are generated through various biodegradation pathways, and their toxicity is not often studied. Sorption of ECs onto dissolved organic matter is also responsible for the presence of ECs in an aqueous medium (Petrie et al. 2015).

Globally, only limited studies are available on the toxicity of ECs on humans and the environment. ECs like bisphenol A, organophosphate and brominated flame retardants, perchlorate, phthalate, polycyclic siloxanes, and triclosan can adversely affect human health (Covaci et al. 2012). Bisphenol A, a universal plasticizer, is responsible for severe endocrine disorders in salivary glands, thyroid, and men's genetic systems. Hormonal imbalance due to bisphenol A can lead to cancer (Bolong et al. 2009). Musk compound like xylol can indirectly affect the human tissues and leads to brain damage (Bolong et al. 2009; Lei et al. 2015). Prolonged exposure to herbicides like atrazine destroys adrenal glands and impairs steroid hormone metabolism (Debnath et al. 2019). About 70% increase in streptomycin, penicillin and tetracycline-resistant bacteria was identified in a dairy farm's soil manure (Qiao et al. 2011). Intake of phthalates leads to increased insulin resistance, abdominal obesity, and neurobehavioral disorder (Dewalque et al. 2014). EDCs like nonylphenol can cause feminization of aquatic organisms and hinder the hormonal system (Saidulu et al. 2021). Analysis of metabolites, in-depth survey of ECs existence, toxicology studies, risk assessment, mitigation measures, and awareness in public is required to plan appropriate mitigative measures and reduce the health impacts from ECs (Fig. 15.2).

Table 15.1 Emerging contaminants detected in surface and groundwater bodies

S. No.	Type of ECs	Name of ECs	Country	Type of water	Detection limit	References
1	Pesticides	Dichloro-diphenyl-trichloroethane	India	Surface water	13.5 μg/L	Nath et al. (2014)
		1*H*-1,2,4-triazole	USA	Ground water	20–430 ng/L	York (2014)
		Acetamiprid	Saudi Arabia	Surface water	1408 ng/L	Picó et al. (2021)
		Metalaxyl	China	Surface water	9.86 ng/L	Zhang et al. (2020)
		Atrazine	Argentina	Surface water	0.64–1.4 μg/L	Gerónimo et al. (2014)
		DEET	Zambian	Ground water	70–330 ng/L	Sorensen et al. (2015)
2	PhACs	Theophylline	Portugal	Surface water	184 ng/L	Gonzalez-Rey et al. (2015)
		Lincomycin	Canada	Surface water	143 ng/L	Kleywegt et al. (2011)
		Ibuprofen	India	Waste water	26.45 μg/L	Singh et al. (2014)
		Metoprolol	Spain	Surface water	380 ng/L	Huerta-Fontela et al. (2011)
		Norfloxacin	Saudi Arabia	Surface water	2763 ng/L	Picó et al. (2021)
		Carbamazepine	France	Drinking water	41.6 ng/L	Vulliet and Cren-Olivé 2009)
3	PCPs	Triclosan	India	Surface water	5160 ng/L	Gani and Kazmi (2016)
		Caffeine	Saudi Arabia	Surface water	12,374 ng/L	Picó et al. (2021)
		Benzophenone-3	Brazil	Drinking water	115 ng/L	Silva and Emídio (2015)
		Antipyrine	China	Drinking water	19.46 ng/L	Qiao et al. (2011)
		Naproxen	Spain	Surface water	278 ng/L	Carmona et al. (2014)
		4MBC	Germany	Surface water	235–2592 ng/L	Montes-Grajales et al. (2017)
4	EDC	Bisphenol F	Japan	Surface water	90–2850 ng/L	Yamazaki et al. (2015)
		Bisphenol S	India	Surface water	58–2100 ng/L	Yamazaki et al. (2015)
		Nonylphenol-tri-ethoxylate	Vietnam	Surface water	38–160 ng/L	Williams et al. (2019)

(continued)

Table 15.1 (continued)

S. No.	Type of ECs	Name of ECs	Country	Type of water	Detection limit	References
		Bisphenol A	Malaysia	Tap water	66.4 ng/L	Wee et al. (2020)
		Methyl paraben	Egypt	surface water	MDL-1780 ng/L	Radwan et al. (2019)
		4-nonylphenol	China	surface water	698–14,936 ng/L	Gong et al. (2016)
5	Flame retardants	Polybrominated diphenyl ether	Canada	Waste water	29–1000 ng/L	Kim and Kannan (2018)
		TCPP (tris (1-chloro-2-propyl) phosphate)	Saudi Arabia	Surface water	387 ng/L	Picó et al. (2021)
		TCPP (tris (1-chloro-2-propyl) phosphate)	China	Surface water	1.14–386 ng/L	Thi et al. (2016)
		Tris(2-butoxyethyl) phosphate (TBOEP)	USA	Surface water	2.53–366 ng/L	Kim and Kannan (2018)
		Tris(2-chloroethyl) phosphate (TCEP)	Korea	Drinking water	0.7–1400 ng/L	Lee et al. (2016)
		Polybrominated diphenyl ether	UK	Surface water	0.295 µg/L	Cristale et al. (2013)

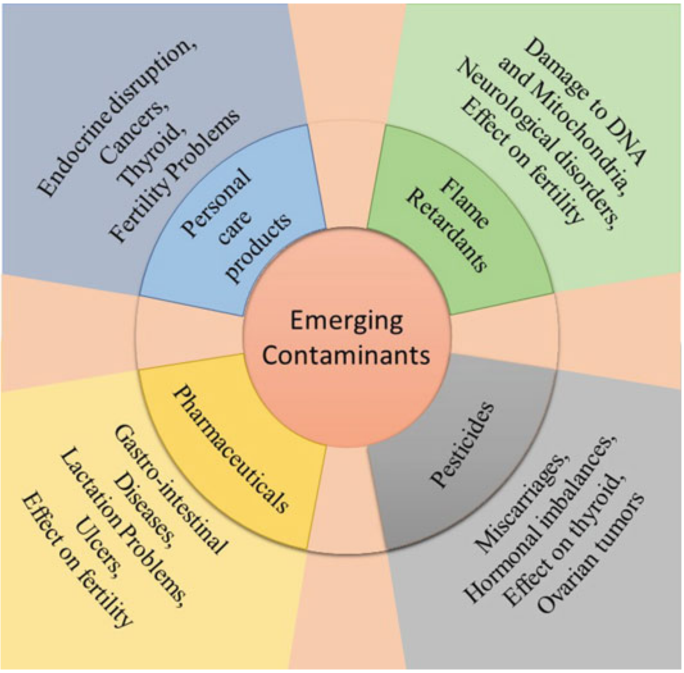

Fig. 15.2 Summary of effects of emerging contaminants on human health. The image is adapted with modifications from Rathi et al. (2021) with the permission from Elsevier

15.3 Advanced Oxidation Process for Removal of ECs in Water

Owing to the widespread occurrence and possible toxic effects on humans and other living organisms, removing ECs from water is imperative. However, most conventional treatment technologies are ineffective in removing ECs due to their complex nature and various physicochemical properties. Even popular technologies such as adsorption and membrane filtration are not attractive due to ECs complex nature and generation of contaminated sludge. In this context, developing more reliable and effective technologies is the need of the hour. The recent advancement in AOP promises a potential treatment option for removing ECs in water. The AOP can degrade and mineralize the ECs to simple carbon dioxide, water, and inorganic acids or ions. The technique utilizes ROS like hydroxyl radicals (•OH), hydrogen peroxide (H_2O_2), ozone (O_3), nascent oxygen (•O), and hydroperoxyl (•HO_2) for degradation. Among these, the •OH is the most reactive radical with an oxidation potential of 2.8 V. The highlights of its properties are shown in Fig. 15.3 (Brillas et al. 2009).

Most of the higher oxidizing potential radicals are short-lived; therefore, in-situ production of RCS is a prime requirement for efficient processing. Many AOPs

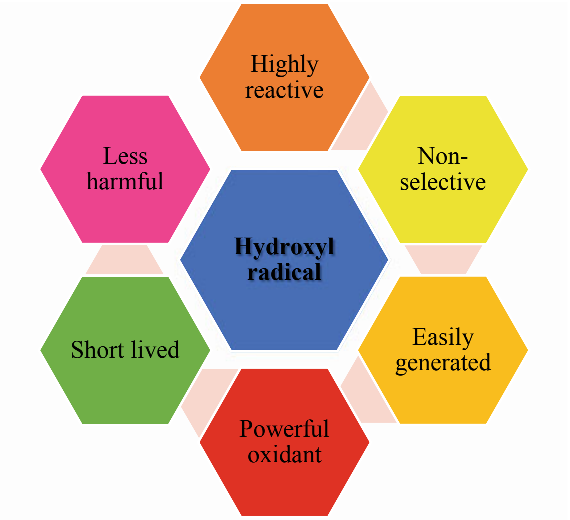

Fig. 15.3 Crucial properties of hydroxyl radicals

such as the Fenton process, heterogeneous photocatalysis, sonolysis, and ozonation along with hybridized techniques such as O_3/photo-Fenton process, O_3/UV, O_3/H_2O_2, UV/H_2O_2, catalytic ozonation, plasma treatment, and O_3/UV/H_2O_2 are developed. A description of pathways of radical production in these processes is shown in Table 15.2.

Though AOPs are promising, the radical generation pathway, water matrices, and experimental conditions significantly influence the degradation/mineralization of ECs. Despite higher oxidation potential, low mineralization values are reported depending on the persistence of the pollutant. For example, Zhao et al. achieved complete degradation of 2.5 μM indomethacin with ozone dosage of 2–35 mg/L in 7 min (Zhao et al. 2017). However, only 50% mineralization was achieved even after extending the treatment to 30 min. Similarly, 0.38 mM of propranolol was degraded entirely in 8 min using the ozone dosage of 0.47 mM. Still, the total organic carbon (TOC) value remained close to 5% even after 60 min exposure with ozone dosages of 3.54 mM (Dantas et al. 2011). Even though ozonation is a proven technique, the limitations like inefficient mass transfer and the formation of secondary intermediates restrict operational capability (Shin et al. 1999). The presence of organic matter in WWTP effluent or surface waters can significantly demand higher ozone dosage and reduce the process efficacy (Almomani et al. 2016).

Fenton and photo-Fenton are extensively studied AOPs. Though the process is promising, large chemical requirements, the generation of chemical sludge, and the high sensitivity of the process are major concerns (Diya'uddeen and Daud 2012). Photocatalysis is also a promising AOPs suitable for removing complex organic molecules in water. However, catalyst fouling, slow rate of degradation, and low penetration of UV photons are some of the disadvantages of photocatalysis despite the less operational cost. The plasma-assisted AOP is an emerging approach that can

Table 15.2 Chemical pathways showing the radical production in various AOPs. This is adapted with modification from Salimi et al. (2017)

Process	Reaction	References
H_2O_2/O_3	$H_2O_2 \leftrightarrows H^+ + HO_2^-$	Gligorovski et al. (2018)
	$HO_2^- + O_3 \rightarrow \bullet OH + O_2 + O_2^{\bullet -}$	
H_2O_2/UV	$H_2O_2 + h\,\nu \rightarrow 2\bullet OH$	Cruz et al. (2013)
UV/H_2O_2/O_3	$O_3 + OH^- \rightarrow \bullet OH$	Joseph et al. (2009)
	$3O_3 + h\,\nu \rightarrow 2\bullet OH$	
	$H_2O_2 + h\,\nu \rightarrow 2\bullet OH$	
	$H_2O_2 + O_3 \rightarrow 2\bullet OH$	
	$H_2O_2 + O_3 + h\,\nu \rightarrow \bullet OH$	
Fenton	$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^- + \bullet HO$	Asghar et al. (2015)
Ultra sound (US)	H_2O + US $\rightarrow \bullet OH + OH^-$	Sathishkumar et al. (2016)
	H_2O + US $\rightarrow (1/2)H_2 + (1/2)H_2O_2$	
Ultra sound/Fenton	$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^- + \bullet HO$	Bagal and Gogate (2014)
	$H_2O_2 + Fe^{3+} \rightarrow Fe^-OOH^{2+} + H^+$	
	Fe^-OOH^{2+} + US $\rightarrow Fe^{2+} + \bullet HOO$	
	$Fe^{2+} + \bullet HOO \rightarrow Fe^{2+} + H^+ + O_2$	
	$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^- + \bullet HO$	
US/advanced Fenton	$Fe^0 + 2H^+ \rightarrow Fe^{2+} + H_2$	Bagal and Gogate (2014)
	$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^- + \bullet HO$	
	H_2O_2 + US $\rightarrow 2\bullet HO$	
	$H_2O_2 + \bullet HO \rightarrow H_2O + \bullet HO_2$	
	$Fe^{3+} + H_2O_2 \rightarrow Fe(OOH)^{2+} + H^+$	
	$Fe(OOH)^{2+}$ + US $\rightarrow Fe^{2+} + \bullet HO_2$	
	$Fe^0 + 2Fe^{3+} \rightarrow 3Fe^{2+}$	
	$Fe^{3+} + HO_2^\bullet \rightarrow Fe^{2+} + H^+ + O_2$	
Sono-Fenton	$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + \bullet OOH + H^+$	Dindarsafa et al. (2017)
	$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + \bullet OH + H^-$	

(continued)

Table 15.2 (continued)

Process	Reaction	References
	$H_2O + US \rightarrow \bullet OH + \bullet H$	
	$\bullet OH + \bullet OH \rightarrow H_2O_2$	
Photocatalysis	$TiO_2 \rightarrow {}^{h\nu}e^- + H^+$	Mahamuni and Adewuyi (2010)
	$TiO_2(H^+) + H_2O_{ad} \rightarrow TiO_2 + \bullet HO_{ad} + H^+$	
	$TiO_2(H^+) + H\ O_{ad} \rightarrow TiO_2 + \bullet OH_{ad}$	
	$TiO_2(H^+) + RX_{ad} \rightarrow TiO_2 + RX^{\bullet +}{}_{ad}$	
Plasma	$H_2O + e^- \rightarrow \bullet OH + H\bullet + e^-$	Itikawa and Mason (2005)
	$H_2O + e^- \rightarrow 2e^- + H_2O^+$	
	$H_2O^+ + H_2O \rightarrow \bullet OH + H_3O^+$	
	$\bullet OH + \bullet OH \rightarrow H_2O_2$	
	$\bullet OH + H_2O_2 \rightarrow H_2O + \bullet HO_2$	

offer an efficient and eco-friendly way to degrade and mineralize ECs in a relatively lower treatment time. The plasma-assisted AOP can produce short and long-lived RCS simultaneously, and its combined effect can significantly enhance the efficacy of the process (Nippatla and Philip 2019). The plasma can also be nonthermal and can produce RCS at ambient pressure, which is very suitable for commercial-scale processing. It can also eliminate the use of any additional precursor for the generation of RCS. The following section discusses the plasma-assisted AOP technique in detail and the recent studies on the degradation of emerging contaminants. The current challenges and prospects of plasma-based technology in treating emerging contaminants and various operating parameters influencing the process are also discussed.

15.4 Introduction to Plasma

As shown in Fig. 15.4, the phase transition from the solid-state to the gaseous state of matter can occur by increasing the system's internal energy. The addition of energy in the gaseous system enhances inelastic collisions that can prompt ionization and dissociation processes. Under appropriate conditions, this shall lead to the transition from the gaseous phase to the plasma, often referred to as the fourth state of matter. For the first time (1928), the "Plasma" ("mouldable substance" or "jelly" in Greek) (Fridman 2008) term was coined by well-known chemist Irving Langmuir to describe the ionized gas system, which is macroscopically neutral and exhibits collective behaviour. All ionized systems cannot be classified as plasma, and the plasma state must fulfil a set of criteria to ensure bulk quasi properties. One of the most exciting properties of plasma is bulk quasi neutrality. In simple terms, quasi neutrality means

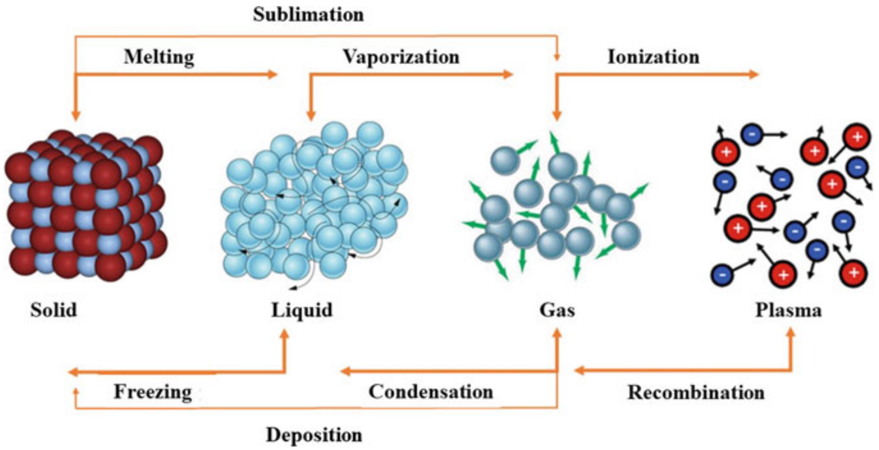

Fig. 15.4 Phase transition diagram highlighting the various states of the matter and underlying processes

that the density of negatively charged species is equal to the density of positively charged species. To achieve quasi neutrality, the smallest dimension (L) defining the volume occupied by the plasma should be much larger than the Debye length (λ_d), i.e. ($L \gg \lambda_d$). The plasma is a collection of charged particles. Therefore, the effective potential due to charge inside plasma at a point is screened Coulomb potential. This screening can be parameterized through the Debye length. The self-screening effect of plasma limits the influence of any given charge particle within the Debye sphere (a sphere with a radius λ_d) and allows quasi neutrality in bulk plasma. The λ_d depends on species temperature and density and can be expressed in Eq. 15.1 (Mozetiˇc et al. 2019).

$$\lambda_d = \sqrt{\frac{\varepsilon_0 k_B T_e}{e^2 n_e}} \tag{15.1}$$

For further details of basic properties, the readers are requested to refer the standard textbooks on introduction to plasma (Fridman 2008; Chen 2003). The plasma state can be characterized through some basic plasma parameters associated with the temperature and density of plasma constituents. Since the electron-induced kinetics is dominant in low-temperature plasma (LTP), the electron temperature (T_e) and electron density (n_e) are crucial plasma parameters. Another key parameter is the gas temperature to ensure the non-thermal nature of the plasma during processing. However, the most exciting feature of the plasma state is that it provides an environment where electrons, positive ions, photons, reactive species (excited molecules and atoms), and radicals can coexist simultaneously. This makes plasma a very suitable candidate for a wide range of applications, including material deposition, etching, surface modification, waste management, material processing, plasma cutting, plasma spraying, biomedical, energy, etc. (Samukawa et al. 2012). A more detailed discussion on various plasma applications is available elsewhere, and this chapter provides the application of plasma for the degradation of emerging contaminants in water.

15.4.1 Classification and Definition of Plasma

Plasma can be classified based on the fundamental plasma parameter related to the temperature (or average energy) of species such as ions, neutrals, and electrons. If the average thermal energy of ions (T_i), neutrals (T_g), and electrons (T_e) are of a similar order, then they can be classified as hot plasmas (HP). The stars and plasma generated in fusion reactors come under HP. Most laboratory plasmas fall in the LTP category, where electron temperature is higher than other heavy mass species' temperatures. The LTPs significantly deviate from equilibrium nature and can also be sustained at ambient pressure and temperature. In few cases where the electron density is very high such as welding or laser-induced plasmas, Local Thermal Equilibrium (LTE) may exist (Mal 2019; Rezaei et al. 2020). Further, in some plasmas, such as surface wave discharges or rotating gliding arcs, the partial local thermal equilibrium may also exist for higher-lying levels due to lower energy spacing (Ananthanarasimhan et al. 2021). Table 15.3 presents the classification of the plasmas commonly described in the literature (Fridman 2008; Yuri et al. 1997).

In recent years, the application domain of non-thermal plasma (NTP) assisted processing is significantly increasing. The major attraction lies in its ability to generate reactive species (ions, metastable, radicals, neutrals) at a relatively lower temperature and often close to room temperature (Shashurin et al. 2008). The generation of such plasmas is challenging as the operating condition are relatively very limited. However, over the years, various schemes to sustain the NTP are proposed.

Table 15.3 The classification of plasma-based on the temperature of its constituents. The information is adapted with modification from Lee et al. (2017)

	Low-temperature		High-temperature
	Non-thermal plasmas	Thermal plasmas	
Species temperature (T_x)	T_i, $T_g \ll T_e$, $T_g \ll 500$ K	$T_i \approx T_g \approx T_e$ $T_g < 10^5$ K	$T_i \approx T_g \approx T_e$ $T_g \gg 10^5$ K
Species densities (n_e)	$\approx 10^{20}/m^3$	$>10^{20}/m^3$	$>10^{20}/m^3$
Classification	Non- thermal plasma (NTP)	Local thermal equilibrium (LTE)	Thermal equilibrium
Examples	DBD's, Corona plasma, glow discharge	Arc torch, laser-induced plasma	Stars, fusion reactors

The following section will overview this NTP generation scheme for water treatment application.

15.4.2 Brief Note of Nonthermal Plasma/Discharges

The critical factor in sustaining the NTP is to limit the collisions between low mass (electron) and heavy mass (ion, neutral) species (Foster 2017). The mass difference leads to different mobilities, and plasma can simultaneously attain different equilibrium temperatures for other mass species. It is worth mentioning that this non-equilibrium regime is susceptible to plasma environment and operating conditions. Therefore, for water treatment application one needs to design the plasma generation scheme carefully so that plasma water interaction (direct or indirect) does not disturb the nonthermal characteristics of the plasma. Typically, a high voltage signal is applied between the two electrodes. When the voltage reaches above the inter-medium breakdown potential, an avalanche of electrons is being produced in the form of streamers. Once the streamers are generated, there are high chances of the thermal runaway due to the electric field application for long durations; this leads to the transition of non-thermal to the thermal regime. Here, the collision frequency of electron-neutral/ion increases and causes gas heating phenomena (Foster 2017; Wang et al. 2013). Thus, through such collisions, a gradual merge can be observed in the electron and gas/ion temperatures. To avoid the transition from non-thermal to thermal plasma or from streamer to arc discharge transition, one can employ a variety of techniques: (1) use of dielectric barriers, (2) shot pulse voltage signal, (3) gas composition, (4) flow rate, (5) variation in frequency, etc. (Bruggeman et al. 2016; Vanraes and Bogaerts 2018).

Over the years, numerous plasma reactors for water treatment applications were reported. Therefore, the classification of these reactors is challenging (Vanraes and Bogaerts 2018). Various criteria can be used to classify these plasmas. For example, based on typical current–voltage characteristics, these plasmas can be classified into three discharge regimes viz. Townsend, glow, and arc discharges (Yuri et al. 1997). Similarly, one can also classify these plasmas based on the applied signal such as DC, low frequency (kHz range), radiofrequency (MHz range), microwave (GHz range), or pulsed wave discharges. In context to plasma water treatment application, the most popular way is to classify the plasma based on the plasma interaction with the water surfaces. In this approach, the plasma can be divide into three categories: (1) Direct (discharge inside the water), (2) Indirect (discharge at the surface of the water without contact), and (3) Hybrid (multi-phase) discharges (Bruggeman et al. 2016). Most of the plasmas employed for the AOP process in water treatment are non-equilibrium discharges that are sustained at high-pressure conditions. Therefore, the present discussion will be limited to such discharge.

In the direct discharge or liquid-phase discharge, the discharge is sustained inside the water. To maintain such discharge, a very high electric field of the order of ~1000 kV/cm is applied between the electrodes. The liquid-phase discharges are

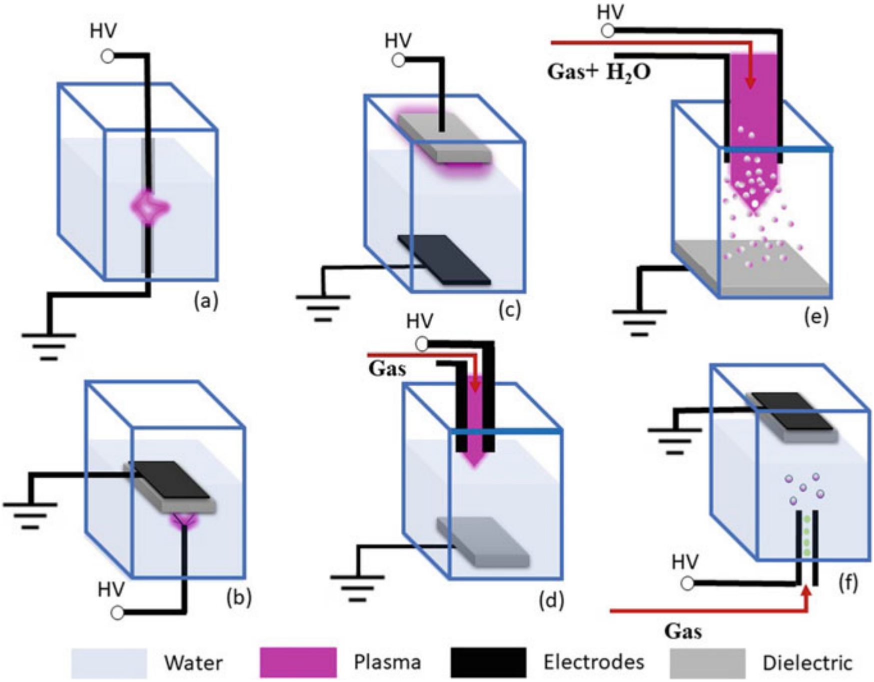

Fig. 15.5 Illustration of different reactors based on plasma water interactions: **a**, **b** direct discharge (liquid phase discharge); **c**, **d** indirect discharge (gas phase discharge); **e**, **f** hybrid discharge (multi-phase discharge). The image is adapted with modifications from Vanraes et al. (2016) (open source subjected to creative common attribution 3.0 license)

energy-intensive, and to limit the heating and electrode erosion, the short pulses of micro-second order are applied. As shown schematically through Fig. 15.5 (a), (b), the pin-to-pin and pin-to-plate are the most common schemes to generate liquid phase discharges. The discharge is highly transient and often comes under the steamer or corona discharge category. Compare to other classes, the electron density in liquid-phase discharge is at least 5 to 6 orders (10^{24}–$10^{26}/m^3$) higher and also produces 3 to 4 times higher radical density (~10^{24} m^{-3} s for OH radical) (Bruggeman et al. 2016; Locke and Thagard 2012).

In the indirect or gas-phase discharge, the discharge is sustained in contact with water. As shown schematically in Fig. 15.5 (c), (d), the discharge is often created between the electrode plate and water surface or remotely generated above the water surface without direct contact. Normally for water treatment applications, the employed gas-phase discharges are based on dielectric barrier discharge concepts. However, spark or corona discharges can also be operated using pin-water electrode configuration. In recent years, remote discharges such as the atmospheric pressure plasma jets (APPJs) and surface dielectric barrier discharge are becoming popular choices because the discharge characteristics are independent of the properties of the treated water sample. The gas-phase discharge can be sustained by a wide range of high voltage signals ranging from DC, kHz, MHz to GHz frequencies (Foest et al. 2005). Compare to liquid-phase discharges, the gas phase discharges consume low power. Consequently, the electron density is of the order of 10^{18}–10^{21} m^{-3} and the radical density of the order of 10^{19}–10^{23} m^{-3} s (Bobkova et al. 2014; Wang et al. 2021a).

In the hybrid or multi-phase discharge category, the widely used approach is to sustain the discharge in the dispersed gas (Bubbles) inside the liquid. The water treatment can also be achieved by dispersing the water droplets (aerosols) in the gas phase plasmas. The schematic representation of both the approaches is shown in Fig. 15.5 (e), (f). The discharge is formed inside the bubbles by creating a sufficiently high electric field gradient around the bubbles. The studies reported that the discharge could sustain for ~100 ps before the bubble breaks off. The discharge is of steamer type and initiated at the bubble water interface. The electron density in hybrid discharges is reported as high as 10^{22} cm^{-3} (Bruggeman et al. 2010). The reactive species are mainly being produced in gas-phase in both hybrid as well as indirect discharges. One of the major advantages of the hybrid discharge approach is that reactive species are immediately dispersed in the water. The highest oxidizing potential species such as OH radicals are short-lived, and therefore the immediate dispersion can significantly enhance the efficacy of water treatment process.

It is worth mentioning that only brief details of the reactors and underlying discharge mechanism are given here and for further information, the readers are direct to published articles (Bruggeman et al. 2016; Vanraes and Bogaerts 2018). In fact, over the years, hundreds of geometries under the categories mentioned above are reported to address the engineering aspect as well as to improve performance (Samukawa et al. 2012; Vanraes et al. 2016).

15.4.3 Plasma Induced Reactive Species

The advanced oxidation process is achieved by the plasma water interaction, which is the source of the generation of various reactive chemical species (RCS). One of the significant advantages is that the RCS generation does not require any additional consumables. Figure 15.6 schematically shows the generation of RCS through plasma-water interactions (Samukawa et al. 2012). The RCS generated immediately after collision with electrons from the plasma is called primary species. Electrons (e^-), excited neutrals (M^*), ion (M^+), neutrals (M), radicals (•OH, N, O), UV photon, etc. are primary species produced in plasma-gas phase interaction. These species usually have less lifetime. For example, the lifetimes of OH radicals, NO, and O_2* are 2.7 μs ~1.2 μs, 1.4 μs, and ~1.3 μs respectively (Attri et al. 2015). However, due to higher reactivity rates, these species get quickly consumed, and secondary species are formed such as H_2O_2, NO_2, NO_3, and O_3. The secondary species have relatively long lifetimes and are dissolved in water media. The secondary species radicals further oxides to form tertiary species such as O_3, H_2O_2, NO_3 that can last

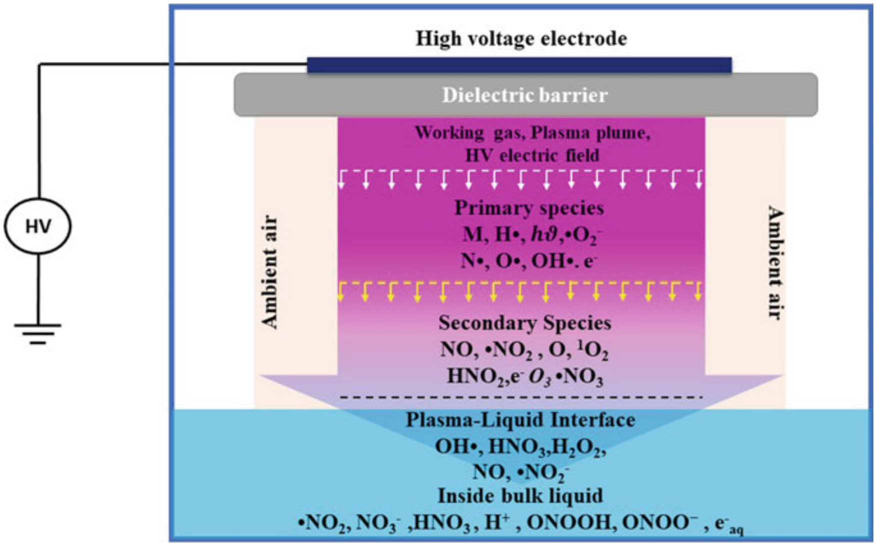

Fig. 15.6 Schematic representation of the formation of reactive chemical species through plasma-water interactions. The image is adapted with modifications from Barjasteh et al. (2021)

up to several days (Kondeti et al. 2018). The RCS generated through plasma water interaction either in gas-phase or inside liquid can broadly be classified into reactive oxygen species (ROS) and reactive nitrogen species (RNS). Sometimes both classes can simultaneously be referred to as reactive nitrogen and oxygen species (RONS).

15.4.3.1 Reactive Oxygen Species

Diatomic oxygen (O_2) contains two unpaired electrons in the valance shell, limiting its scope to react and degrade ECs in water. An electron or energy transfer process can convert O_2 into ROS, atoms or molecules with at least one unpaired electron. These include both radicals and non-radical such as hydroxyl radical (•OH), hydrogen peroxide (H_2O_2), singlet oxygen ($^1O_2$), superoxide anion ($\bullet O_2^-$) and ozone (O_3). (Nosaka and Nosaka 2017). Figure 15.7 represents an overview of various possible reaction routes for ROS production during the plasma-assisted advanced oxidation process. A few of the significant ROS species are discussed below.

Hydroxyl radical (•OH): Hydroxyl radicals are the most potent ROS with the oxidation potential value of 2.8 V, which is only next to the oxidation potential of fluorine (3.03 V). The high oxidising potential and short lifetime (~1 ns) allow OH radicals to react with most organic molecules, including ECs in water and degrade them. In the plasma-mediated treatment of water, the OH radicals are produced by the interaction of high energetic plasma electrons with water molecules by dissociating them into •OH and H• and relatively less energetic secondary electrons. Ozone

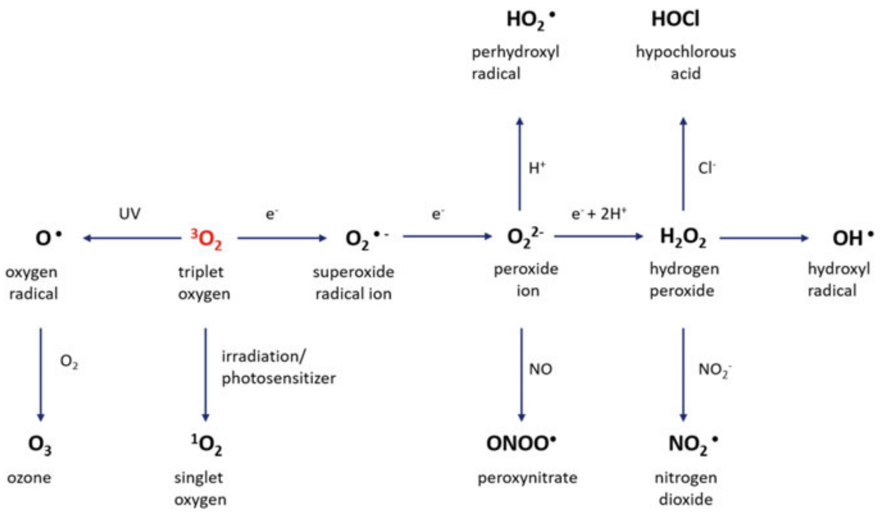

Fig. 15.7 Overview of the mechanisms of RCS production during plasma assisted advanced oxidation process. Reprinted from Mitra et al. (2019)

generated during the plasma process can react with water molecules and produces diatomic oxygen and OH radicals. Plasma also produces UV light along with a variety of RCS. This UV light lies in VUV to UV region and break H_2O_2 into two OH radicals (Mitra et al. 2019).

$$H_2O \; + \; e^- \rightarrow \; \bullet OH \; + \; \bullet H \; + \; e^- \tag{15.2}$$

$$3O_3 + H_2O \rightarrow 2 \bullet OH + 4O_{2^-} \tag{15.3}$$

$$H_2O_2 + h\nu \rightarrow 2 \bullet OH \tag{15.4}$$

Superoxide anion ($\bullet O_2^-$): The monovalent reduction of diatomic oxygen (O_2) by electron impact ionisation produces a superoxide anion ($\bullet O_2^-$). It is one of the few molecules among the ROS that are considered radical and anion with low reactivity. Due to its unstable nature and low reactivity, it quickly disintegrates into two weak potential reactive species, namely oxygen and hydroperoxide anion (HO_2^-), as shown below (Hayyan et al. 2016).

$$O_2 + e^- \rightarrow \bullet O_{2^-} \tag{15.5}$$

$$2 \bullet O_2 + H_2O_2 \rightarrow O_2 + HO_2^- + OH^- \tag{15.6}$$

Singlet oxygen ($^1O_2$): Singlet oxygen is not radical but highly reactive. It is usually formed when the two unpaired valance electrons attain anti-parallel spin and occupy the same orbital. It is the first excited state of diatomic oxygen (O_2), and the removal of spin restriction makes it a powerful enough oxidant. Singlet oxygen is thus capable of reacting with electrons of all spin orientations, unlike molecular oxygen. The primary source of $^1O_2$ is the photoexcitation process (Wu et al. 2011).

Hydrogen peroxide (H_2O_2): Hydrogen peroxide (1.75 V) is a potent oxidizing agent in removing ECs in water. The relatively high solubility and stability help it participate in reactions with molecules distant from the point of their origin. Its ability to reproduce OH radicals upon reacting with photons make it suitable to degrade a wide range of contaminants (Kehrer et al. 2010). The hydrogen peroxide is typically formed in aqueous media subjected to plasma treatment is by the recombination of OH radicals as per the reaction. They can also form when plasma is produced from gases containing O and H_2, but the route is perilous as both the reactants are highly combustible. When plasma is in direct contact with water, it can entail hydrogen peroxide by the relatively less probable reaction mechanism, as shown below (Nosaka and Nosaka 2017).

$$\bullet OH + \bullet OH \rightarrow H_2O_2 \tag{15.7}$$

$$H_2 + O_2 \rightarrow H_2O_2 \tag{15.8}$$

$$\bullet OH + H_2O^* \rightarrow H_2O_2 + H \tag{15.9}$$

$$H_2O_2 + h\nu \rightarrow \bullet OH + \bullet OH \tag{15.10}$$

Ozone (O_3)*:* Ozone (2.07 V) is a powerful oxidising agent extensively employed in water and wastewater treatment. Ozone is produced through a three-body recombination mechanism where the atomic oxygen is reacted with the diatomic oxygen in the presence of a third colliding body produces ozone, where the electron-O_2 collisions produce the atomic oxygen (Zangouei and Haynes 2019).

$$O + O_2 + M \rightarrow O_3 \tag{15.11}$$

15.4.3.2 Types of Reactive Nitrogen Species

Nitrogen molecules have numerous excited states and metastable states, which often leads to many interactions with the liquid media. As a result, it is not easy to understand the mechanism of nitrogen plasmas or plasmas produced with ambient air. Nitrogen subjected to excitation has high vibrational energies ranging from a few

100–1000 K and less rotational energies lying around room temperature. This distinctive characteristic results from the low probability for collisions between neutral-vibrationally excited N_2 molecules. As the dissociation energies lie well below the excitation energy of N_2, the collision between these exited atoms leads to ionization, resulting in the formation of positive ions and electrons inside the plasma. Due to the richness in exited states and metastable stable states, nitrogen species are formed with oxygen available in ambient air or liquid media. One explanation for this phenomenon is that the dissociation energy of water molecules and oxygen lies par below some nitrogen species' excitation energy (Bradu et al. 2020).

Nitric oxide (•NO)*:* During the gas phase discharge of N_2 in the presence of O_2 or ambient air, the RNS reacts with ROS and leads to the formation of nitric oxide, which is one of the prominent secondary species of nitrogen (Tian and Kushner 2014).The key mechanisms through which •NO produced are given in Eqs. (15.12)–(15.16).

$$N_2 + \bullet O \rightarrow \bullet NO + \bullet N \tag{15.12}$$

$$\bullet N + \bullet O + M \rightarrow \bullet NO + M \tag{15.13}$$

$$\bullet N + O_2 \rightarrow \bullet NO + \bullet O \tag{15.14}$$

$$\bullet N + \bullet OH \rightarrow \bullet NO + H \tag{15.15}$$

$$\bullet N + O_3 \rightarrow \bullet NO + O_2 \tag{15.16}$$

Nitrogen dioxide (•NO_2): In oxygen-rich plasmas, nitric oxide produced can interact with species that can donate oxygen or O-donors such as •OH, •O and O_3 to produce nitrogen radicals. In dry gases, the ozone plays a significant role in nitrogen dioxide formation, whereas in humid gases, it is substantially less (Chen and Davidson 2002; Chen and Wang 2005). The possible chemical pathways of •NO_2 formation are:

$$\bullet NO + \bullet O + M \rightarrow \bullet NO_2^- + M \tag{15.17}$$

$$\bullet NO + O_3 \rightarrow \bullet NO_2 + O_2 \tag{15.18}$$

$$\bullet NO + \bullet HO_2 \rightarrow \bullet NO_2 + \bullet OH \tag{15.19}$$

$$\bullet NO + O_2 \rightarrow \bullet NO_2 + O \tag{15.20}$$

Nitrous and nitric acids (HNO_2, HNO_3): Discharges happening during the humid air or N_2/O_2 mixtures form nitric and nitrous acids through the following reactions (Tas et al. 1997; Chase and Hunt 2002)

$$\bullet NO + \bullet OH + M \rightarrow HNO_2 + M \tag{15.21}$$

$$\bullet NO + \bullet HO_2 + M \rightarrow HNO_3 + M \tag{15.22}$$

$$\bullet NO_2 + \bullet OH + M \rightarrow HNO_3 + M \tag{15.23}$$

$$N_2O_5 + H_2O \rightarrow 2HNO \tag{15.24}$$

Nitrogen species in aqueous media: Alongside the humid air, the interaction of plasmas formed in nitrogen-rich environments can also produce RNS in the liquid–gas phase and inside the liquid phase. The most dominating ones are the nitrogen oxy, and per-oxy molecules, nitric acids, nitrites and nitrates are formed from the interaction of water molecules and oxygen radicals such as •OH, •O with the dissolved nitrogen oxides produced in gas phase interactions (Bruggeman et al. 2008; Parvulescu et al. 2012).The interaction of nitrogen dioxide (•NO_2) species with oxygen radicals such as •OH forms the nitric acid in aqueous media similar to the reaction in the gas phase. Peroxy-nitrous acid can be formed by the interaction of •NO_2 with H_2O. Hydrogen peroxide and several other ROS can also produce peroxy-nitrous acid by reacting with nitrites, nitrogen dioxide and nitric oxide, which are listed below (Tian and Kushner 2014).

$$\bullet NO_{2\ (aq)} + H_2O \rightarrow NO_{2^-(aq)} + NO_{3^-(aq)} + 2H^+ \tag{15.25}$$

$$\bullet NO_{2(aq)} + \bullet NO_{(aq)} + H_2O \rightarrow 2NO_{2^-(aq)} + 2H^+_{(aq)} \tag{15.26}$$

$$\bullet NO_{3(aq)} + \bullet HO_{2(aq)} \rightarrow NO_{3^-(aq)} + O_{2(aq)} + H^+_{(aq)} \tag{15.27}$$

$$\bullet NO_{2(aq)} + \bullet OH_{(aq)} \rightarrow HNO_{3(aq)} \tag{15.28}$$

$$\bullet NO_{2(aq)} + \bullet OH_{(aq)} \rightarrow ONOOH_{(aq)} \tag{15.29}$$

$$\bullet NO_{(aq)} + \bullet HO_{2\ (aq)} \rightarrow ONOOH_{(aq)} \tag{15.30}$$

$$\bullet NO_{(aq)} + \bullet O_{2^-(aq)} \rightarrow ONOO^-_{(aq)} \tag{15.31}$$

15.4.4 *Quantification of Reactive Chemical Species*

The quantification of whole range of RCS is a challenging task due to uncertainty in formation pathways of these chemical species. So, the quantification of the radicals like •OH, $^1O_2$, H_2O_2, and O_3 through well-known characterisation methods is discussed briefly in the following section.

15.4.4.1 Quantification of OH Radicals

Direct measurement of OH radicals in an aqueous solution is difficult due to its low concentrations and short lifetime. One must rely on relatively more straightforward methods, like the chemical probe method, to quantify the OH radicals. Among all the available techniques, sodium terephthalate as OH radical probe has many advantages. Its symmetric structure forms only a single ring compound, 2- hydroxy terephthalic acid, upon interaction with OH radical, as given in Fig. 15.8. In addition to this, it has low limits of detection, typically 2 nM and strongly fluorescent (Gonzalez et al. 2018; Tampieri et al. 2021).

Another popular technique used to detect the presence of OH radicals is Electron Spin Resonance (ESR). It is a conventional spin trapping method for which DMPO (5,5-dimethyl-1-pyrroline N-oxide) is used as a spin trapping compound. During the plasma treatment of water, the OH radicals react with DMPO to form stable DMPO-OH radicals and can be detected by ESR spectroscopy (Dvoranová et al. 2014).

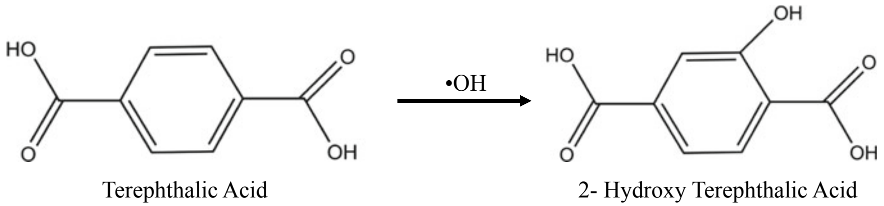

Fig. 15.8 Formation of 2-Hydroxy terephthalic acid from terephthalic acid

15.4.4.2 Quantification of H_2O_2

Hydrogen peroxide is typically quantified by a colorimetric method using an aqueous titanium reagent solution as an indicator. Metal complexes are formed in acidic solution, resulting in the formation of pale-yellow solution whose absorbance can be measured using a UV–Visible spectrophotometer at 410 nm (Eisenberg 1943).

$$Ti^{4+} + H_2O_2 + 2H_2O \rightarrow H_2TiO_4(\text{perititanic acid}) + 4H^+ \qquad (15.32)$$

With titanium-oxy-sulphate solution as a colorimetric probe, other probes such as N, N-diethyl-p-phenylenediamine (DPD) method are based on the oxidation of DPD on reacting with H_2O_2 catalysed using horseradish peroxidase. The reactant DPD generated during the reaction can form long-lasting colour and exhibit an absorption peak at 551 nm (Wu et al. 1999).

15.4.4.3 Quantification of ($^1O_2$)

Sterically hindered cyclic amines such as 4-hydroxy-2,2,6,6- tetramethylpiperidine (HTMP) generate 1-oxyl radical by reacting with singlet oxygen. The reaction produces stable compounds such as 4-hydroxy-2,2,6,6-tetramethylpiperidine 1-oxyl, also called nitroxide radical. The chemical reaction is shown below. This can be detected using ESR spectroscopy (Dimitrijevic et al. 2009; Brezová et al. 2005) (Fig. 15.9).

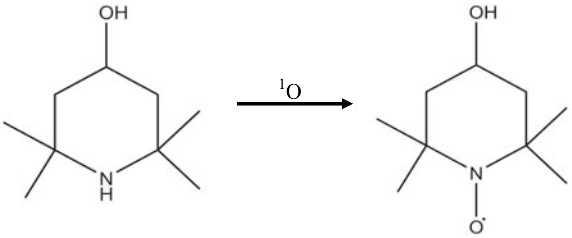

Fig. 15.9 Formation of TEMPOL from HTMP

15.4.4.4 Quantification of O_3

In recent years a rapid detection method for the ozone based on N-diethy1-1,4-phenylen-ediammonium sulphate coupled with spectrophotometry is proposed. When ozone reacts with potassium iodide in a buffered solution (pH 3.35) iodine is formed. This iodine would undergo a reaction with DPD reagent and produces a characteristic pink colour which gives an absorption peak at 510 nm (Song et al. 2000).

$$O_3 + 2I^- + 2H^+ \rightarrow O_2 + I_2 + H_2O \tag{15.33}$$

15.5 Plasma Assisted Degradation Process for ECs

The past decade has witnessed several works from the scientific community to enhance plasmas efficiency, especially NTP, for removing ECs in water. Phenols, azo dyes, pharmaceutical compounds, and pesticides have dominated this spectrum primarily due to their extensive usage in household and commercial purposes. Knowledge of plasma operating parameters like input voltage, electrode distance, flow rate, gas composition, reactor geometry, and type of discharge is essential. The water quality parameters such as pH, conductivity, concentration of organic contaminants, and background ions can also affect the process. The effect of these parameters on the plasma process is discussed in the following subsections.

15.5.1 Effect of Initial Pollutant Concentration

The influence of initial pollutant concentration on degradation kinetics is systematically studied. It is found that the rate of reaction increases with an increase in initial pollutant concentration but fails to keep up when the concentration further increased. The observed decrease in the rate constant at higher pollutant concentrations is attributed to the limited generation of RCS. Wang et al. studied the effect of triclocarban (TCC) concentration on the degradation efficiency in a DBD reactor. They observed a significant improvement in reaction rate when the pollutant concentration is varied from 2 to 7 mg/L. However, the increase in TCC above 7 mg/L did not keep up the trend (Wang et al. 2016). A similar study was performed on Rhodamine B (RhB) solution by Panomsuwan et al. by changing the initial concentration from 1 to 15 mg/L. The first-order kinetic model fits well for the lower concentrations than the higher concentrations (Panomsuwan et al. 2016). The data reveals that there is much competition between dye molecules and their byproducts at higher pollutant concentrations to react with available RCS, which leads to the deviation of the first-order kinetics model.

15.5.2 *Effect of pH*

pH is one of the critical factors that decide the efficiency of the plasma degradation process. Previous studies report a proportional relationship between the pH of the sample and the decomposition rate (Reddy et al. 2013). We can explain this phenomenon based on the change in OH radicals' production when medium pH changes from alkaline to acidic. In an alkaline medium, ozone decomposes to form more powerful OH radicals because of the favorable presence of OH^- ions, as shown in the equations below (Peleg 1976).

$$O_3 + H_2O \rightarrow HO_3^+ + OH^- \tag{15.34}$$

$$HO_3^+ + OH^- \rightarrow 2H_2O \tag{15.35}$$

$$3O_3 + H_2O \rightarrow 2\,{\bullet}OH + 4O_2 \tag{15.36}$$

In alkaline conditions, the ozone can be further reacted with water to form a superoxide ion and the recombination of OH radicals to form hydrogen peroxide. In acidic media ozone participates in degradation process more actively compared to basic medium because of relatively higher oxidation potential ranging from 1.4 to 2.8 V. Chen et al. have studied the influence of pH on methylene blue (MB) degradation (Chen et al. 2019). They reported that the shorted lived species have limited penetration inside the water; the long-lived species performed better under acidic conditions. OH radicals dominate the higher pH, but the degradation efficiency is suffered from the recombination of OH radicals to form peroxides. Although the initial pH is high while the experiment is initiated, due to the presence of N_2, O_2 in feeder gas produces secondary and tertiary species like HNO_2, HNO_3 along with peroxy-nitrous acid, which results in the reduction of pH values as low as 2. Peroxy-nitrous acid is a powerful oxidant and very unstable in acidic conditions, thus enhances the degradation of MB.

Ghodbane et al. reported that in acidic conditions, increased efficiency of AB25 dye is due to the protonation of $SO_3{}^-$ results in increased hydrophobic character of the dye molecules, which leads to the greater reactivity with reactive species generating higher degradation rates (Ghodbane et al. 2015; Ghodbane and Hamdaoui 2009). Zhao et al. performed the degradation of p-nitrophenol (PNP) using the microwave ambient air discharge observed that in the first minute of treatment the pH of the solution dropped significantly from 5.5 to 4 (Zhao et al. 2021). It slowed down during the next 11 min of treatment time. Further analysis with IC confirmed that the drop in the pH is due to the formation of tertiary compounds like acetic acid and formic acid when PNP is reacted with secondary species formed from the N_2 discharge interacting with the liquid phase.

Wu et al. have also studied the effect of pH on the degradation of MB using a dielectric barrier discharge and observed that pH of all the samples reached a specific value, which is in the range of pH 2–4 (Wu et al. 2019). It is inferred that the fall in pH is due to the solubility of nitrogen species in water and forming nitrous and nitric acid, as shown in Fig. 15.6.

15.5.3 *Effect of Water Conductivity*

The effect of the initial conductivity of the water sample significantly affects the discharge propagation, initiation in underwater discharges, and the number density of species. Hamdan et al. studied the effect of water conductivity using microwave underwater bubble discharge using argon as a trace gas (Hamdan et al. 2020). They have reported that the plasma electron density has increased with an increase in the conductivity of water from 10 to 100 µS/cm. No significant change in degradation was observed with a further increase in conductivity of sample from 100 to 1000 µS/cm. However, the further increase in the conductivity did not show any significant impact on the degradation due to the interaction of electrons with ions available in liquid media, leading to the formation of lower oxidative species.

The conductivity of the solution can also be increased by plasma exposure. Rancev et al. investigated the degradation of reactive orange 16 in a nonthermal multipin electrode atmospheric plasma reactor as a function of plasma exposure time and initial dye concentrations (Rancev et al. 2018). The conductivity of the sample was increased from 11.1 to 77.4 µS/cm within a minute of plasma exposure time. In some cases, the sample having high initial concentrations of the pollutant upon subjected to plasma treatment accumulates reactive nitrogen species like nitrite, nitrate ions and peroxy-nitric acid as discussed in the above Sect. 4.3 will increases the system conductivity and shifts the pH towards acidic.

15.5.4 *Effect of Feed Gas Composition and Flow Rate*

The choice of the feed gas is crucial in the plasma process as it influences the choice of input voltage, reactor configuration, electrode gap, input waveform etc. It can also affect the density of RCS formed during the plasma operation. It is observed that the use of oxygen as feeder gas results better degradation efficiencies followed by noble gas mixtures and ambient air. The flow rate of the feed gas is another important parameter that decides the total number of reactive chemical species being generated at a particular instant of time. The higher the flow rate, the more species will be generated as long as the applied electric field has enough strength to ionize more atoms. This increased species density results in a higher removal rate. Yamatake et al. studied the influence of gas flow at different velocities with inlet diameter changing from 200 to 300 µm (Yamatake et al. 2006). The degradation rate increases with an increase in a gas velocity proportional to the flow rate. The system's efficiency is higher at inlet diameters 200 µm compared to 300 µm because of a substantial increase in gas velocity due to smaller diameters. A possible conclusion from this study is that the generation and lifetime of ROS like •OH and •O are in the range of milliseconds, so they need to be transported fast to achieve faster degradation rates.

15.5.5 Effect of Reactor Configuration and Plasma Discharge

The last decade has witnessed several reactor configurations deployed to study degradation of ECs in terms of power consumption, formation of byproducts, toxicity of plasma-treated water and complete mineralization. It can be observed that DBD reactors have been most successful and leading the battle against the removal of ECs from the water with low power consumption and breaking of ECs into less nontoxic byproducts and time required to mineralize the target contaminant completely.

In summary, numerous reactors under various plasma conditions were employed for water treatment; commonly used configurations are summarized in Fig. 15.5. Further, the reactors mentioned in Table 15.4 are being often utilized for the removal of ECs. The DBD discharges, also called silent discharges, are quite energy-efficient. DBDs can be produced by covering either or both the electrodes using a dielectric material. The role of dielectric is to prevent the transition from glow/Townsend mode to arc mode. A schematic configuration is illustrated in Fig. 15.5 (c) (Magureanu et al. 2011). In DBDs the electron density is of the order of 10^{18}–10^{21} m^{-3} (Bruggeman et al. 2016). Micro-arc discharges both in-liquid and above the liquid surface are energy-intensive. However, they produce a high amount of RCS of the order of ~10^{24} m^{-3} s (Chen et al. 2019). Nevertheless, electrode erosion during this type of direct plasma treatment is a significant concern. A schematic discharge of this category can be seen in Fig. 15.5 (a). Bubble discharges are hybrid discharges and similar to those mentioned in Fig. 15.5 (e). The hybrid discharges are not pure in-liquid or in-gas phase discharges but discharges induced in liquid media inside the gas bubble (Shimizu et al. 2010). In hybrid discharge, the energy consumption is relatively lower than the direct discharges inside the liquid. The electron density in hybrid discharge is in the range of 10^{21}–10^{23} m^{-3} (Bruggeman et al. 2010). Gliding arc discharges are formed between two electrodes exposed to open air, one of which is ground and another connected to a high voltage source. At atmospheric pressure, this discharge can produce high electron densities (10^{19}–10^{21} m^{-3}) (Bruggeman et al. 2016; Du et al. 2007).

Further, some innovative reactor designs were also reported to enhance the degradation efficiency, including plasma-catalyst hybrid processes. Shang et al. has studied the DBD reactor with the addition of persulfate (PS) (Shang et al. 2017). The PS greatly enhanced the degradation efficiency of acid orange 7 (AO7) by almost 60%. The interaction of plasma with PS enhanced the production of •OH and $•SO_4^-$ radicals. It also improved the TOC removal efficiency. Ajo et al. (2017) have proposed

Table 15.4 An overview of various plasma reactors employed in EC removal applications

S. No.	Reactor type	Chemical compound	Concentration (mg/L)	Time (min)	Degradation (%)	Power/voltage	References
1	DBD	Amoxicillin	100	10	100	2 W	Magureanu et al. (2011)
		Dichlorvos	2	8	78.9	80 kV	Sarangapani et al. (2016)
		Crystal violet	100	25	94.2	18 kV	Reddy and Subrahmanyam (2012)
		Carbamazepine	23.6	30	38	250 W	Krause et al. (2009)
2	Micro needle	Atrazine	5	10	100	60 W	Mededovic and Locke (2007)
		Carbofuran	30	10	100	101.5 W	Singh et al. (2016)
		Chlorobenzene	200	12	100	17 kV	Jose and Philip (2019)
		Methyl orange	10	15	84.3	22 kV	Sun et al. (2012)
3	Gliding arc	Paracetamol	200	60	98	10 kV	Slamani et al. (2018)
		Bisphenol	28	30	100	10 kV	Abdelmalek et al. (2006)
		Perfluoroalkyl carboxylates	100	60	98	150 W	Lewis et al. (2020)
		Acid orange	100	20	98	10 kV	Yan et al. (2008)
4	Bubble discharge	Ibuprofen	60	25	82	11.6 kV	Wang et al. (2021b)
		Phenol	100	120	100	21 kV	Wang et al. (2008)
		Aniline	10	60	82.7	38.2 W	Liu et al. (2018)
		Alizarin yellow	30.9	10	100	14.4 kV	Zhou et al. (2021)

a pulsed corona discharge (PCD) reactor consisting of a vertical wire plate configuration where the water is sprayed using five atomizers from the top and plasma directly contact the water. It is reported that the gas–liquid interface is a barrier for the effective decomposition of ECs in aqueous media by OH radical. The generation of OH radicals is possible through the enhanced interaction of plasma-liquid interface through which high energetic plasma electrons directly collide with water molecules to generate •OH. Raji et al. added ZnO nanoparticles in a non-thermal atmospheric pressure plasma jet and compared the efficiency under various feed gases like oxygen, argon, and ambient air. The degradation of their pharmaceutical compound valsartan has decomposed more effectively by the addition of ZnO nanoparticles (Raji et al. 2020).

15.5.6 Effect of the Electrode Gap

An optimal electrode gap is crucial to achieving the maximum degradation capacity of the given configuration. The higher or lower gap can have adverse effects on degradation efficiency. Sugiarto and Sato have investigated the impact of electrode gap in removing phenols from water in the needle to plate configuration (Sugiarto and Sato 2001). Three configurations with 45, 15 and, 6 mm electrode gaps were investigated, and the 6 mm electrode gap yielded the highest removal rate. The formation of multiple small steamers to single spark transition creates shock waves, intense UV radiation, and high-energy electrons produce copious amounts of radicals. At 45 mm electrode gap, it is observed that there are multiple streamers, yielding less amount of RCS and lower degradation efficiency. Shang et al. studied the effect of different air discharge gaps in the degradation of acid orange 7 (Shang et al. 2017). The study reported that the degradation efficiency increases as the electrode gap increases from 2 to 4 mm. The further increase in the gap from 4 to 6 mm showed no significant improvement in the degradation efficiency. It is worth mentioning that at a low electrode gap, the discharge can significantly disturb by the water surface turbulence. Chen et al. has pointed out cathode fall as an important factor in producing reactive species (Chen et al. 2019).

15.5.7 Effect of Input Power

Several studies have been conducted to explore the input power or voltage influence on the degradation efficiency of ECs in water. As the input voltage increases, it is reported that the overall degradation efficiency increases. Energy yield increases proportion to the applied voltage up to a certain point and decreases, because production of reactive species reaches to saturation stage which cannot be further enhanced unless external parameters like feeder gas composition and flow rate are varied. The increase in removal rate can be attributed to high electric field strength and associated increase in electron temperature. High collision rates with the gas molecules lead to the rise in the concentration of reactive species, high-intensity UV-light and induced ionization of oxygen and water molecules.

Wu et al. studied the removal of tetracycline antibiotics (TCs) from water using a DBD reactor (Wu et al. 2021). The reactor was operated at different voltages (12, 15 and 18 kV) to investigate the influence of voltage on degradation kinetics of TC. The degradation efficiency increases proportionally to the rise in voltage. Reddy et al. also studied methylene blue (MB) removal using a non-thermal DBD discharge at different operating voltages (14, 16 and 18 kV). It is observed that 90% decolorization is achieved with an initial concentration of MB 100 mg/L in the first 25 min of plasma exposure at an applied voltage of 14 kV. The efficiency slightly increased to 94% as the voltage increased to 18 kV. The TOC removal % also slightly improved from 18 to 20% (Reddy et al. 2013). Pankaj et al. also reported the degradation kinetics of various dyes like methyl red, crystal violet and fast green FCF by deploying a high voltage atmospheric air and modified air cold plasma (Pankaj et al. 2017). It was reported that all the dyes showed higher removal rates at a maximum applied voltage of 80 kV.

15.6 Conclusion and Future Prospects

The widespread occurrence of emerging contaminants in water bodies is a serious concern in the water sector worldwide. Unlike conventional water pollutants, these contaminants are complex, diverse in their physicochemical properties. As a result, traditional water and wastewater treatment processes are ineffective in removing emerging contaminants from water. However, many of these compounds are potentially toxic to all life forms, and their removal from water is imperative to protect public health and the ecosystem. Considering the various physicochemical properties and complex fate and transport, a thorough understanding of various aspects of emerging contaminants, including their source, occurrence, classification, fate and transport, detection method, and treatment techniques, are essential in developing an effective management strategy.

The advanced oxidation process is a promising treatment technology for the removal of ECs in water. Among advanced oxidation processes, NTP looks attractive due to its ability to generate RCS at a controlled rate and ambient conditions. The process is also attractive due to its ability to produce reactive species like ROS and RONS, non-selective behaviour towards target pollutants, and sludge-free operation. However, the application of NTP in removing organic contaminants is mainly limited to small-scale laboratory experiments. The biggest challenge in scale-up is high energy consumption and lack of information on the process that dictates liquid plasma interaction. To overcome these issues, new models that include all the effects ranging from transport phenomenon, fluid dynamics effects, complex plasma chemistry, and fundamental collision process need to be set up, explaining the plasma liquid interaction phenomenon alongside the experimental observations.

However, the plasma modelling studies with experimental validation are limited. The plasma diagnostics in the liquid phase are also challenging due to the interference of many species; most of the available methods are not selective. Moreover, physical techniques like optical emission spectroscopy (OES) and optical absorption spectroscopy (OAS) are limited to estimating the species densities in the gas phase. The liquid surrounding the plasma or species poses a severe challenge in quantification using spectroscopic techniques. Besides, radical transport in liquid and liquid surfaces is still unknown. The effect of UV radiation and shockwaves produced by plasma on intermediate compounds and the plasma decomposition of water is clueless. For plasma technology to become dominant, these complex problems have to be addressed.

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