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# Utilization of plasma in water desalination and purification

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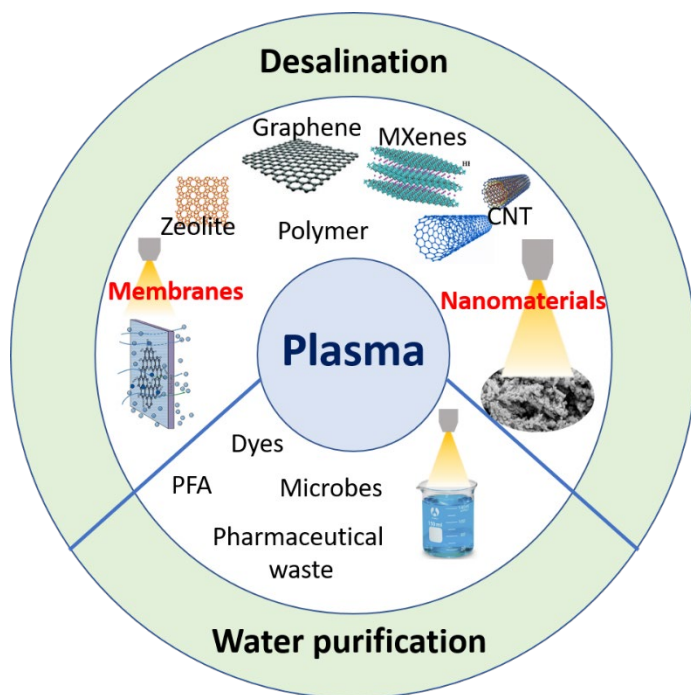
## Abstract

Supplying fresh drinking water to the world population is a persistent global challenge. Therefore, effective and efficient desalination processes are becoming increasingly important. Oceans account for most of water on Earth and the presence of salts and other contaminants in seawater prevents them from being used as a source of drinking water. Owing to this challenge, non-thermal plasma can be utilized in order to enhance the existing desalination processes via membrane or material modification while it can also be used as a direct tool for seawater desalination leading to significant process improvements. A direct non-thermal plasma-based desalination process is a new emerging area of research and recent efforts have shown its

promise with many unexplored mechanisms, providing benefits that conventional desalination processes cannot offer. Here we critically review the use of plasma technologies in water desalination including membrane modification by plasma for pressure, thermal, photothermal processes and direct plasma-based desalination process. We also address the use of plasmas in water purification. Finally, the existing challenges and future prospects are outlined.

**Keywords:** Plasma process, Membrane modification, Nanomaterial modification, Water desalination, Water purification

**Graphical abstract**



<b>Abbreviation</b>	<b>Explanation</b>
<b>MD</b>	<b>Membrane distillation</b>
<b>CNT</b>	<b>Carbon nanotubes</b>
<b>WCA</b>	<b>Water contact angle</b>
<b>SEM</b>	<b>Scanning electron microscopy</b>

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XRD	X-ray diffraction
FTIR	Fourier transform infrared spectroscopy
XPS	X-ray photoelectron spectroscopy
RF	Radio frequency
AOP	Advanced oxidation processes
APP	Atmospheric pressure plasma
CFU	Colony forming units
DBD	Dielectric barrier discharge
DCMD	Direct contact membrane distillation
DDW	Double desalinated water
MD	Membrane distillation
PA	Polyamide
PADW	Plasma activated desalinated water
PAN	Porous polyacrylonitrile
PAW	Plasma activated water
PDRO	Pressure driven reverse osmosis
PES	Polyethersulfone
PFS	Perfluoro substance
PTFE	Poly(tetra-fluoro-ethylene)
PVDF	Poly(vinylidifluoride)
RO	Reverse osmosis
IEM	Ion exchange membrane
CEM	Cation exchange membrane
AEM	Anion exchange membrane
PVC	Polyvinylchloride

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CDI	Capacitive deionization
SVGMD	Solar vapour gap membrane distillation
TFC	Thin film composite
TFN	Thin film nanocomposite
VMD	Vacuum membrane distillation

## Introduction

Water covers two thirds of the Earth's surface, yet, only a small portion (around 2.5%) of the available water is suitable for direct human use and consumption [1]. Fresh water scarcity is an important problem due to rapid population growth, expanding agricultural and industrial activities leading to water pollution and inefficient water management systems [2-4]. The ever increasing global freshwater demand escalates the need for developing efficient technologies to make use of various water sources with varying salinity to obtain fresh water from brackish water, contaminated fresh water, industrial waste water, and seawater where the latter is the most abundant water source [5, 6]. Hence, the development of efficient and effective water desalination and purification processes is essential for addressing the global water scarcity challenge.

Desalination refers to the process of removing salts and minerals from saline water sources including seawater or brackish water in order to obtain clean water suitable for human consumption and industrial usage [7]. Two prominent desalination methods currently used are thermally driven and membrane-based desalination processes [8-10]. Thermally driven desalination processes utilize the phase change of water, where thermal energy is used to evaporate the saline feedwater and then condensation takes place to obtain potable water. The most commonly used thermally driven desalination methods are multi-stage flash distillation,

multiple-effect distillation and vapor compression distillation [11, 12]. Membrane-based desalination processes use membranes with unique properties to separate the salt ions from the saline feedwater to obtain fresh water. The driving force for the water/water vapour transportation through the membrane can be a pressure gradient, a temperature gradient, or an electrical potential gradient [8, 13, 14]. Reverse osmosis (RO) is the most widely used membrane-based desalination technology which is based on applying excess pressure to reverse the spontaneous process of osmosis, where the water from a pressurized saline feed solution moves across a semi-permeable membrane to a permeate side which contains low solute/salt concentration [15]. Besides RO, electrodialysis, and membrane distillation (MD) membrane-based methods are also used in water desalination [8, 16]. Both thermal and membrane based desalination processes are important fields of research in which continuous process improvements are occurring [17, 18].

However, there are drawbacks associated with the current methods used in water desalination such as high energy consumption depending on the mode of desalination, disposal of brine that contains high concentrations of salts which causes marine or soil pollution, frequent cleaning and replacement of the membrane due to membrane fouling, as well as the high capital cost [6, 9]. Nevertheless, extensive research is being undertaken to achieve efficient water desalination with lower specific power consumption ( $\text{kWh/m}^3$ ). Recently, the use of non-thermal plasma processes in the development of advanced desalination and water purification processes has attracted significant interest owing to the unique properties of plasmas which can be utilized as an efficient and effective tool for water desalination and purification [19, 20].

Plasma is considered as a fourth state of matter, which is an electrically conducting medium containing positive and negatively charged ions, reactive radicals, and quanta of electromagnetic radiation [21]. Plasmas can be classified into two main groups, namely thermal

and non-thermal plasmas. Thermal plasma is also known as a local thermodynamic equilibrium plasma, which features high temperatures (2000-20000 K), high-pressure operation (>10 kPa), and near complete ionization of the gaseous species involved. In thermal plasmas, the temperature of the ions is close to the temperature of electrons and all the particles are in thermodynamic equilibrium [22, 23]. High power DC transferred arcs, high power plasma torches and radio frequency inductively coupled discharges are examples of thermal plasma generators.

The other plasma type is called cold or non-thermal plasmas. In cold plasmas, the temperatures of the electrons and the ions are different: usually electrons have higher temperature compared to the colder ions. The cold plasmas can be generated under milder conditions in comparison to thermal plasmas [24]. There are several techniques used to generate cold plasmas such as corona discharge, glow discharge, dielectric barrier discharge, low power plasma torch, and plasma jets [22, 24-26]. Cold plasma has been commonly used as a tool for the surface modification of materials [24, 25].

Though plasma technology has been developed extensively in other applications, the use of plasma technologies especially non-thermal plasma in water desalination and purification, has only been investigated recently yet gained significant interest. Non-thermal plasma is an energy efficient, green, scalable, and versatile technique which can be used to modify the physical and chemical nature of materials including membrane materials (organic, inorganic, nanomaterials) and surfaces to enable effective water desalination and purification. [22, 23, 27]. Moreover, direct plasma discharge on saline water or contaminated water has been performed to obtain water with low salinity or contaminant levels. Therefore, non-thermal plasma is an emerging technology in the field of desalination and water purification.

Various membrane materials and nanomaterials such as polymers [28-34], graphene [35], zeolites [36], MXenes [37], and carbon nanotubes [6] were modified by non-thermal plasma processing and used in water desalination systems to enhance desalination performances. In addition, plasma technology has been used in various water purification applications including the removal of dyes [38, 39], microbes [40], perfluoro alkyl substances [41], pharmaceutical waste [42] from contaminated water to obtain potable water. Lastly, a direct non-thermal plasma-based desalination process can address many of the drawbacks associated with conventional desalination processes such as high energy consumption, membrane fouling, and environmental pollution arising from brine discharge. Therefore, the work presented in this review highlights recent developments in the application of plasma processes in water desalination and purification, which is envisaged to be a topic of emerging importance.

## **1. Utilization of plasma for membrane and material modification for various water desalination processes**

### **1.1 Plasma modification of membranes for the membrane distillation process**

There is a growing interest for using membrane distillation (MD) technology in water desalination due to advantageous features such as ease of operation, the use of a low grade energy source, and the development of enhanced membrane materials [43]. Generally MD is divided into four main categories; direct contact membrane distillation (DCMD), vacuum membrane distillation (VMD), sweeping gas membrane distillation, and air gap membrane distillation [8]. Plasma is often used as a versatile tool for surface modification of MD membranes to enhance the MD process performance. In general, plasma modification of membranes can be achieved in several ways including 1) plasma coating of the membrane surface with other polymers, 2) introduction of functional groups on the membrane surface,



and 3) alteration of the roughness and porosity of the membrane surface. For such surface modification of the membranes, the plasma power needs to be low (<100 W) and well controlled, as energetic radicals and ions can damage the polymeric chain or backbones, which may lead to degradation of the membrane properties if not properly controlled [44, 45].

DCMD is driven by a vapour pressure gradient induced by temperature difference across a hydrophobic membrane [32]. Membrane performances can be altered by membrane structural parameters such as thickness, wettability, pore size, pore size distribution, porosity, and tortuosity [46]. Membrane performances such as flux, salt rejection rate, energy efficiency, and long term stability can be improved by optimizing the structural parameters of the membranes [47]. Generally, commercial hydrophobic membranes such as poly(propylene), poly(ethylene), poly(vinylidene fluoride) (PVDF) or poly(tetrafluoro-ethylene) (PTFE) are used in membrane distillation, due to their intrinsic hydrophobic nature that resists wetting of the membranes which prevents the transport of bulk water along with salts and other contaminants. However, water vapour permeation through thick, hydrophobic membranes with small pore sizes gives rise to low water vapour permeability [46, 47]. Moreover, MD membranes experience temperature polarization in the MD process which leads to lower differential temperature (low vapour pressure gradient) gradient at the interface of the membrane where the transport of the water vapour occurs [25]. To mitigate this low water vapour flux issue in conventional MD membranes, several studies have reported the modification of hydrophilic surfaces into hydrophobic surfaces via plasma polymerization which were then used in a MD process [25]. In this way thin, modified hydrophobic layer can induce high mass transfer across the membrane while hydrophilic membrane pores filled with water can prevent the heat losses across the membrane, effectively enhancing the water vapour permeability and the membrane performances.

A plasma polymerization process can introduce a smooth, ultra-thin coating either by surface adsorption or covalent binding of radicals to the surfaces of materials. Radicals and fragmented molecules generated from the chemical precursor and the plasma discharge gas are created and polymerized on the membrane surface. By selecting suitable chemicals, a hydrophobic polymer coating can be deposited on the membrane surface [44, 45]. The plasma treatment or coating of the membranes, can then be fine-tuned by adjusting the parameters such as plasma power, the type of plasma gas, the type of plasma equipment used and the treatment time [25].

Using the plasma polymerisation approach, the synthesis of a dual-layered membrane for the formation of a hydrophobic layer on top of a hydrophilic base membrane gained considerable attention in MD membrane research as a viable solution in improving MD membrane performance [46]. For example, plasma polymerization of octafluorocyclobutane was studied to modify the surface of hydrophilic microporous cellulose nitrate membranes. The microporous hydrophilic cellulose nitrate membrane sandwiched between the modified hydrophobic layers were used as a composite membrane for MD. Glow discharge plasma was used for the polymerization process under a high vacuum where a deposited polymeric layer exhibited the water contact angle of  $>100^\circ$  was achieved [46]. Mild polymerization conditions were proposed, since it was observed that both the pore diameter and number of pores in the modified membrane decreased with longer plasma treatment time and hence the water vapour flux decreased due to membrane pore blocking. The resultant plasma polymerized MD membrane exhibited a high water vapour flux of  $32 \text{ kg m}^{-2} \text{ h}^{-1}$  and a salt rejection rate of 92.1% at a feedwater temperature of  $70^\circ \text{C}$  using 0.3-0.5 M NaCl solution as the feed [45, 48].

Another widely studied plasma polymerization process for MD applications is the deposition of fluorocarbon compounds on the MD membrane. This is because plasma polymerization of fluorocarbon compounds exhibits advantageous features such as the low

surface free energy, high thermal stability, high chemical resistance, and the low friction coefficient [44]. It was observed that the plasma polymerization process often resulted in the formation of a three-dimensional, cross-linked network rather than forming chains with repeating units (conventional polymerization) which arises due to the precursor molecules being randomly fragmented and recombining on the membrane surface [44]. Liu et al. demonstrated hydrophobic surface modification of hydrophilic porous polyacrylonitrile (PAN) membranes for VMD via dipping the PAN membrane in a fluorine containing solution followed by a plasma treatment process [19]. Prior to the plasma polymerization, the pristine PAN membrane was pre-treated by Ar plasma etching, which increased the surface pore size and roughness of the membrane surface, which improved the VMD performance and the stability of the modified surface. The Ar plasma pre-treated membrane was then dipped in 1H, 1H, 2H, 2H-perfluorodecyl methacrylate monomer solution followed by the dried membrane being exposed to a glow discharge Ar plasma. The plasma polymerised PAN membrane exhibited a high-water vapour flux of  $32.9 \text{ kg m}^{-2} \text{ h}^{-1}$  with 99.93% salt rejection at  $70 \text{ }^\circ\text{C}$  feedwater under vacuum conditions (pressure of 95.2 kPa), when 3.5 wt% NaCl was used as the feed solution. The resultant membrane exhibited stable membrane performance for 80 hrs [19].

Similarly, a commercially available, hydrophilic, microporous polyethersulfone (PES) membrane was plasma treated under ambient conditions using an atmospheric pressure dielectric barrier discharge (DBD) plasma device. As an example of this approach, a mixture of perfluorooctyltriethoxysilane and (3-aminopropyl) triethoxysilane was used to generate a hydrophobic coating on the membrane surface. A dielectric barrier discharge (DBD) Ar plasma was used and the effect of the plasma discharge time on the membrane properties and performance was investigated. This PES membrane exhibited a hydrophobic coating with a water cascade analysis (WCA) of  $110^\circ\text{C}$  after the optimum plasma treatment time of 54 s. The

resultant plasma polymerized membrane exhibited a high water vapour flux of  $20 \text{ kg m}^{-2} \text{ h}^{-1}$  which was sustained for 100 hrs of MD operation [25].

Another approach in improving MD membrane properties is via direct modification of the hydrophilic membrane surface to a hydrophobic surface using a plasma treatment which changes the surface properties of the membranes. For example, conversion of a hydrophilic (WCA -  $60^\circ/0^\circ$ , top/bottom) flat PES membrane to a hydrophobic membrane surface was reported via  $\text{CF}_4$  plasma treatment of a PES membrane without using an additional monomer coating on the membrane surface [29].  $\text{CF}_4$  plasma treatment introduced fluorine functional groups on the PES membrane surface which reduced the surface free energy via moderate etching and binding of the functional groups on the membrane surface [28, 29]. The effect of plasma discharge time and power on the surface WCA was also analysed. In their study, the WCA showed a sudden increment from  $60^\circ/0^\circ$  to  $115^\circ/116^\circ$  with 50 W power and 10 mins of discharge time [29]. A very slight change of the WCA was observed upon further increasing the time and the discharge power; indicating that the surface modification reached a saturation point. The binding of the reactive fluorine species on the membrane surface led to the increased hydrophobicity of the surface while the reactive radical species in the plasma were able to penetrate through the pores of the PES membrane modifying the interior parts of the membrane, thereby preventing pore wetting during the membrane distillation process. The plasma modified PES membrane showed a high-water vapour flux of  $40.9 \text{ kg m}^{-2} \text{ h}^{-1}$  with an excellent salt rejection of 99.97% using a 4 wt% NaCl solution as the feed solution while the feedwater temperature was maintained at  $74.5^\circ \text{C}$ . However, a reduction in the mechanical strength of the PES membrane was observed after the  $\text{CF}_4$  plasma modification [29], which could be attributed to the interaction of  $\text{CF}_3$  radical species from the plasma with a polymer chains in the membrane leading to partially damaged C-C bonds.

Plasma modification of a MD membrane with varying plasma parameters and the impact on membrane characteristics were investigated by using commercial (PTFE) membranes to elucidate the impact of varying Ar, and air plasma conditions on the membrane surface energy and surface roughness [49]. The same report also investigated the plasma modification and impact on the membrane's surface porosity and wettability with varying plasma power, chamber pressure and treatment duration. Using the results, they revealed the membrane structure-property relationship for the membrane performance in a direct contact membrane distillation process using a model seawater solution as the feedwater. The authors observed that fluorine from the PTFE reacts with a radical species from the plasma after 1-2 mins of treatment which altered the chemical properties of the membrane leading to loss of fluorine functional groups from the PTFE membrane as well as modification of its physical properties such as roughness and porosity. Furthermore, for short plasma treatment durations of up to 10 mins, the surface roughness gradually increased, however, when the plasma treatment was longer than 10 mins, the surface became denser and the surface porosity was reduced. It was found that the water vapour flux of the membrane was proportionally dependent on the surface wettability. As revealed in the abovementioned studies, plasma condition, working gas and related plasma parameters needs to be well controlled to obtain desirable enhancement in membrane properties and membrane performances. Otherwise, it could lead to detrimental effect in membrane properties such as significant changes in membrane wettability, surface properties and loss of mechanical strength in the membranes.

In another study, H<sub>2</sub>/N<sub>2</sub> plasma was used to improve the performance of a PTFE layer in a PTFE composite membrane composed of a porous woven support and a dense PTFE layer [50]. 7 min of the plasma treatment time and 100 W of plasma power were found as the optimum process conditions. According to the SEM results, the pore sizes of the PTFE membrane were slightly increased with increasing the plasma treatment time and plasma

power, whereas the pores were significantly enlarged after 10 min of the plasma treatment. The average surface roughness also increased from 44 nm (1 min) to 66 nm (7 min) and decreased to 61 nm after 10 min of the plasma discharge. It is important to note that the plasma treatment only changed the porosity of the PTFE layer and the effect was not strong enough to change the porosity of the supported matrix structure.

A similar result on the plasma modification of MD membranes was reported [51]. The CO<sub>2</sub> plasma treatment was used to improve the water vapour flux of the polysulfone membrane. It was observed that the surface morphology of the modified membranes was strongly affected by the treatment time and plasma power. Based on the WCA and water vapour permeation results, it was reported that the surface tension of the membrane was affected by the plasma treatment, where it effectively reduced the temperature polarization of the MD membrane which effectively enhanced the membrane performance in terms of the water vapour flux compared to the pristine PTFE membrane.

Although the water vapour flux and salt rejection rate are the key criteria for a MD membrane, other considerations are also important as they often cause membrane fouling or wetting problems under the presence of low surface tension compounds such as oil and surfactants which can be present in seawater or contaminated water sources [52, 53]. To overcome such issues, an omniphobic PVDF membrane was produced by CF<sub>4</sub> plasma treatment [32]. The membrane exhibited wetting resistance to different types of low surface tension liquids such as methanol, mineral oil and ethylene glycol [32]. Firstly, the authors prepared a PVDF membrane by an electrospinning process which exhibited superhydrophobicity. According to their study, when the electrospun PVDF membrane was exposed to the radio frequency glow discharge CF<sub>4</sub> plasma, the formation of new CF<sub>2</sub>-CF<sub>2</sub> and CF<sub>3</sub> bonds was observed in the plasma treated electrospun PVDF membranes, which caused a further reduction of the surface free energy of the membrane thereby inducing omniphobicity. We emphasize

that the plasma treatment did not induce any significant changes in the surface structure or morphology but introduced a fluorinated layer by binding of fluorine functional groups on the membrane surface.

Although many studies test their membranes on relatively clean water or simulated seawater, report [32] used real RO brine from CSG (also known as coal seam gas) produced water with the addition of a problematic wetting agent such as a surfactant as the feed solution to evaluate the membrane performance of the plasma modified nanofiber membrane via an airgap membrane distillation mode. The authors observed a high water vapour flux of  $15.28 \text{ L m}^{-2} \text{ h}^{-1}$  and a near 100% salt rejection rate while exhibiting stable membrane performance without much sign of wetting using a saline feed solution containing 0.7 mM sodium dodecyl sulphate surfactant.

**Table 1.** Summarises the effectiveness of the reported membrane distillation techniques.

MD application	Membrane	Water flux	Salt rejection	Operating temperature	Ref
DCMD	plasma polymerized octafluorocyclobutane	$32 \text{ kg m}^{-2} \text{ h}^{-1}$	92.1%	70 °C	[45]
DCMD	Ar plasma polymerised PAN	$32.9 \text{ kg m}^{-2} \text{ h}^{-1}$	99.3%	70 °C	[19]
DCMD	Ar plasma polymerized PES	$20 \text{ kg m}^{-2} \text{ h}^{-1}$	99%	60 °C	[25]
DCMD	CF <sub>4</sub> plasma modified PES	$40.9 \text{ kg m}^{-2} \text{ h}^{-1}$	99.7%	74.5 °C	[29]
AGMD	CF <sub>4</sub> plasma treated Electrospun PVDF	$15.2 \text{ kg m}^{-2} \text{ h}^{-1}$	99.9%	60 °C	[32]
PDRO	O <sub>2</sub> plasma treated graphene membrane	$36 \text{ kg m}^{-2} \text{ h}^{-1}$	99.9%	40 °C	[54]
SVGMD	P-G-Ni <sub>foam</sub> + PVDF	$3.87 \text{ kg m}^{-2} \text{ h}^{-1}$	99.6%	25 °C	[35]

The above discussion suggests that plasma modified membranes for MD applications have demonstrated improved membrane performance in various aspects including enhanced water vapour flux, membrane stabilities and its ability to resist problematic wetting agents such as surfactants. Though, several studies report plasma modification or the plasma polymerization of the membrane exhibiting enhanced membrane performances in MD application for short testing duration, however, there isn't many studies demonstrating its long-term stability of the coating synthesized by plasma approach. Therefore, studies including plasma enabled hydrophobic coating's long-term progression of wetting and scaling tendency as well as its long-term performances needs to be further evaluated in future to assess the potential of plasma modified membrane's industrial impact. Therefore, it is a promising avenue for further studies.

## **1.2 Use of plasma in membrane and nanomaterials modification for pressure driven desalination processes**

Currently, RO is a leading membrane desalination process due to its low energy consumption to produce fresh water which is significantly lower than that of thermal or other desalination technologies [14, 43, 55, 56]. However, continuous research efforts are in progress in terms of RO membrane research to improve water permeability and achieving lower energy consumption, lower membrane fouling and scaling, and to improve the mechanical and long term stability of the membrane [13, 14, 53, 57, 58]. Typical RO membranes that are used are known as, thin film composite (TFC) membranes, where it consists of a dense, active layer made from polymeric material such as polyamide (PA) which is responsible for the solvated salt ion or the contaminant rejection and the support layer which provides effective water permeation and the mechanical support to the active layer, often made from polymeric material such as polyethersulfone (PES). In TFC membranes, other hetero materials or nanomaterials can be incorporated inside the PA active layer which can then form a thin film nanocomposite (TFN) membrane which usually exhibits enhanced membrane performances in pressure driven



desalination systems compared to polymeric TFC membranes. Such additive or filler materials can be organic or inorganic materials including zeolites, titania, silica, alumina and biomimetic materials [14]. For application in RO membranes (pressure driven desalination systems), plasma was often used to induce direct modification of the TFC membrane surfaces as well as modifying the nanomaterials which could be embedded in the PA active layer for TFN membrane synthesis.

For example, Safarpour *et al* fabricated a TFN membrane by interfacial polymerization of m-phenylenediamine and trimesoyl chloride with natural clinoptilolite zeolite embedded in the PA active layer [33, 36]. In their work, non-thermal glow discharge plasma was used to modify the surface of a natural clinoptilolite zeolite and then, was embedded into the PA layer of the TFN membrane. Plasma modification of natural zeolite is a promising route, as synthesis of synthetic zeolite involves sophisticated equipment setups and the use of toxic metal-organic precursors. According to the XRD and SEM results, they observed that the crystal structure of clinoptilolite was not destroyed or significantly affected by the plasma treatment, whereas the modification only affected the microstructure and the surface morphology of clinoptilolite. FTIR results revealed the formation of new Si-OH-Al bonds during the plasma treatment. Incorporation of plasma modified clinoptilolite contributed to higher hydrophilicity, higher water flux, higher salt rejection and improved antifouling ability compared to the pristine polymeric TFC membrane.

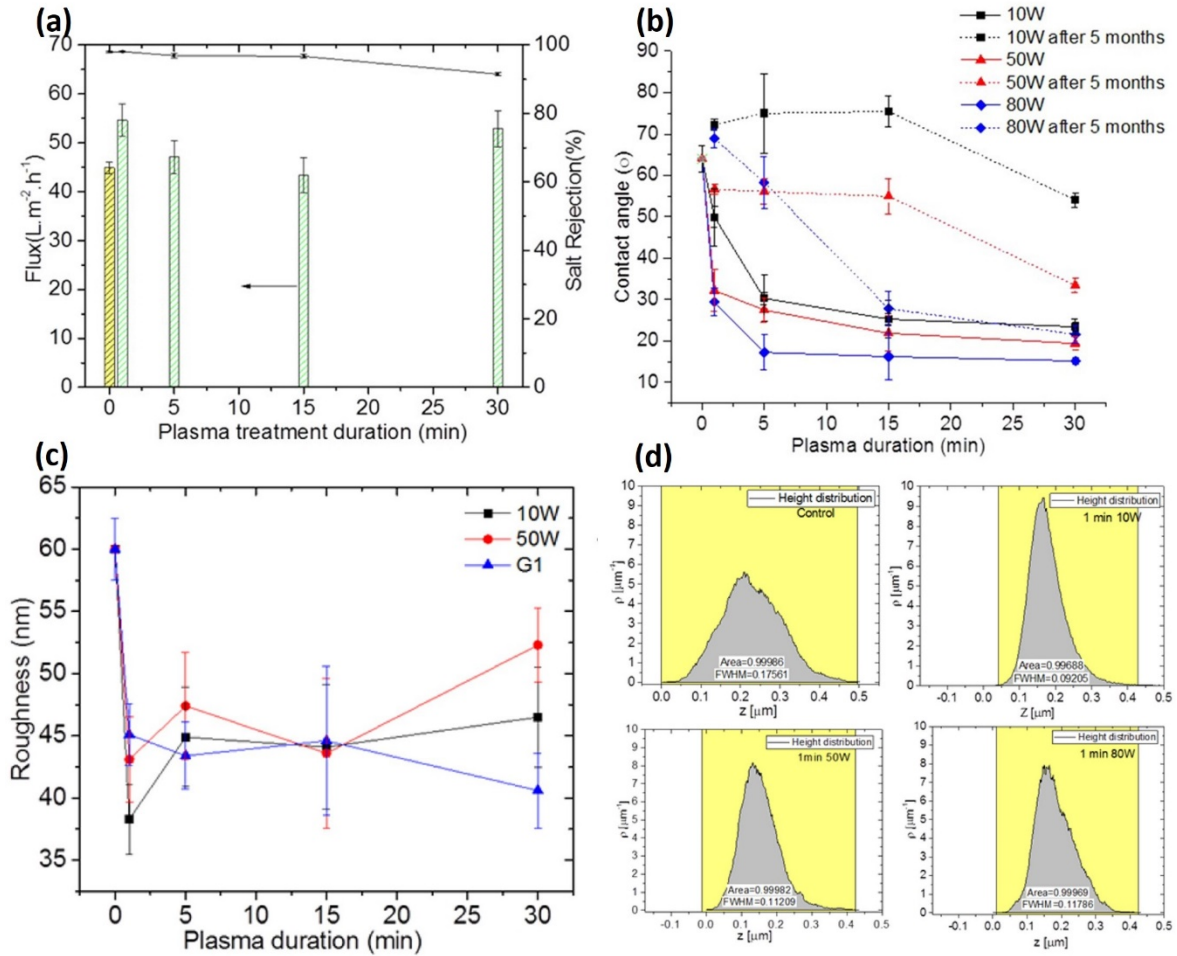
The scaling of salts in RO membranes is another problem for achieving high water recovery in water desalination. Scaling of the mineral salt is caused by the deposition of salt crystals onto the membrane surface via direct nucleation on the membrane surface, and subsequent crystal growth. Such mineral salt scaling deteriorates the membrane performance as well as shortens the lifetime of the RO membranes. To address this problem, a plasma modified TFC membrane was able to effectively suppress mineral salt scaling. Notable work

by Kim *et al.* demonstrated an approach to reduce the membrane mineral salt scaling propensity of TFC membrane by nano-structuring the membrane surface via atmospheric pressure plasma-induced graft polymerization [59]. In their work, firstly, the active PA layer of a TFC membrane was activated by the atmospheric pressure plasma treatment, which resulted in the formation of peroxide groups on the surface. Subsequently, a radical graft polymerization was carried out using a water soluble methacrylic acid monomer onto active layer sites to achieve a hydrophilic surface with a WCA of 10-17°. The plasma modified-graft polymerized TFC membrane was then evaluated for calcium sulphate dihydrate (gypsum) scaling propensity. The authors observed a measurable reduction of gypsum salt scaling relative to a commercial RO membrane which exhibited similar salt rejection, while yielding higher water permeability. The salt rejection, based on NaCl feed solution permeation test, for the prepared membrane was 94.5-95.8% while the commercial membrane (LFC1) gave a value of 94.4%, with a calcium salt rejection rate of  $\geq 95\%$  for the prepared membranes. The prepared membranes also demonstrated higher hydraulic permeability ( $2.0-3.4 \times 10^{-10}$  m/s Pa) relative to the commercial membrane ( $1.5 \times 10^{-10}$  m/s Pa). Furthermore, the membrane scaling was measured by analysing the time at which the flux decline commences, and according to the results, the prepared membrane showed 10.2 hrs before permeate flux decline was observed compared to 4.5 hrs for the commercial membrane revealing a significant reduction in scaling propensity [59]. We can attribute the observed effects to the dense coverage of plasma grafted hydrophilic water-soluble polymer chains on the active PA layer reduced the membrane scaling propensity, by reducing the deposition or adhesion of salt nuclei and bulk formed mineral crystals onto the membrane surface.

Similarly, the plasma polymerization was applied to a commercial TFC membrane in order to tune the surface charge which improved the water permeation property of the membrane [31]. Maleic anhydride and vinylimidazole were polymerized using an AC low-

pressure plasma, which provided higher controllability in terms of both deposition kinetics and chemical activation of the coated polymer layer since it operated at a much lower carrier gas pressure. The resultant plasma polymerized membrane exhibited the enhanced water permeation by 10% compared to the pristine reference TFC membranes.

In another study, low-pressure Ar plasma activation of the active layer was used to enhance the performance of commercial TFC membranes in water desalination [20]. In their study, TFC membranes were exposed to an Ar plasma and the surface characteristics were analysed under the varied power densities, and plasma exposure durations. According to the model saline water permeation results, a 10 W plasma exposure for treatment durations between 1 and 5 min led to a water flux enhancement of 22%, compared to the control membrane, while the salt rejection rate remained unchanged compared to the control membrane materials (Figure 2a). With increasing power density, the plasma treatment duration enhanced the hydrophilicity of the surfaces by binding hydroxyl groups on the membrane surface, as well as decreasing surface roughness (Figure 2c, d), where water contact angles decreased by 70% (Figure 2b). We note that this finding strongly correlates with the observation of an increased negative charge via zeta potential measurement and smooth uniform morphology of the membrane surface.



**Figure 1** (a) Flux and salt rejection for control and plasma-modified membranes, plasma treated sample at 10 W, (b) Water contact angle (mean of three replicates associated with their standard deviations) with varying plasma treatment duration, (c) average roughness (Ra) calculated from AFM surface roughness maps and (d) height distribution of the inset calculated from  $5 \times 5 \mu m$  AFM maps, indicated by values of  $Z$  and  $\rho$  represents the density or histogram-frequency of respective height values. Reproduced from [20].

Plasma can be also used as a tool for generating atomic defects on nanomaterials such as graphene, enabling them to work as an effective desalination membrane for pressure driven systems. There are research efforts being carried out to realize the potential of nanoporous graphene as a next generation RO membrane owing to several advantages such as high strength, low atomic thickness ( $d \approx 0.34$  nm), chemical resistance and antimicrobial properties [54, 57].

To generate a nanoporous morphology, pores in a monolayer graphene structure can be generated using various methods [54]. Recently, plasma has been used to generate a high density of nanopores with controlled pore sizes on the graphene film. Oxygen plasma has recently been used to generate nanopores with precisely controlled pore sizes in a monolayer graphene film [59]. The developed freestanding graphene membrane exhibited near complete ion rejection of dissolved ionic species such as  $K^+$ ,  $Na^+$ ,  $Li^+$ ,  $Cl^-$  with a near 100% salt rejection rate with rapid water transport. However, the ion rejection rate decreased when the nanopore sizes in the graphene film were enlarged by longer plasma exposure. The developed membrane exhibited an ultra-high-water flux of  $10^6 \text{ g m}^{-2} \text{ s}^{-1}$  at  $40 \text{ }^\circ\text{C}$  when the pressure difference was used as the driving force. Clearly, plasma modified membranes and plasma modification of nanomaterials for water desalination membranes have been proven to deliver enhanced membrane performances which require further in-depth research required to better elucidate the possibilities of this approach.

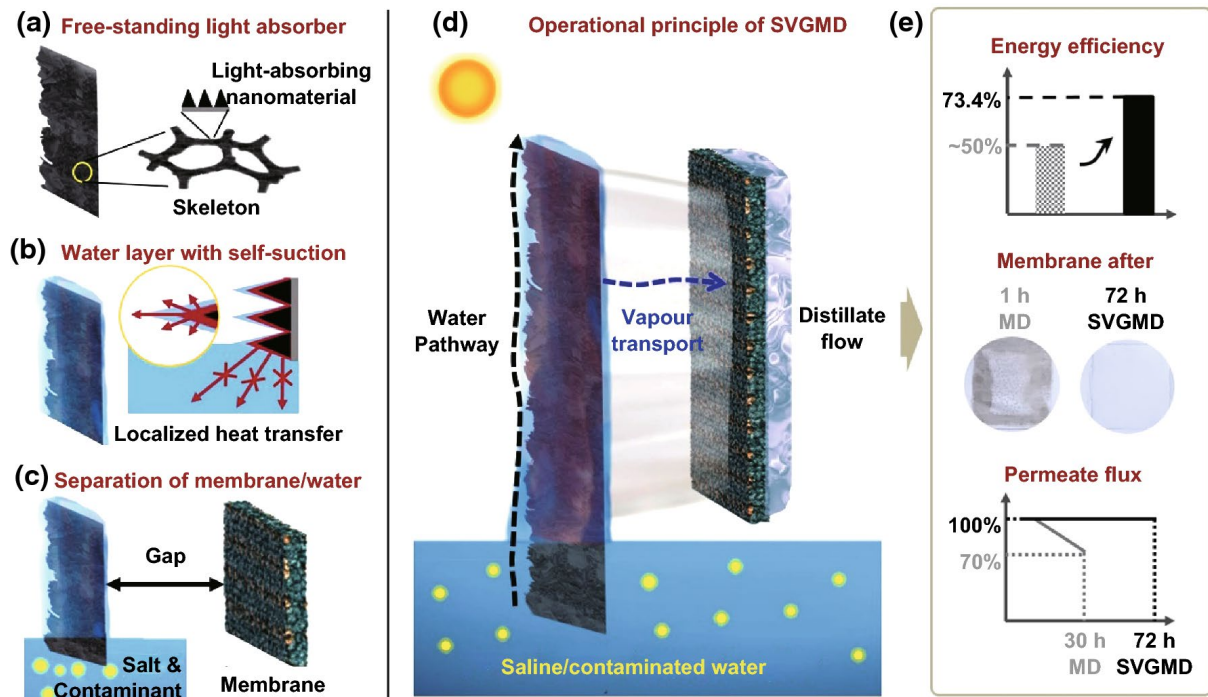
### **1.3 Plasma enabled material synthesis for photothermal membrane distillation**

Photothermal membrane distillation has become an emerging area of research for MD applications as it utilizes a renewable and abundant energy source such as sunlight to drive the MD process. However, solar energy driven desalination processes suffer from challenges such as low photothermal conversion efficiency, and membrane fouling. Therefore, many research efforts have been undertaken to address such challenges via heat and sunlight localization achieved with advanced material engineering or synthesis of nanostructured materials, which minimizes heat and photon losses within the photothermal materials leading to enhanced photothermal conversion efficiency [60]. For example, a new concept of solar vapour gap membrane distillation (SVGMD) was developed to synergistically combine self-guided water transport, localized heating, and separation of the membrane from the feed solution [35]. Moreover, a unique 3D graphene structure known as vertical graphene was synthesized using

the customized plasma enhanced chemical vapour deposition process. Utilizing a macroporous Ni foam as a substrate, a 3D, dense, uniform, micro-interconnected graphene structure enabled effective localization of light and heat which enhances the photothermal conversion efficiency. Next, poly(3,4-ethylenedioxythiophene)poly(styrenesulfonate) solution was sprayed onto the 3D graphene/Ni foam to prepare an advanced nanostructured light absorber denoted as P-G-Ni<sub>foam</sub>. In order to carry out the experiments, an apparatus was made with three components: a quartz plate which forms a window to allow sunlight passage with a stored feedwater in a tank below, a polypropylene spacer in the middle of the membrane and the P-G-Ni<sub>foam</sub>, and a plate with a small window which allows water vapor to escape. Effective water transport to the solar absorber layer/P-G-Ni<sub>foam</sub> surface from the bulk feed solution was realized and the effective heat and light localization was achieved by the microstructures of the 3D graphene produced from the plasma (Figure 3). Upon solar light illumination, clean water vapour was efficiently generated due to excellent light trapping and heat localization leading to high photothermal conversion efficiency. The condensed vapour was then collected by a distillate flow at the opposite side of the solar absorber. The membrane fouling issue due to salt crystallization on the membrane surface was addressed by placing a gap which separates the feed solution and the condensate part. As a result, a high photothermal conversion efficiency of 73.4% at 1 sun and a water vapour generation efficiency of 82.3%, along with the excellent antifouling properties, were achieved. Moreover, the membrane and the designed system exhibited a stable water flux for over 72 hours.

The novel plasma synthesized 3D graphene microstructure and the smart system design [60] address drawbacks related to conventional materials and design for photothermal membrane distillation such as low water vapour flux and low photothermal conversion efficiency, heat dissipation and membrane fouling. This work is another demonstration of how unique nanomaterials synthesized by a plasma process can lead to next-generation solar

harvesting and high performing solar driven efficient membrane distillation technology. As such, more novel nanomaterial synthesis for water desalination membranes via plasma processes require additional studies in the near future.



**Figure 2.** Solar vapour gap membrane distillation (SVGMD): concept, key features, and operational principle. (a–c) Summary of the key features: (a) free-standing light absorber consisting of interconnected skeleton and light-absorbing nanomaterials; (b) effective water transport across the whole solar absorber surface via self-suction and for localized heating. (c) Gap which separates the membrane from the feedwater and the solar absorber region. (d) Schematic of SVGMD. Superhydrophilic light absorber ensures fast water transport. Under sunlight illumination, localization of heat lead to the efficient generation of water vapour which transport through the gap, which passes through the membrane and condenses with the distillate flow. (e) The SVGMD concept and scalable water purification system with high solar-water energy efficiency, excellent anti-fouling performance, and stable permeate flux. Reproduced from [35]

#### 1.4. Use of plasma in electromembrane desalination processes and modification of ion exchange membranes

The use of electromembrane processes in desalination for the production of high-quality water from seawater or brackish water is another intensely researched topic. Electromembrane desalination processes involves the removal of solvated salt ions using an electrical potential difference as a driving force to remove charged ions through ion-selective membranes [61, 62]. These membranes have negative or positive charged functional groups and can be named as cation and anion exchange membranes (CEM and AEM) depending on the charge of the permeated ions. The performance of the ion exchange membranes (IEMs) depends on the ion exchange capacity, transport number, permselectivity, mechanical strength, thermal stability and ionic conductivity [62-64]. Currently, many research works are being carried out to improve the membrane performances of the IEMs including plasma modifications of the membranes [65-67].

Incorporation of various nanomaterials and the plasma modification of the material & the IEM surface was used to enhance the membrane performances which were reported in numerous past researches [68-70]. For example, S.M. Hosseini *et al.* prepared polyvinylchloride (PVC)/styrene-Butadiene-Rubber blend heterogeneous CEM by solution grafting technique [65]. Tetrahydrofuran was used as a solvent and to introduce functional groups, cation exchange resin powder was used. In their work, silver nanoparticles were used as a modifier of the membrane surface while using an argon plasma treatment for IEM modification. According to the scanning electron microscopy results, uniform distribution of silver nanoparticles on the plasma treated membrane surface was observed, and the uniformity has increased with the increment of plasma treatment time. The potential, surface charge density, transport number, and permselectivity of the membranes were increased gradually with increment of the silver nanoparticle layer up to 40 nm and then from 40-60 nm of silver nanoparticle layer, the above-mentioned properties were decreased. The electrical property and adsorption characteristics of silver nanoparticles could enhance the membrane performances up to 40 nm in thickness.



Therefore, the membrane with 40 nm of silver nanoparticle layer was chosen as optimum thickness and it showed comparable membrane performances compared to the other reports. Similarly, A. Zendehtnam *et al* modified the PVC based heterogeneous CEM using Ar plasma treated graphene oxide/silver composite (GO-co-Ag) nanoplates [67]. They noticed an enhancement of the membrane's properties such as water content, transport number, and permselectivity of the membrane upto 0.5 wt% of GO-co-Ag addition. However, concentration beyond 0.5 wt% of GO-co-Ag, it revealed a reduction in the membrane's properties. The addition of the such composite material enhanced the membrane's mechanical strength, and exhibited good antibacterial properties against *E.Coli*. Furthermore, A. Zendehtnam *et al* fabricated PVC based heterogeneous CEM mixed with synthesized multiwall carbon nanotubes-co-copper (MWCNT-co-Cu) nanolayer composite by solution casting techniques using cation exchange resin powder as functional group agent and tetrahydrofuran as solvent [71]. In order to fabricate the MWCNT-co-Cu, copper was sputtered on the substrate with MWCNTs in a vacuum reactor by argon plasma treatment using magnetron sputtering method. According to the scanning optical microscopy results and XRD results, uniform particle distribution and crystalline composite structure was observed. Addition of the MWCNT-co-Cu nanocomposite improved the membrane potential, transport number, selectivity, and electrical conductivity. 4 wt% of composite nanoparticle mixture exhibited optimum electrochemical properties compared to other modified membranes. Usage of MXenes in capacitive deionization (CDI) can be a promising technique owing to the potential of MXenes for selective ion adsorption and high ion removal capacity [72]. Recently,  $Ti_3C_2Tx$  was used in CDI applications [37]. Ar plasma was used to effectively modify the surface of  $Ti_3C_2Tx$  without disrupting the inner structures. In this work, an enhanced salt removal capacity (26.8 mg g<sup>-1</sup>) was achieved compared to the existing CDI electrodes, as Ar plasma treatment increased the interlayer spacing for efficient sodium ion transport and induced hydrophilic groups in

MXenes. According to the previous studies, usage of plasma treatment in the process of incorporating nanoparticles/nanocomposites in the IEMs, introduced several advantages such as improved adhesion of nanoparticles to the substrate, size uniformity, and controllable deposition rate and high purity [65, 67, 71].

Furthermore, direct plasma exposure on the IEMs can alter the surface properties of the membrane. Z. Zhao *et al.* used nitrogen and oxygen plasma treatment on AEM to improve the antifouling property of the membrane [73]. XPS results showed the incorporation of nitrogen and oxygen containing functional groups on the membrane surface after the plasma treatment. Surface morphology and surface roughness was changed due to the etching effects of plasma. As a result, they observed a pronounced antifouling property in oxygen plasma treated membrane than the nitrogen plasma treated membrane. Other notable work in direct plasma modification of IEM includes the work by F.M. Allieux *et al.* where they prepared hybrid ion exchange membrane using commercial porous stainless-steel filters as a backbone [74]. Prior to the sol-gel silane grafting within the porous metal structure, the metal surface was modified by plasma treatment with using an oxidative working gas. The plasma treatment greatly altered the physical-chemistry of the metal surface by introducing a homogeneous oxide layer on stainless steel surface thereby, facilitating a better wetting of the silane solution across the porous metal substrate, thus increasing the output of the sol-gel based silane coating reaction. As such plasma was effective in modifying both membrane material and the nanomaterial's surface, physical and chemical properties for IEM and electromembrane based desalination processes. Therefore, further studies are required using various plasma treatment processes and modification of various nanomaterials for improving the IEM should be explored in future.

### **1.5 Plasma for direct water desalination**

The previous sections explored the use of non-thermal plasma in membrane and material modification which are used as water desalination membranes as well as routes for synthesizing novel nanomaterials which are effective for use as a membrane material for water desalination. This section explores the use of plasmas for direct water desalination from saline feedwater sources. Direct plasma desalination is a newly emerging area of research and recent efforts have shown that it is promising as a water desalination route, with advantages that conventional desalination methods cannot offer. As discussed previously, a non-thermal plasma is defined as a plasma that contains low energy ions and as a result, these plasmas are typically at temperatures below 500 K and are often generated at atmospheric pressure [23, 25, 26, 75]. The use of non-thermal plasma for water purification has been investigated around the world with great interest over the last two decades with recent application for water desalination [76-81]. This section explores the advances in the use of non-thermal atmospheric pressure plasma for direct water desalination.

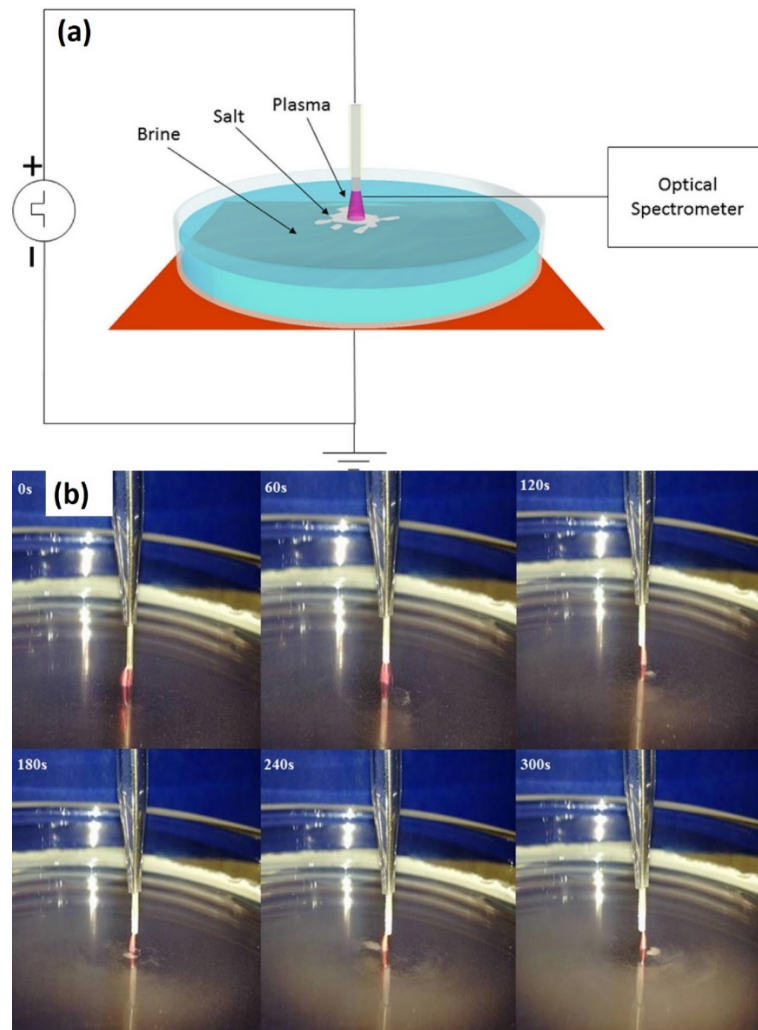
When an atmospheric pressure plasma is incident upon water, the energetic electrons and ions interact with water molecules and the solvated salt ions in the saline water which causes the direct crystallisation and precipitation of salts within the saline feedwater which can then be removed via a separation process [78, 80]. Moreover, the plasma treatment of water has been reported as generating what is known as the plasma activated water (PAW) which exhibited many useful characteristics, making it an effective tool for plant growth and germination in agriculture, bacterial deactivation and disinfection and use in food quality control [82-85]. PAW contains many reactive chemical compounds such as hydrogen peroxide and high concentrations of nitrate, as well as significant concentrations of other reactive oxygen and nitrogen species [77, 78, 84, 85]. Due to the unique salt crystallisation ability inside the liquid phase of water and the unique properties of the PAW, potentially any source of saline water can be desalinated by the plasma route which can be utilized in a diverse range of the

aforementioned applications, making direct plasma desalination route a promising area of research for further exploration.

Recently, there has been an effort to better understand the observation of such crystallization of salts at the plasma-water interface, however there are significant research gaps in the current knowledge which requires further exploration. Recently, the chemical reactions induced by DC argon plasma treatment at the surface of sodium halide solutions were studied [67]. This study focused on the short-lived active species generated by the plasma which reacts with solutes at the plasma-liquid interface. The concentration of halogens in solution were measured based on colorimetry using a DPD (N,N-diethyl-p-phenylenediamine) method. The authors discovered that when the non-thermal plasma source such as corona glow discharge produced the argon plasma at the vicinity of the sodium chloride, bromide and iodide solutions at concentrations of  $2.1 \text{ mol L}^{-1}$ , only the  $\text{Br}^-$  and  $\text{I}^-$  ions were detected within 30 seconds of plasma treatment period as halide ions are located closer to the gas-liquid interface in an order of  $\text{I}^-$ ,  $\text{Br}^-$  and  $\text{Cl}^-$ . The chlorine was not detected even after 10 mins of the plasma operation. This is because the  $\text{Cl}^-$  ions are excluded from the top-most layer in the plasma-liquid interface due to preferential ordering of the ion species inside the sodium halide solution and hence  $\text{Cl}^-$  ions couldn't react with plasma to form a chlorine whereas  $\text{I}^-$ , and  $\text{Br}^-$  were able to react into iodine and bromine. The concentration of  $\text{Br}^-$  and  $\text{I}^-$  were found to be  $8.89 \mu\text{mol L}^{-1}$  and  $21.3 \text{ mol L}^{-1}$ , respectively. Since the chloride ions were not detected, therefore it was concluded that its concentration remained less than  $0.42 \mu\text{mol L}$ . This observation of clearly distinctive reaction phenomena induced by Ar plasma is likely to be related to the different ion concentrations in the topmost layer of the feedwater mixture where the interaction with plasma and solvated salt ions are the strongest, and is supported by other studies [86]74].

The study [66] and the findings in other reports are important when investigating the use of non-thermal plasma for direct water desalination as similar complex chemistries

involving competing salt ion and water molecules interaction at the top most surface of the feedwater and the plasma would determine the effectiveness and the efficiency of water desalination via a direct plasma process. Moreover, such interactions potentially remove most of the undesirable contaminants from the saline feed solution to generate clean water. We emphasize that the above studies show insight into how electrical discharge can be utilized to treat brine solution containing high concentration of salts and how it can be directly crystallised in rapid, single step, low-energy process. For example, a low temperature corona discharge irradiation upon the surface of saturated sodium chloride solution with a density of  $1.28 \text{ kg L}^{-1}$  lead to salt crystallization and precipitation of the salts on the solution surface within 1 min of the plasma discharge with no significant temperature increment in the solution which implies that the thermal energy had no significant effect on the crystallisation of the salt on the surface. (Figure 4a,b) [80]. We would like to draw reader's attention that the salt solution was synthetic so that no other ions were present other than sodium chloride.

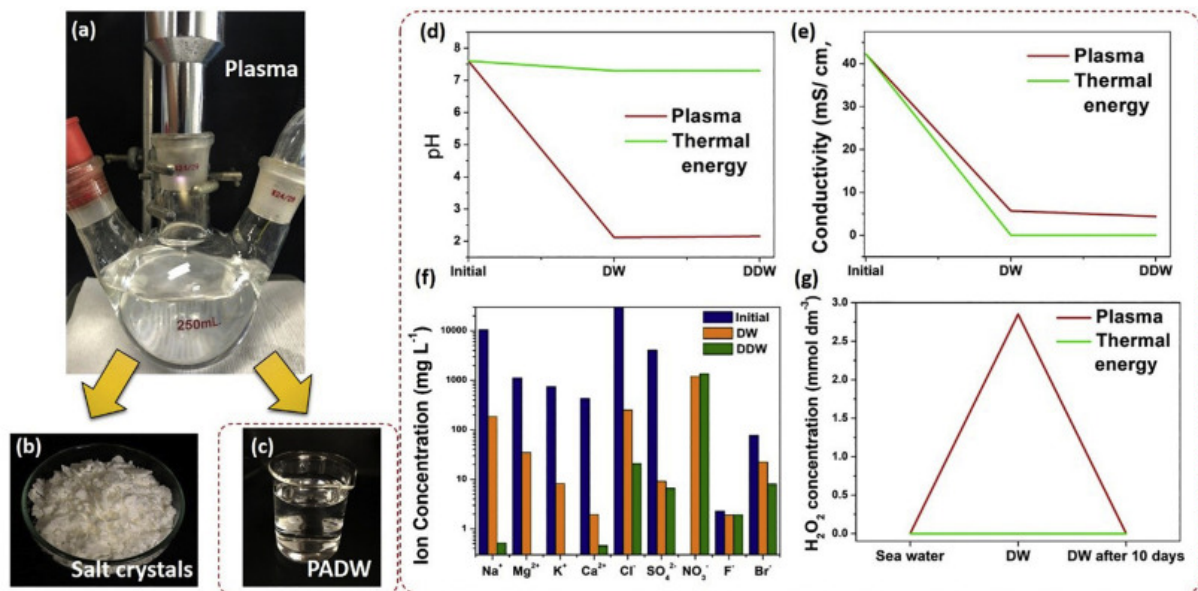


**Figure 3** Schematic representation of the apparatus to start the discharge on the surface of the brine, (b) Crystals forming under the electric discharge at 0, 60, 120, 180, 240, and 300 s, Reproduced from [80].

Plasma-water interactions, such as energetic electron bombardment and radicals produced from the plasma preferentially interacting with solvated salt ions have been suggested as the potential cause of salt crystallisation within the bulk saline solution. A non-equilibrium evaporation process occurs at the topmost surface of the feed solution, ejecting the species present at the top of the surface into the gaseous state without having to input a large amount of thermal energy to evaporate the water [80]. This process is caused by ions generated in the plasma contacting the solution surface, and is accelerated by the electric field [87]. These ions produced from the plasma transfer non-thermal, kinetic energy, to perturb the equilibrium bonding state between the solvated salt ions and the water molecules inside the feedwater. The

prevailing view in the community suggests that the ions present in the water can be ejected from the surface of the bulk water due to a combination of injected kinetic energy and a columbic interaction with the ions generated by the plasma.

The above phenomena were further studied by using a non-thermal plasma that operates at approximately 500 K [64]. In this work, instead of synthetic seawater, natural seawater was used to test the real-world feasibility of using plasma for direct water desalination. The most important discovery made in this work was that the desalinated water was not potable, as plasma activated desalinated water (PADW) exhibited a pH of 2.1, however, valuable industrial applications of both the water and the extracted salts were explored [78]. In this study, an atmospheric pressure plasma (APP) torch was used as the plasma source (Figure 5a) and the apparatus consisted of a water-cooled condenser and thermometer in the other two side-necks in which the APP was discharged directly on the seawater containing flasks while the PADW was collected using the water-cooled condenser. (Figure 5b) The precipitated salt crystals (Figure 5c) were collected from the flask at the end of the process [64].



**Figure 4** Exposure of sea water to atmospheric pressure plasma produces crystalline salt and plasma activated water. (a) APP torch; (b) Salt crystals; (c) PADW; Variation of (d) pH and (e) conductivity

of initial, desalinated water (DW) and double desalinated water (DDW) produced from seawater using plasma and thermal energy. (f) Different ion concentrations of initial seawater, DW and DDW produced using APP as the energy source (g) The  $\text{H}_2\text{O}_2$  concentration of seawater, DW and DW after 10 days produced using APP and thermal energy. Reproduced from [78]

By utilising an APP, desalination was more energy efficient than the conventional thermal evaporation process using a bench top heat plate at lab scale evaporation test (energy consumption of  $2140 \text{ kW h m}^{-3}$  for the hot plate compared to  $29,000 \text{ kW h m}^{-3}$  for the APP). The effectiveness of the plasma was further pronounced when comparing its evaporation rates to the conventional thermal evaporation process. Within 15 minutes of plasma discharge, 35 mL of versatile PADW was collected at a lower temperature ( $75 \text{ }^\circ\text{C}$ ) than the boiling point of water at atmospheric pressure. In the thermal evaporation case, only 10 mL of desalinated water was collected after 2 hours of heating. The difference in vaporisation rate is likely due to non-equilibrium evaporation caused by the kinetic energy transfer of the ions produced from the plasma to the seawater. The collected PADW showed a significant reduction of salt concentration compared to the original seawater indicating that effective desalination had taken place (Figure 5e, f). The pH of the PADW went down from 7.6 to 2.1 (Figure 5d).

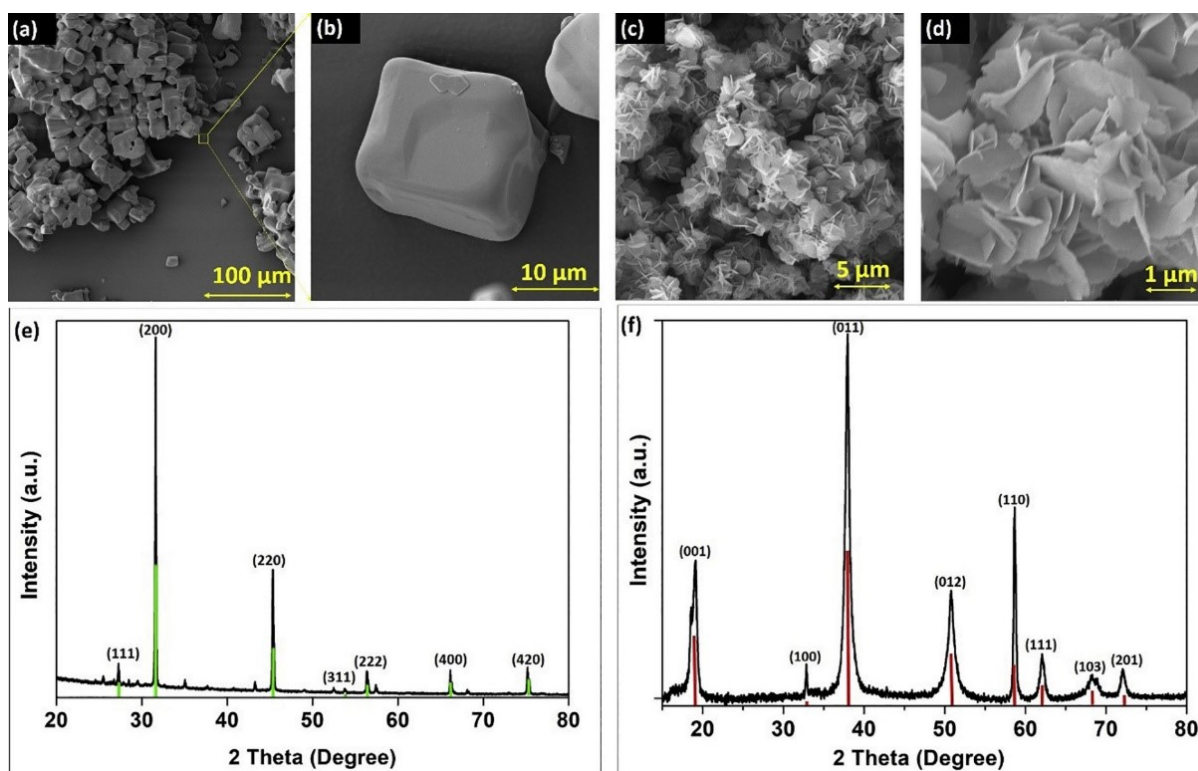
The PADW showed enhanced catalytic activities when it was used as an electrolyte for the hydrogen evolution reaction and oxygen evolution reaction processes. In the cyclic voltammogram (CV) for the hydrogen evolution reaction, the current density of the PADW was  $-50.85 \text{ mA/cm}^2$  at  $-0.4 \text{ V vs. RHE}$ , which is significantly higher than the control solution ( $-27.08 \text{ mA/cm}^2$ ) produced by acidifying distilled water with  $\text{HNO}_3$  to a comparable pH value of 2. In the oxygen evolution reaction process a higher current density ( $10.71 \text{ mA/cm}^2$ ) and lower onset potential ( $1.5 \text{ V vs. RHE}$ ) was observed for the PADW compared to the control solution (current density of  $7.42 \text{ mA/cm}^2$ , onset potential of  $1.60 \text{ V vs RHE}$ ).



Moreover, the collected PADW was used as an additive to enhance the seed germination of mung beans [64]. According to the results, mung beans, which were cultivated in the PADW-spiked water exhibited an enhanced seed germination rate and germination percentage compared to normal tap water. The reactive oxygen and nitrogen species in the PADW enhanced the germination of mung beans.

Furthermore, functional nanomaterials such as magnesium hydroxide ( $\text{Mg}(\text{OH})_2$ ) was synthesized from the salt recovered from the plasma enabled desalination process [64]. By using a single-step thermal process, high quality, homogenous 2D nanosheets of  $\text{Mg}(\text{OH})_2$  were produced (Figure 6).

Collectively, work [64] demonstrates the potential of using plasma as a route for effective and efficient seawater desalination which enables salt recovery at the same time. In a conventional desalination process, additional thermal evaporation is used to precipitate the salts from the brine. This new plasma approach may help to address the problems of the energy intensive brine concentration process and provide a solution to effectively use the brines to generate high value salt compounds. By comparing the work [78] to the other work [80] one can clearly notice that the crystallisation rate of the salts heavily depends on the energy density and the power of the plasma used (corona discharge, gliding arc, plasma torch etc.) and their plasma properties. The properties of the plasma activated water are determined by the ionic species produced by the plasma which depend on the feed gas used for the plasma generation. Investigations performed by several researchers confirm that the ions produced from the plasma are the critical factor in determining the plasma activated water production from a plasma-water interaction [85, 88, 89].



**Figure 5** (a) SEM image of precipitated salt; (b) Magnified SEM image of precipitated salt; (c) SEM image of synthesised  $\text{Mg}(\text{OH})_2$  2D nanosheets; (d) Magnified SEM image of  $\text{Mg}(\text{OH})_2$  2D nanosheets; (e) XRD spectra of precipitated salt; green lines represent XRD peak positions for Halite lattice structure; (f) XRD spectra of synthesized  $\text{Mg}(\text{OH})_2$  nanosheets; red lines represent the XRD peak positions for the Brucite lattice structure. Reproduced from [78]

Non-equilibrium evaporation using non-thermal plasma is an important phenomenon to enable effective water desalination as the solution does not become hot enough to effectively vaporise solely due to the thermal energy [78, 80, 87]. This phenomena was further investigated [90] focusing on the effect of the solution composition on the rate of non-equilibrium evaporation. This work substantiates non-thermal plasma-based desalination as parent ionic species that are generated from the plasma alter the solution composition with the injection of various reactive oxygen and nitrogen species which cause a reduction in pH and increase in ionic conductivity of the solution [89, 91-93]. Moreover, previous work reveals the inverse correlation between the solution pH and the plasma induced non-equilibrium evaporation [90].

Specifically, a solution with lower pH shows a higher rate of mass loss when exposed to the plasma. It was further demonstrated [90] that this relation holds for sodium chloride solutions which exhibit neutral pH (pH of 7) and a pH of 1.2 where the acidic salt solution showed a significant difference in the transfer coefficient from 133 to 516 after 10 min of the plasma irradiation, respectively. The transfer coefficient is defined as the number of water molecules transitioning into the gaseous phase due to non-equilibrium evaporation per ion arriving at the solution surface. This research [90] offers valuable insights into the observed non-thermal direct plasma desalination performed in reference [78] and the mass loss observed by the work presented in reference [80] due to the reactive oxygen and nitrogen species generated in the plasma lowering the pH of the solution and accelerates the evaporation of water from the feed water solution during plasma discharge [91, 92].

The aforementioned research demonstrates that the non-thermal plasma is an effective tool which can foster energy efficient desalination. However, future research challenges remain for producing pH neutral, potable water (low ion concentration) using a direct plasma desalination route. Moreover, the current studies are mostly based on the dead end, lab scale test which gives rise to high energy consumption for the plasma-based desalination routes compared to the well-established thermally driven desalination processes such as distillation or the membrane distillation processes (50~100 kWh/m<sup>3</sup>) [94]. Therefore, further studies are needed to design a smart, large scale, flow-based system to conduct the plasma-based desalination tests which will provide more accurate estimate of the energy efficiency of the plasma-based desalination routes as well as allowing one to assess the feasibility and the potential industrial impact of plasma-based desalination.

## **2. Water purification by non-thermal plasma exposure**

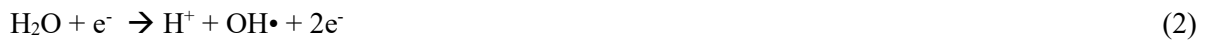
The ocean is not the only source of fresh water that can be reclaimed through water treatment processes. Recycled water facilities around the world reliably reclaim clean water from non-potable sources, generating millions of cubic litres of drinkable water per day [95]. Many countries are increasing their effectiveness and therefore the output capacity of their water reclamation facilities by improving their water treatment technology [96-98]. In this regard, direct plasma treatment of water is a promising technology which can be widely implemented in industry as recent research demonstrates the effectiveness of plasma treatment in water purification [99, 100].

The effects of non-thermal plasmas on water are well known in the literature; energetic electrons and ions are produced along with peroxy nitrite and peroxide radicals, reactive oxygen and nitrogen species, as well as UV radiation under ambient air condition. These species diffuse into the water causing secondary reactions, leaving behind nitrite, nitrate, hydrogen peroxide, nitric oxide and other strong oxidising species [101, 102]. These species are the distinct features of PAW as mentioned above and when a bacteria or certain chemical is exposed to these radicals created in PAW, they are quickly deactivated/degraded via advanced oxidation processes (AOP) [79, 99]. These AOPs are categorised as processes that generate large volumes of OH radicals in solution which causes mineralisation of carbon and organics and is an important mechanism for the anti-bacterial and water purification applications of plasma technologies [103].

The use of non-thermal plasmas at atmospheric pressure for water purification is a topic which requires further research effort. These studies could enhance the understanding of plasma-water-contaminant interactions and will lead to the discovery of benefits which are unique to the plasma-based processes in comparison to conventional water purification processes.

Amongst the important factors in plasma-assisted water treatment, the plasma dose or density is one of the most critical factors along with UV radiation production rate, OH radical production rate, and the interaction area over which the plasma-water are interacting [103]. Non-thermal plasma generates low-energy ions which prevents ions-water-contaminant interactions to take place beyond the top layer of the solution surface [104]. According to simulations performed in this work, chemical reactions caused by electron solvation dominates the plasma-water interactions with ions produced from the plasma creating a sheath at the solution surface when coming into contact from the gas phase.

The chemical reactions due to electron solvation and bombardment within a plasma-water interaction are as follows [84]:



Reactions (1) and (2) produce the hydroxide radical directly via electron solvation and reaction (3) creates hydrogen and oxygen radicals which can easily turn into a hydroxide radical showing that the energetic electron interaction with water does not require large surface area as electrons can penetrate far into solution to cause chemical changes in feedwater. Due to the higher penetration depth of electrons into a solution compared to the low energetic ions produced from plasma, a combination of effective electron solvation and surface ion bombardment is likely to be the mechanism for utilizing plasma for water purification applications.

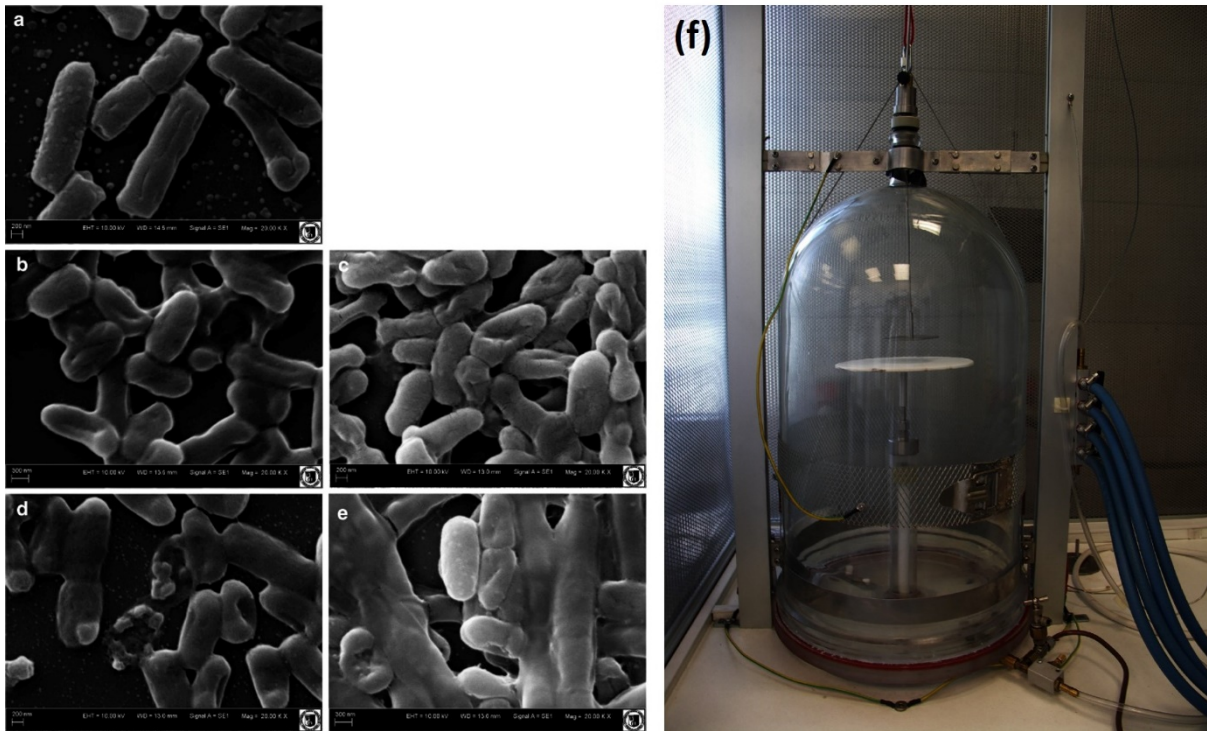
## 2.1. Use of non-thermal plasma for bacteria and virus deactivation in water

Non-thermal plasma treatment of water in order to deactivate water-borne bacteria is an effective strategy for anti-bacterial treatment and sterilization processes which could outperform conventional methods, as plasma treatment of water is potentially low cost, which produces minimal waste and no added chemicals are needed. Moreover, plasma processing is a versatile water treatment process which can be easily integrated with existing water treatment methods, complementing shortcomings of conventional purification processes.

For example, a plasma reactor was designed for treatment of micro-organism contaminated water where *A. ferrooxidans* and *L. gratiana* were used as microorganism contaminants [79]. In this work, the plasma was generated in a solution submerged configuration and the plasma generating probes were rotated at 1000 rpm. When the contaminated water samples were exposed to the non-thermal plasmas, in both cases, a 6-log reduction in colony forming units were detected in both bacteria species after 40 and 20 sec exposure time, respectively.

Further study [105] demonstrated that low doses of UV radiation at 25 mJ/cm<sup>2</sup> were able to deactivate *Giardia muris* cysts, however this did not result in permanent deactivation of bacteria. After 10 days, the bacteria began to repair the UV damaged DNA. Moreover, treatment with a UV/H<sub>2</sub>O<sub>2</sub> combination is a robust method for treating pathogenic microorganisms and organic micropollutants which could lead to permanent bacteria deactivation [92]. Furthermore, such combinatorial treatment satisfied the microbiological, organic EC and Dutch standards. Therefore, the ability of non-thermal plasma to generate both UV and H<sub>2</sub>O<sub>2</sub> simultaneously offers significant advantages when treating water containing microorganisms and organic micro-pollutants.

*Escherichia coli* is a bacterium that humans frequently come into contact with which can lead to water-borne diseases. Therefore, the ability to effectively and efficiently treat water that is contaminated with various bacteria sources including a *E. Coli* is important. Multiple bacteria including *E. Coli* can be inactivated by a cold plasma discharge (Figure 7f), [106]. Various surface analysis techniques were used to understand the effects of the plasma interaction with the bacteria. Helium and argon plasmas were used, and it was found that both plasmas exhibited similar deactivation rates of  $10^3$  colony forming units (CFU)/ml of *E. Coli* after 10 min of the plasma discharge [93]. Interestingly, the helium plasma exhibited significantly higher early deactivation rates. After 2, 5 and 10 min of the plasma discharge, *E. Coli* exposed to helium plasmas showed a reduction of 0.44, 1.99, 2.01 log orders of magnitude in CFU, respectively and 0.01, 0.49, 1.99 log orders of magnitude in CFU, respectively from exposure to argon plasma from an initial population of  $10^3$  CFU/ mL. The physical change in bacteria was observed by scanning electron microscopy which revealed that plasma treatment caused a shrinking in the cell wall as well as cell lysis which is caused by UV radiation and etching caused by ions produced from the plasma (Figure 7a-e) [107]. Such findings are important for choosing the right plasma conditions when using plasmas for water disinfection purposes.



**Figure 6** SEM images of *Escherichia coli*. **a** untreated control, **b** After helium plasma exposure for 2 min. **c** After argon plasma exposure for 2 min. **d** After helium plasma exposure for 10 min. **e** After argon plasma exposure for 10 min (f) Prototype generator of cold plasma. Reproduced from [106].

A later study [108] investigated the effect of air, nitrogen and oxygen plasma on the inactivation of *E. Coli* contaminated water. Ke observed a descending efficiency in bacteria inactivation from air plasma followed by nitrogen to oxygen plasma where the effectiveness of plasma in bacteria deactivation was attributed to the produced concentration of nitrite in the feed solution.

The discharge of air plasma at the surface of the solution containing 104 CFU/mL of *E. Coli*, demonstrated 99% inactivation of *E.Coli* after 20 sec, 82% inactivation after 40 sec for the nitrogen plasma and 90% inactivation after 120 sec using oxygen plasmas. Unlike reported in the study [43], the deactivation of *E. Coli* is greatly assisted by the plasma being submerged in water. The many energetic ion and electron interactions with water caused by plasma in the discharge area, leads to secondary reactions inside the water, significantly increasing the inactivation efficiency of the bacteria. From the results of the study [95] it becomes apparent



that air plasma, discharged at the solution interface shows the highest bacteria deactivation efficiency. This observation is confirmed by other studies investigating the inactivation of *E. Coli* in and out of the solution where different mechanisms play a role [107, 109].

The plasma treatment of water for *E. Coli* inactivation was extended to other applications such as disinfection of meats in the food industry [110]. By utilizing the plasma activated water with various radicals which exhibit anti-microbial and anti-bacterial properties [110], a chemical free treatment method was demonstrated for de-activating bacteria such as *E. Coli* and *S. Aureus* infected meat samples.

This inactivation efficiency trend is not just applicable to the *E. Coli* case, it is also valid for Gram-negative bacteria which is an important finding when treating a diverse range of bacteria using plasma processes [109, 111]. Gram-negative bacteria show particular susceptibility to cold plasma treatment due to their cell walls being very thin [112]. Due to the thin cell walls (typically less 10 nm), a gram negative bacteria is more vulnerable to ion bombardment and will also suffer cell wall degradation due to interaction with nitrites, UV radiation, H<sub>2</sub>O<sub>2</sub> or other radicals produced by the plasma discharge quicker than a gram positive bacteria which possess thicker cell walls, typically thicker than 50 nm [113-116].

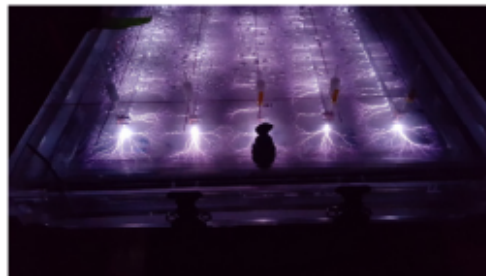
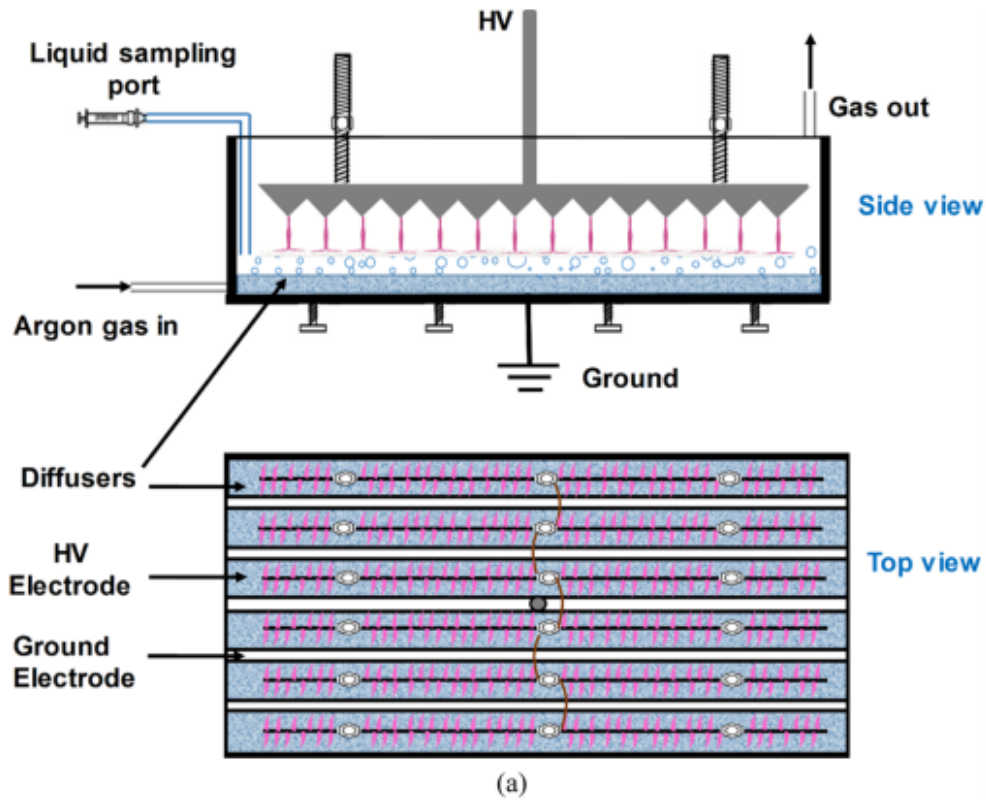
Recently, it was proposed that the deactivation of the devastating SARS-CoV-2 virus containing water via plasma treatment could be effective in deactivating the COVID-19 virus [117]. They demonstrated SARS-CoV-2 virus elimination from both water and wastewater by employing a submerged plasma reactor of an arc discharge that formed UV light, reactive oxygen species, and reactive nitrogen species.

## **2.2. Use of non-thermal plasma in emerging pollutant and chemical degradation and removal**

By developing our understanding of plasma-water interactions, we can be well-positioned to make the best use of this knowledge in various water purification applications. For example, a recent study [103] investigated the relationship between the dose of plasma and the rate of change in molar concentration of contaminants, focusing on AOPs. Promising results have been obtained in removing harmful toxins such as 1,4-dioxane from potential drinking water via AOPs, while also preventing the toxin from returning to other water sources [103, 118].

Another study was conducted using plasmas for the treatment of ground water [119]. Atmospheric pressure Ar plasma was used to treat emerging pollutants such as perfluoroalkyl substances (PFAS) and perfluorooctanoic acid (PFOA) and perfluorooctanesulfonate (PFOS). When non-thermal plasma was used to treat the solutions containing fluorocarbon contaminants, the concentration of PFAS, PFOA and PFOS was reduced to near 0 nmol/L as could be detected via mass spectrometry. Acids were produced as a by-product of the plasma interacting with various perfluoro substances (PFS); however, the concentration of these acids did not exceed 0.05 nmol/L, two orders of magnitude lower than the initial concentrations. In a later study, the effectiveness of plasma in the treatment of water containing various PFSs were tested by installing a pilot scale plasma reactor (Figure 8a,b) [100]. In 13 derived waste samples collected from US Air Force installations, 9 of those samples exhibited a reduction in concentration of PFSs to within US health advisory concentration levels (USHAL) within 1 minute of plasma treatment. The final 4 samples took up to 60 minutes of plasma treatment to reach the USHAL concentration. Furthermore, the removal efficiency for long chain PFSs were observed to be higher than for shorter chain PFSs which is in good agreement with the author's previous study [119]. Perfluoroalkyl sulfonates were removed from the water, in general, at a higher rate than perfluorocarboxylic acid which the authors credit to a higher surface activity by perfluoroalkyl sulfonate due to more  $\text{CF}_2$  content in its structure. These subsequent studies

confirm the effectiveness of treating water containing PFSs using plasmas with good reproducibility.



**Figure 7** Pilot scale plasma reactor: (a) the schematic and (b) reactor in operation. Reprinted with the permission from Singh RK, Multari N, Nau-Hix C, Anderson RH, Richardson SD, Holsen TM, et al. Rapid Removal of Poly- and Perfluorinated Compounds from Investigation-Derived Waste (IDW) in a Pilot-Scale Plasma Reactor. Reproduced from [100]

According to a recent study [119], the submerged configuration of plasma is inherently less effective at solute degradation than gas phase plasma discharges due to the heterogeneous

nature of the plasma interaction. Moreover, in this work, ground electrodes of various diameters ranging from 0.64 cm to 7.6 cm were used in order to test the effect of discharge area on solute degradation. It was found that the production of  $\text{H}_2\text{O}_2$  is best represented by rate equations that account for the area of the plasma solution interface and when area is the only factor in the reaction, the removal of a model dye contaminant such as Rhodamine B (which was used as the model solute for this work) and the generation of  $\text{H}_2\text{O}_2$  correlated linearly with the plasma-solution interaction area. This information is crucial when utilizing plasma in water treatment because one of the primary reactions induced by non-thermal plasma is generation of OH radicals Eq. (1) and (2) which then leads to secondary reactions causing the generation of  $\text{H}_2\text{O}_2$ . Being able to produce exact quantities of  $\text{H}_2\text{O}_2$  is an important factor in water treatment as UV/ $\text{H}_2\text{O}_2$  treatment of water alone is able to reduce the concentration of many contaminants by up to 3.8 log units [105].

It was recently demonstrated that a DBD plasma discharge was able to completely remove (degrade and mineralise) different chemical species of contaminants [120]. These chemical species include methylene blue, ceftriaxone, phenol, paracetamol and caffeine. After 5 min of the plasma discharge, phenol and paracetamol groups were completely degraded. Similarly, 25 minutes of plasma discharge were sufficient to degrade phenol, paracetamol and caffeine. As the above examples demonstrate non-thermal plasma has been shown to be highly effective for the removal of organic contaminants, chemicals, pharmaceuticals, bacteria and viruses and **Table 2** summarizes many of the contaminants that have been removed or degraded through a non-thermal plasma treatment of contaminated water.

**Table 2.** Various plasma configurations used in water purification to remove the various contaminants and their removal efficiencies

Species name	Plasma method	Power input for plasma generation	Removal effectiveness	Treatme nt time	Ref
<i>L. gratiana</i>	Submerged gliding- arc	500V, 3.5A	6-log units	20 s	[79]
<i>A. ferrooxidans</i>	Submerged gliding- arc	700V, 3A	6-log units	40 s	[79]
Perfluorohexanoic acid	DBD	35kV	90% removed	40 min	[100]
Perfluorahexane sulfonate	DBD	35kV	100% removed	40 min	[100]
Perfluoroheptane sulfonate	DBD	35kV	100% removed	40 min	[100]
Perfluorooctanoic acid	DBD	35kV	100% removed	40 min	[100]
Perfluorooctane sulfonate	DBD	35kV	100% removed	40 min	[100]
Perfluorononanoic acid	DBD	35kV	99% removed	40 min	[100]
Perfluorodecanoic acid	DBD	35kV	99% removed	40 min	[100]
Methylene blue	DBD	25kV	100% removed	5 min	[120]
Ceftriaxone	DBD	25kV	100% removed	5 min	[120]
Phenol	DBD	25kV	100% removed	15 min	[120]
Paracetamol	DBD	25kV	100% removed	15 min	[120]
Caffeine	DBD	25kV	100% removed	25 min	[120]
<i>E. Coli</i>	Gliding arc	3kV	5.15-log units	30 min	[121]
Methylene blue	Plasma Au-doped Fe/TiO <sub>2</sub>	-	66% degradation	4 hr	[122]
Amoxicillin	DBD	16kV	100% removed	10 min	[123]
Oxacillin	DBD	16kV	100% removed	30 min	[123]

Ampicillin	DBD	16kV	100% removed	20 min	[123]
Diclofanac	Pulsed corona	35kV	100% removed	15 min	[124]
Clofibrac acid	DBD	35kV	100% removed	30 min	[124]
Iopromide	DBD	35kV	99% removed	15 min	[124]
Indomethacin	Pulsed corona	20kV, 400A	100% removed	5 min	[125]

As shown in Table 1, the dielectric barrier discharge (DBD) method is a popular plasma system for water treatment due to low operation cost and ease of implementation. The water being treated is typically held in a chamber and the dielectric barrier discharge plasma is able to interact with a large surface area of water which greatly increases the treatment effectiveness as demonstrated in reference [119]. As discussed in the above sections, non-thermal plasma is an effective tool in degradation/deactivation of various organic, chemical compounds, bacteria and viruses, which requires further studies in the near future.

### 3. Future prospects

Water desalination and purification technologies are intensely researched topics, where continuous improvements are being made in various aspects such as energy consumption, water quality, environmental and technological compatibility, as well as techno-economic studies and water treatment in microgravity [7, 9-11, 43, 126-128]. Industry is slowly adopting non thermal plasma processes such as the removal of odour and allergens from air using pulsed corona discharges [129]. Plasma technology is an emerging technology for the future of water desalination and purification processes and membrane fabrication which could be widely implemented in various stages of water desalination and treatment processes [25, 28, 30, 130].

In the research area of membranes for water desalination via pressure driven systems, several studies reveal that advanced nanomaterial based membranes exhibit several advantages

over conventional polymeric membranes such as high water permeability, good mechanical stability with enhanced membrane anti-fouling and high ion selectivity [131]. Such nanomaterial-based membrane's performance can be further improved when nanomaterials are treated with plasmas which can induce physical, chemical and mechanical modification in the materials. For example, uniform pores in a nanomaterial such as graphene can be generated along with chemical functionalities in a controlled manner using a plasma treatment processes. In this area, only a few studies have been carried out to explore the potential of plasma treatment of nanomaterials as a membrane for water desalination and further research in this area is needed.

In the research area of membranes for water desalination via thermally driven systems, the recent work [35] demonstrated a new concept of solar vapour gap membrane distillation using a 3D graphene array synthesized by plasma processes as an effective light absorber. Therefore, further studies are required to explore unique nanomaterial synthesis via plasma processes which can offer similar advantages as 3D graphene arrays in solar vapour gap membrane distillation. Plasma enabled synthesis of nanomaterials is a particularly promising avenue for research as it offers rapid, low-temperature, highly ordered nanomaterial synthesis with unique structural characteristics such as vertical alignments. Moreover, controlling the hydrophobic membrane's properties in terms of controlling the plasma treatment conditions for MD application needs further exploration.

In the research area of using plasma as a direct desalination tool, further research is needed to deepen the understanding of the plasma interaction with various salts for water desalination. Such studies would aid in the further development of a direct plasma-based water desalination route. Though the direct use of non-thermal plasma in water desalination is in its infancy, it has demonstrated many advantageous features such as low energy consumption and the fast rate at which desalination occurs, without leaving high salinity brine wastes which

requires further research effort in the future [78, 132]. Furthermore, deepening the knowledge surrounding the crystallisation of salts within the solution phase of saline water by non-thermal plasma treatment and the control of plasma discharge parameters such as gas species, energy, discharge time etc. to selectively and efficiently crystallize the many valuable resources contained within seawater could be another topic of further research. Controlling the plasma discharge parameter has been shown to have a significant impact on the outcome for treating saline or contaminated waters [77, 107, 111] which could also be applicable to the direct use of non-thermal plasma for water desalination [133], and better understanding on the effects of different plasma parameters is required. Moreover, applications of water produced from direct plasma desalination is another area of research which requires further exploration. Furthermore, so far, the demonstration of plasma in direct desalination tool has remained in lab scale tests and the field is in its infancy state. Therefore, pilot scale studies are needed in the future to re-assess the effectiveness of plasma based desalination process and a proper techno-economic analysis based on the pilot scale studies would be required to accurately assess the economic viability of plasma based process as a means of direct water desalination.

Already in the fields of agriculture, disinfection and bacterial deactivation, non-thermal plasma has found great success and is becoming more widely integrated in various industries [134]. Plasma processes have also become an important part of advanced technology. Some of these include the treatment of textiles to make them more water resistant, treatment of lenses for anti-reflective coatings and scratch resistance and even plasma treatment of PET plastic bottles to assist in product preservation [134].

As discussed in the above sections the effectiveness of non-thermal plasma in treating various model bacteria and toxic chemicals in various water bodies were clearly demonstrated. Therefore, further research efforts are needed in terms of using plasma in water purification to treat various bacteria, viruses and toxic chemicals. Though many studies report the treatment



of model bacteria and viruses, in the future, treatment of a diverse range of bacteria and viruses should be conducted to further exploit the advantages of using plasma in disinfection and anti-bacterial applications. Similarly, further investigation is required on degrading various organics and harmful chemical compounds to exploit the unique advantages which plasma offers in chemical and organic degradation. Given the advancement in the use of plasma in various fields, the use of non-thermal plasma for water purification is envisaged to also rapidly develop into a potentially viable solution in the field of water desalination and purification.

### **3. Conclusion**

In this article, the use of plasma technology in various water desalination and purification applications has been critically reviewed. Non-thermal plasma technologies can offer advantages which conventional water desalination and purification processes cannot offer, including unique and rapid interaction with targeted solvated ions or molecules inside the contaminated water, compatibility with existing treatment processes, energy efficiency, potential scalability and low capital costs. The use of non-thermal plasma in membrane-based desalination technologies is expected to address many of the drawbacks related to conventional desalination membranes. Various types of plasma are used in order to modify the membrane surface properties, such as wettability, porosity, chemical resistance, and decreased membrane fouling to achieve efficient desalination. Although plasma modified membranes exhibit efficient and improved membrane performances, careful control of the plasma treatment is essential. Indeed, uncontrolled plasma treatment of membranes or membrane material modification could lead to detrimental effects.

Overall, non-thermal plasma is a promising tool with unique features which could be widely implemented in the field of water desalination and purification in the near future. Therefore, more research effort is needed on exploring the large, pilot scale plasma based water

desalination and purification experiments, optimizing plasma treatments for specific water desalination systems and various membranes and material modifications which are needed to exploit the full potential of plasma technology. This will improve water desalination and purification processes to make them more energy efficient, effective, with minimal discharge of brine or harmful contaminant into the environment for future generations.

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