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# Impact of multi-air plasma jets on nitrogen concentration variance in effluent of membrane bioreactor pilot-plant

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**Abstract**

An investigation of an atmospheric non-thermal air plasma effect on the modification of nitrogen concentration in the effluent of membrane bioreactor (MBR) pilot-plant has been proposed in this paper. The plasma treatment system has been a vertical liquid circulating system with horizontal multi-air plasma jets generated by a 125 W neon power supply. It has been found out that the variation trend of nitrogen concentration in air plasma-treated MBR effluent and deionized water (DI water) with different electrical conductivity (EC), ranging from $361.3 \pm 21.3$ to $2,153.3 \pm 83.1$ μS/cm, has been similar. The trend has been a positively skewed bell-like curve. The highest nitrogen concentration in the plasma-treated effluent has been found out at 15 min plasma treatment, which has been almost two times higher than that of the untreated effluent. The experimental results show that the EC of treated media had not significantly influenced the nitrogen concentration variance. However, considerably low EC (less than 1 μS/cm) of liquid media affects the difficulty of plasma generation. Regarding the experimental results, the atmospheric non-thermal air plasma has shown a positive impact on the nitrogen enhancement of MBR effluent, which could provide information in water reuse applications.

**Keywords:** Air discharge, Wastewater treatment, Membrane bioreactor (MBR), Circulating system, Water reuse

## 1. Introduction

The membrane bioreactor (MBR) system is a novel effective wastewater treatment technology that offers many advantages, including excellent effluent quality, stable operation performance, a small footprint, and reduction of excess sludge production [1, 2]. Although MBR technology is well-positioned to play a critical role in reuse applications, the MBR effluent has not met the water reuse standard for human consumption [3]. Among the existing utilization of reclaimed water, the reuse of MBR effluent for the agricultural farm would also be another practical application [1]. However, regarding the effective treatment system of MBR, the essential plant nutrient concentration in the effluents, such as nitrogen (N) and phosphorus (P), is usually low, and this does not meet the requirement for plant growth. Even though applying fertilizer directly into the water is the most efficient method, it could result in the long term side-effect of an environmental issue caused by chemical fertilizers [4, 5].

Recently, an atmospheric non-thermal plasma (ANTP) has been promoted as a novel technology for advanced oxidation processes (AOPs) applications in water and liquid treatment [6-9]. Regarding the AOPs, many reactions and byproduct chemical substances could be achieved depending on the plasma experimental conditions, the plasma model configuration, the treatment time, the generation power, and the career gas, including the target liquid chemical compounds. Some notable ANTP applications in water treatment are chemical decontamination, microorganism disinfection, water quality improvement, plasma-activated water for the bio-medical treatment or sterilization, and liquid fertilizer for agricultural applications [6-8, 10-14]. Besides various beneficial reactive oxygen species (ROS) and reactive nitrogen species (RNS), other phenomena that have occurred during the plasma treatment process, such as ultraviolet (UV) radiation, photon ($hv$), electric field (EF), and shock wave, could also contribute in water quality enhancement [9, 12, 15, 16].

In this paper, the influence of the ANTP by multi-air plasma jets on nitrogen concentration modification in MBR effluent has been investigated. The possibility of an enhancement of nitrate-nitrogen ($NO_3$-N) concentration in MBR effluent by air plasma treatment for liquid fertilizer or liquid disinfectant (in the form of unstable structural nitrate isomer) in agriculture has been studied. The plasma treatment system has been designed as a liquid circulating system. The pumped liquid stream released at the top of the treatment system would be directly treated by multi-air plasma jets falling into the reservoir container. This would increase the plasma contact surface ratio and the probability to be directly treated with the target liquid. The treated liquid temperature could be significantly decreased, compared to the static treatment system [9, 10], owing to the thermal diffusion to the ambient air during the circulation. Moreover, the problem of dielectric relaxing time and liquid conductivity tolerance, for long time operation, affecting plasma generation in the in-liquid discharge system could be neglected since the air gap distance between electrodes would be kept constant [17, 18]. The variation

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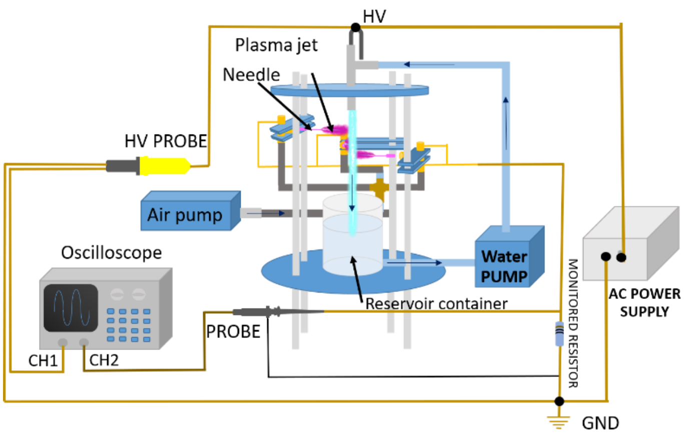
of plasma treatment time on the target MBR effluent treatment on nitrogen concentration has been investigated comparing it with the control-deionized water. The effect of electrical conductivity (EC) of the deionized water (DI water) on nitrogen concentration variation due to plasma treatment has been investigated. The air plasma jets and other physicochemical parameters of liquid such as pH, EC, oxidation-reduction potential (ORP), temperature, and dissolved oxygen (DO) have been also investigated.

## 2. Materials and methods

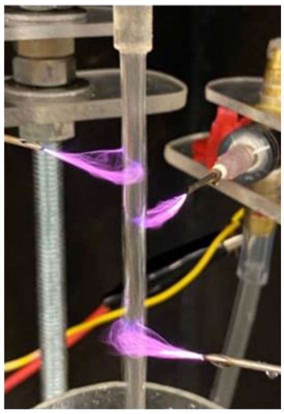
### *2.1 Plasma model and experimental setup*

The proposed plasma-activated water treatment system has been designed in a circulating treatment system as illustrated in Figure 1(a). The target liquid stored in the reservoir container has been pumped by a 600 ml/min water pump to the 0.5Ø cm three-way joint quartz tube located at the top of the treatment system. At this water flow rate, the water stream has been visually observed as a stable laminar flow. A copper wire has been inserted into the center of the three-way joint as an electrode. When the water pump has been operated, the copper wire has been immersed in a target liquid. The liquid stream has fallen vertically to the reservoir container and pumped back to the top of the treatment system. Three syringe needles as plasma jet nozzles have been hooked on the exoskeleton of the plasma treatment system surrounding the target water stream. The needle tip has been far from the water stream for 1.5 cm, approximately. Three needles have been arranged radially symmetric surrounding the water stream. However, they have not been arranged at the same level. The distance between each needle has been 2 cm, horizontally. Fifteen lpm of air from the air pump have been supplied to every needle via a plastic tube. The center of the syringe needle tip has been rotated 10 degrees clockwise from its basement (the axial direction from the needle tip axis has been in a tangent line of the water stream surface) in order to avoid water stream interruption from the flowing air. A terminal of 15 kV from a commercial neon transformer (125W commercial neon transformer, Topneon, TPN-1520A) has been connected to the copper wire electrode. In contrast, another terminal has been grounded and connected to the 100-ohm monitor resistor in series before connecting it with three parallel syringe needle electrodes. The target liquid has been treated at different treatment times at 0 (control group), 15, 30, 45, and 60 mins in the open environment.

The electrical characteristics of discharge waveforms have been recorded by an oscilloscope (SIGLENT, SDS2304). A high voltage probe (Pintek HVP-39 pro) has been connected across the anode and the ground electrode in order to monitor discharge voltage ($V_d$) characteristics. The discharge current ($I_d$) waveform has been retrieved from the voltage dropped across the monitor resistor. A charge-coupled device (CCD) spectrometer (Newport 71SI00087) has been used to observe the optical emission spectroscopy (OES). An optical detector has been perpendicularly located at the center between the needle tip and the water stream. The detector tip has been far from the plasma jet for 1 cm, approximately.




(a)



(b)

**Figure 1** (a) A schematic drawing of the plasma model and the experimental setup, and (b) Image of MBR water stream being treated by multi-air plasma jets.

### *2.2 The effluent of the MBR pilot-plant*

These investigations have been conducted using all the samples from the MBR pilot-plant, which has received mainly wastewater from a household building in Nakhon Nayok, Thailand. It has been installed to treat up to 9.6 $m^3$ of wastewater per day. The membrane has been a submerged microfiltration hollow fiber membrane with a nominal pore size of 0.1 µm from Sumitomo Electric Company, Japan. The total membrane area of each membrane has been 6 $m^2$, and six membrane units have been installed. The water quality parameters of the MBR effluent from the plant used in this study are shown in Table 1.

**Table 1** Water quality of MBR effluent

| Parameters | Unit | Value |
|---|---|---|
| pH | - | 7.0 ± 0.4 |
| EC | µS/cm | 649.3 ± 92.7 |
| COD | mg/l | 19.2 ± 12.0 |
| Total Nitrogen (TN) | mg/l | 21.5 ± 3.6 |
| Nitrate-Nitrogen ($NO_3$- N) | mg/l | 17.9 ± 2.4 |
| Total phosphorus (TP) | mg/l | 1.2 ± 0.5 |

*2.3 Laboratory analyses*

The physicochemical parameters have been characterized before and after the treatment. The pH levels, the temperature, and the DO have been measured using a Portable pH-DO meter (model HandyLab 100, SI Analytics). The EC and the ORP have been measured using a conductivity meter (model HI4321, Hanna). Samples have been collected periodically from the flasks in order to determine nitrate-nitrogen ($NO_3$-N), ammonium-nitrogen ($NH_3$-N), and total nitrogen (TN) concentrations. TN and $NH_3$-N analyses have been performed using Hach-Lange cuvettes and a DR900 spectrophotometer (Hach-Lange, Belgium), while the Environment Protection Authority (EPA) has performed $NO_3$-N analyses Approved Brucine Method.

*2.4 Statistical analyses*

The experimental data have been obtained from triplicated experiments (n = 3). The experiment has compared the characteristic values using one-way analysis of variance (One way-ANOVA). A *p*-value lower than 0.05 ($p < 0.05$) within groups has been considered.

**3. Results**

*3.1 Air plasma jet characteristics*

After the airflow rate and the gap distance have been adjusted optimally to the supplied source voltage from a neon transformer as mentioned in the experimental setup, air plasma jets have been uniformly generated between the needle tips and the water stream [13, 19]. The air plasma jet characteristic is illustrated in Figure 1(b). It has been a streamer-like discharge with a transient spark discharge as the intensified purple filaments could be noticed in the plasma stream. The air jets have been blown along with the axial needle axis. However, the air plasma plumes have been attracted to the water stream surface due to the electrically driven between the needle tips and the water stream [20, 21]. Therefore, it could be implied that the air plasma jets could effectively interact with the continuously flowing water stream surface.

Regarding the circulating plasma treatment, the treated water temperature could be greatly decreased compared to the static treatment. After 60 min of treatment, the plasma-treated water temperature has been around 42.5° Celsius, which is approximately 6.5° Celsius lower than the one of the 15 min static treatment. Moreover, target water could have interacted with air plasma jets thoroughly.

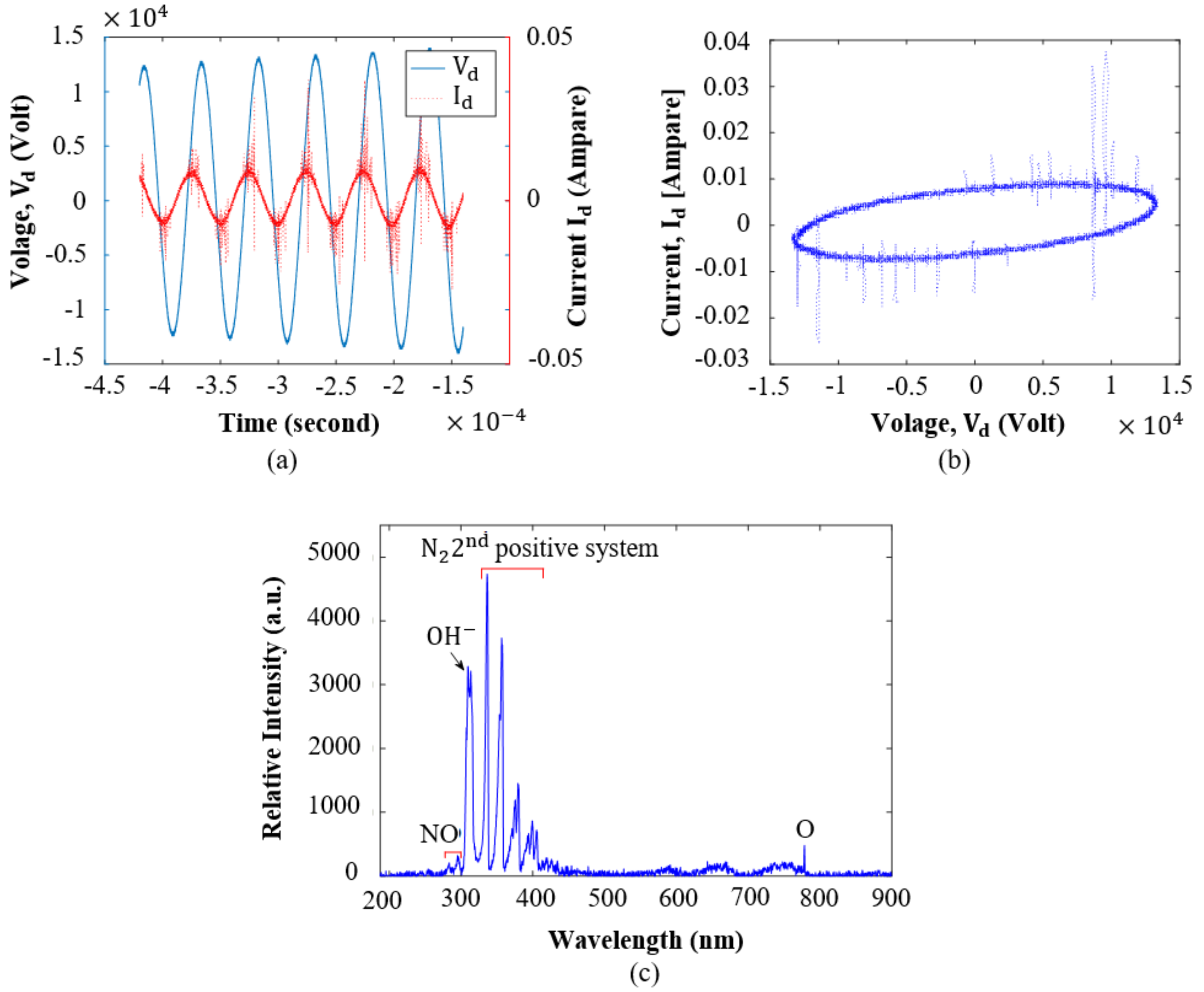



**Figure 2** (a) Electrical characteristics of voltage and current waveforms, (b) The voltage and current plot over one period, and (c) Optical emission spectra of air plasma jets.

The electrical waveforms of voltage across the plasma jets and a current flowing in the treatment system are presented in Figure 2(a). The voltage and the current have been out of phase owing to the capacitive plasma load. The voltage across the plasma model has been 9.26 $kV_{rms}$, with the peak voltage around 13.45 kV. The voltage frequency has been 20 kHz, while the micro discharge current frequency has been 10 MHz, approximately. The primary conducting current flowing through the plasma has been about 7 $mA_{rms}$ with the micro discharge pulses ranging from 12-37 $mA_{rms}$ generated during the rising edge of every half-cycle. Figure 2(b) presents the voltage-current (V-I) plot over one period of air plasma jets. It could be noticed that the average micro discharges in the negative cycle have been comparatively lower than the positive cycle owing to the space charge effect between the electrodes. Therefore, it could be implied that the discharge power dissipation is an asymmetry in each half voltage period [22, 23]. The optical emission spectrum (OES) could be observed regarding these micro discharges, as illustrated in Figure 2(b).

Various optical emission spectrums could be detected by OES investigation during air plasma generation, as shown in figure 2(c). The second positive system of $N_2$ could be observed in the range of 300-400 nm. The atomic oxygen emission line has been detected at 777.4 nm. Moreover, the hydroxyl ion ($OH^-$) radical emission lines at 309.6 nm, and the NO γ bands ranging from 270-295 nm have been noticed [24-27]. These radicals play an essential factor as a primary reactive species resulting in other by-product reactive oxygen and nitrogen species (RONSs) which are beneficial for the water treatment process, such as hydroxyl radical ($OH^-$), superoxide anion ($O_2^-$), hydrogen peroxide ($H_2O_2$), ozone ($O_3$), nitric oxide (NO), Peroxynitrous acid (ONOOH), and peroxynitrite ($ONOO^-$).

*3.2 Nitrogen concentration variance during plasma treatment*

In order to investigate the impact of air plasma on nitrate-nitrogen conversion, it is necessary to compare it with the control water media. In this time, a commercial deionized DI water (Maxzaa®, LCH Chemical, Thailand) has been used as the control group. However, at the same experimental condition of plasma generation, air plasma could not be generated between the needle and the DI water stream [17, 18, 28-32]. This is because the DI water has a very low EC (less than one µS/cm). It should be noted here that the proposed treatment system is a gas-liquid-phase discharge, in which liquid media serves as a cathode in every half-cycle [33]. Since the cathode is the major secondary electron emission source to sustain the discharge [32, 33], the electrode's conductivity has played an essential role in the gas discharge process. In order to generate gas discharge between the very low EC (quasi-nonconductive) electrodes, very high supplied voltage is required, compared to the conductive electrode [17, 18, 28-32]. Therefore, the EC of the DI water has been adjusted by adding potassium chloride (KCl), which is a common standard electrolyte used for EC adjustment of electrochemistry solutions, until the EC level has been closed to the one of the target MBR water (649.3 ± 93 µS/cm) [18, 29, 30]. However, in order to confirm whether KCL has influenced the nitrate-nitrogen conversion, the DI waters with various dissolved KCl concentrations have been treated by multi-air plasma jets and investigated.

The concentrations of total nitrogen (TN), ammonia-nitrogen ($NH_3$-N), and nitrate-nitrogen ($NO_3$-N) have been measured immediately after the treatment process of each experiment. The result has shown that $NH_3$-N has not been detected in any sample tested, because any residual ammonia-nitrogen has been likely oxidized to another form of nitrogen. The $NO_3$-N and the TN concentrations are plotted against plasma treatment time, as shown in Figure 3. For both samples, nitrate-nitrogen has been induced upward at a contact time of 15 min and then declined (positively-skewed bell curve). However, it can be observed that the initial concentration of $NO_3$-N and TN in MBR is higher than the one of the EC-adjusted DI water. This will be discussed again in section 4.3. The maximum concentrations of nitrate-nitrogen and total nitrogen of MBR effluent after 15 min plasma treatment have been 31.10 ± 2.44 mg/l and 33.80 ± 0.60 mg/l, respectively, which are higher than the initial concentrations around 1.74 and 1.68 times, respectively. Moreover, these ones are higher than the ones of 649.3 ± 93 µS/cm DI water around 1.13, and 1.21 times, respectively.

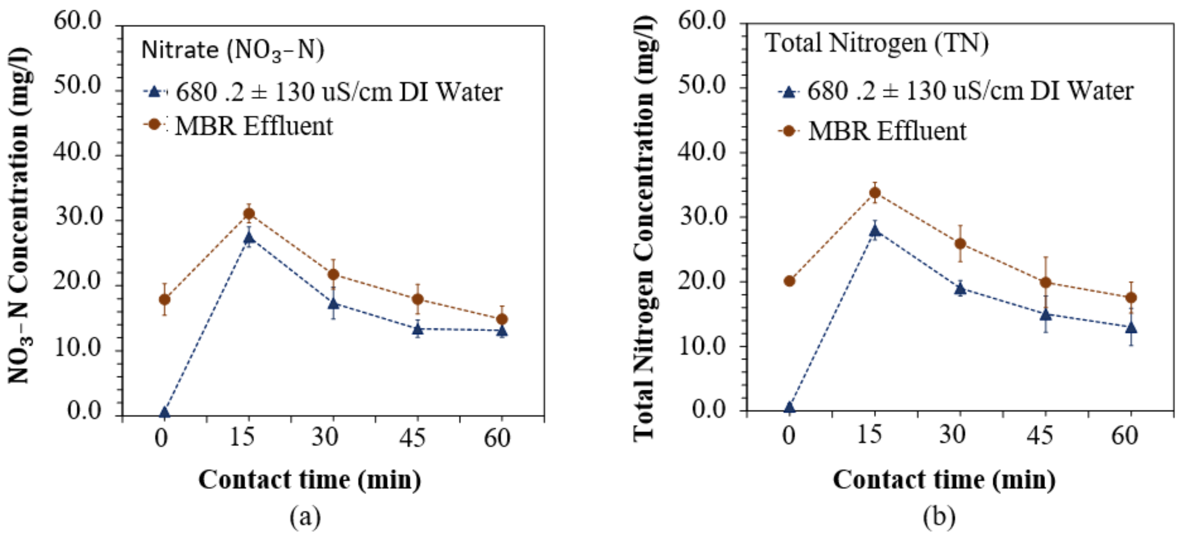



**Figure 3** (a) Nitrate-nitrogen concentration, and (b) Total nitrogen concentration in MBR effluent and EC-adjusted DI water at various treatment times.

The effect of the parameters on the performance of plasma in liquid samples has been studied. Figure 4 presents the variance of the operating parameters, which are pH, EC, ORP, and DO, after plasma treatment at various treatment times. It could be seen that all the parameters of both conditions have the same trend. The DO concentration and the pH have been declined, while EC and ORP have been increased when the treatment time increased. It could be noticed that the pH and the ORP of both cases have seemed to saturate around 3.15, and 548.95 mV, respectively. This has happened possibly because the chemical compounds have reached a stable state [34].

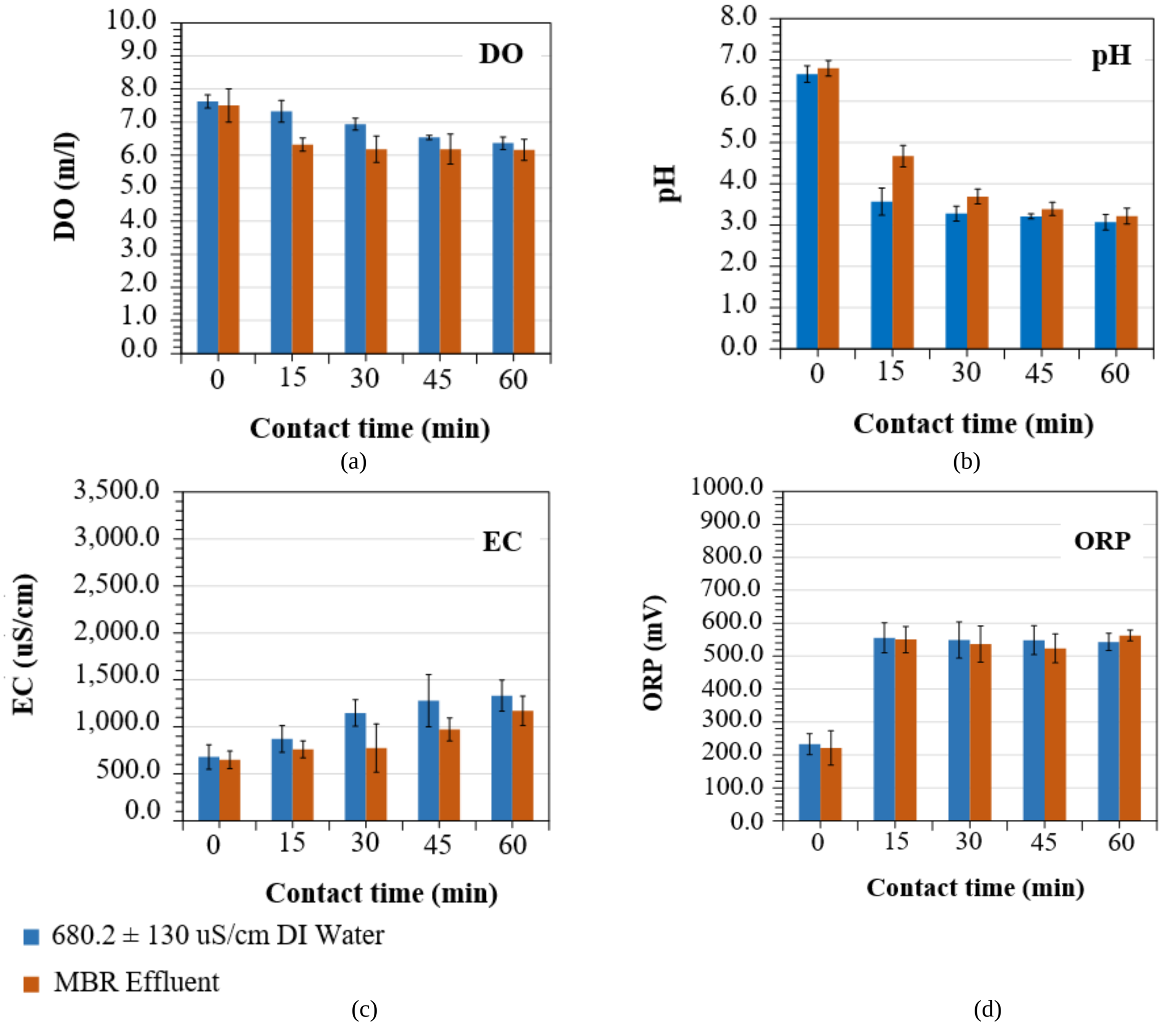



**Figure 4** (a) DO, (b) pH, (c) EC, and (d) ORP of liquid media samples after air plasma treatment at various treatment times.

*3.3 Effect of potassium chloride during plasma treatment*

In order to study the effect of KCl concentration on the nitrate-nitrogen conversion during plasma treatment, the KCl solution has been prepared with different levels of concentration at 2, 4, 6, and 12 mol/l, resulting in the EC of DI water become 361.3 ± 21.3, 700.2 ± 23.5, 1265.7 ± 83.1, 2153.3 ± 83.1 µS/cm respectively. The nitrate nitrogen concentration of every KCl concentration has been rapidly increased during the first 15 min and then gradually declined, as illustrated in Figure 5. It can be observed that the trend has been also the positively skewed bell curve. From the experimental results and the statistical analysis in this study, it could be implied that the KCl (in the range mentioned above) dissolved in DI water has not significantly affected nitrate-nitrogen yield. However, it could enhance the plasma generation in terms of increasing the electrical conductivity of the liquid electrode, as discussed in section 3.2 [17, 18, 28-32].

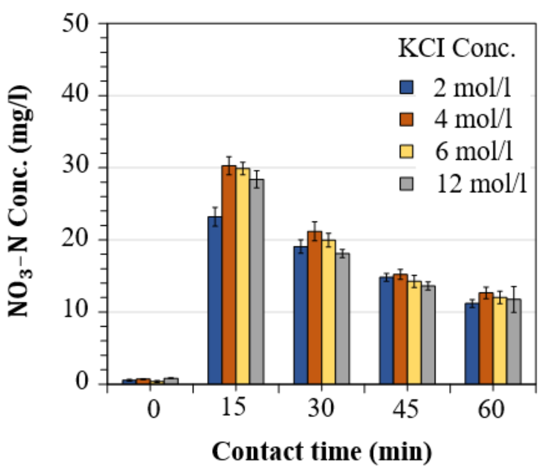



**Figure 5** The changes of nitrate-nitrogen yield in various KCl concentrations during air plasma treatment at various treatment times.

## 4. Discussion

From the experimental results, it could be confirmed that the atmospheric non-thermal air plasma jets can be applied effectively in order to improve nitrate-nitrogen concentration and other parameters such as pH, ORP, and EC of the treated liquid media. The influence of air plasma on active constituents in the plasma-treated liquid media would be explained by the interaction among reactive radicals with the treated liquid media, and physicochemical properties of a treated liquid media.

### *4.1 The influence of reactive species with the treated liquid media*

The hypothetical mechanism of nitrogen variance during plasma treatment caused by the reactive species (ROS and RNS) can be seen in Figure 6. The mechanism of the interactions of reactive species generated during air plasma generations with the treated liquid could take place in three primary phases, which are the reaction in the gas phase, the gas-liquid interface phase, and the bulk liquid phase. Firstly, the short-live radicals, photons, electric field (EF), UV radiation, and shock wave have been generated in the gas phase; these species would be called primary species. At the gas-liquid interface, the rather long-live radicals would interact with other chemical compounds of liquid, causing secondary reactive species and dissolved into the bulk liquid phase. The long-live radicals would remain in the bulk liquid phase and would continue reacting with other chemical compounds in bulk liquid [11, 35].

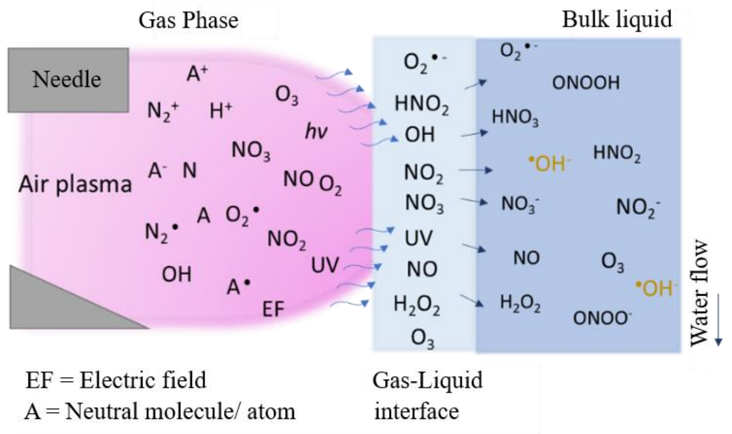



**Figure 6** The schematic drawing of the interactions of reactive species generated during air plasma generations with the treated liquid.

### *4.2 Physicochemical properties of liquid*

The influence of air plasma on active constituents in a plasma-treated liquid would be explained by the physicochemical properties of the treated liquid and the interaction among reactive radicals and the treated liquid. In the experiments, changes in water properties, which correspond to the change of reactive species level and concentration, have been observed both physically and chemically. As noticed in Figure 4(b), the pH of the liquid has drastically decreased with an increase in treatment time, implying that the presented treatment has led to water acidification. Kaushik et al. reported that the acidification of aqueous liquids through the plasma reaction results in the generation of reactive chemical species, including hydrogen peroxides ($H_2O_2$), nitric acid ($HNO_3$), and peroxynitrous acid (ONOOH) [11]. Dissolved oxygen has also slightly decreased with an increase in treatment time due to the chemical reactions in the liquid phase. Some typical reactive chemical species, which consume oxygen, are ozone ($O_3$), $H_2O_2$, and organic peroxyl radicals ($ROO^-$) [7]. The formation of those reactive species during plasma treatment has also contributed to an increase of EC and ORP in the plasma-treated liquid media [11]. Since ORP is considered as the most important factor affecting on microbial inactivation [36], this could imply that the plasma-treated liquid media has another potential ability for inactivation of microorganism.

### *4.3 The influence of air plasma on nitrate generation*

Regarding the experimental results in section 3.2, it could be explained that the nitrate generated in the solution comes possibly and majorly from the dissolution of nitrogen oxides ($NO_x$) formed in the gas phase during plasma treatment. In the elementary processes of air discharge, several reactive species, including the atomic species (N and O) and the excited states, are produced. They further react with others to form nitric oxide (NO) in significant amounts [37]. In this experiment, NO bands of the optical emission spectrum, as seen in Figure 2(c), can be observed. The produced NO can be further oxidized to $NO_2$ and other $NO_x$ and then dissolved into the solution to form nitrite ($NO_2$) and nitrate ($NO_3$) [37], accompanied by acidifications of the solution as seen in Figure 4(b). Therefore, the enhancement of nitrate concentration in liquid media by optimal air plasma treatment could be confirmed, and possibly employed in the agricultural application as liquid fertilizer.

Moreover, the experimental results in Figure 3 also show that the concentration of TN and $NO_3$-N in MBR effluent have been higher than the ones of the EC-adjusted DI water. This would be due to the initial concentration offset of TN, and $NO_3$-N concentration in MBR effluent. However, it could be noticed that the increase of TN, and $NO_3$-N in the case of MBR effluent has been relatively slower than the one of DI water. This would be possible due to absorbing reactive radicals reaction by organic compounds since the MBR effluent used in this study still remains some COD concentration [11].

Several researches have reported that the long-lived nitrate ions in plasma-activated water are formed as the secondary products and extend the anti-microbial activity on storage [38, 39]. The mechanism for nitrite ion induces microbial inactivation is that $NO_2^-$ reacts with $H_2O_2$ in acidified liquid to form a strong oxidant peroxynitrite acid (ONOOH and $ONOO^-$) which is strongly dependent on pH [40]. In this study, the liquid pH has decreased dramatically during plasma treatment, and so the peroxynitrite is mainly in the form of ONOOH, which is much more reactive than $ONOO^-$. These results also show a possible role of acidified nitrite on plasma-induced microbial inactivation. However, the detailed mechanism of plasma-induced acidified nitrite needs further investigation.

**5. Conclusions**

This study has demonstrated that an atmospheric non-thermal air plasma jet could be applied possibly and effectively in order to improve the nitrogen concentration in MBR effluent. The effect of treatment time on treatment efficiency has been investigated. The results indicate that an optimum plasma treatment time in this study has been 15 min resulting in the highest percent of the enhancement. The reduction of nitrate-nitrogen after a long-time plasma treatment can also provide essential knowledge for the possibility of nitrate removal by air plasma. It has been found out that the variations of EC of treated liquid media have not significantly affected nitrate-nitrogen yield, but it has enhanced the plasma generation. It has been hypothesized that the enhancement of nitrate is due to the formation of atomic species (N and O) in the air discharge, which can be further oxidized to $NO_2$ and $NO_3$. From this study, it could be confirmed that not only air plasma could enhance nitrate-nitrogen concentration in the MBR effluent, which could be employed as liquid fertilizer, but an acidified nitrite in the air plasma treated MBR effluent could also be possible for microbial inactivation.

**6. Acknowledgements**

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