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The Effect of Air Injection Flow Rate towards The Formation of Radicals in Liquid Glow Discharge Plasma Electrolysis with K_2SO_4 Solution

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Abstract

Glow discharge is a part of plasma formation phenomenon which occurs on the electrode side in contact with the electrolyte solution, started with an electrolysis reaction with direct electric current. In this research, 0.02 M K_2SO_4 electrolyte, tungsten anode and stainless-steel cathode was used in a direct current plasma electrolysis reactor. The plasma formation is demonstrated by a strong current and voltage characteristic curve. In the first stage, characterization was carried out in the form of current and voltage strength measurement at various air-injected flow rates in order to obtain the position of the glow discharge zone. In the second stage, measurement of the emission intensity of radicals was carried out to identify the radicals formed and in the third stage, the nitrate produced was measured. There are three plasma formation zones: the ohmic zone, the transition zone and the glow discharge plasma zone. Air injection affects the formation of glow discharge plasma and radicals. In this study, emission intensity measurements were carried out to determine the radicals that contribute to the formation of nitrate in nitrogen fixation using plasma electrolysis. Without the injection of air, radicals formed only •OH, •H, and •O with emission intensities of 20012 a.u, 10121 a.u and 10245 a.u. air injection of 0.8 $L.min^{-1}$ produced radicals •OH, •N, $•N_2*$, $•N_2^+$, •H and •O with emission intensities of 30863a.u, 20139 a.u, 28540 a.u, 18023 a.u, 12547 a.u and 49800 a.u, respectively. Many radicals are generated when the plasma reaches its stability. In addition, at 0 $L.min^{-1}$, 0.2 $L.min^{-1}$, 0.4 $L.min^{-1}$ and 0.8 $L.min^{-1}$ stable plasma were formed at 675 V, 660 V, 650 V and 650 V. It is concluded that the injection of air accelerates the formation of a gas envelope to reduce energy.

Keywords: air injection, radicals, emission intensity, glow discharge, plasma electrolysis

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INTRODUCTION

Electrolysis is a method of separating ionic compounds into ions by passing an electric current between two inert electrodes in solution. Negative ions will be excited towards the anode and vice versa. Plasma electrolysis has the same principles as general electrolysis. The difference lies in the use of high voltage in plasma electrolysis, resulting in an electric discharge. As a result, the electrons will be excited and cause the gas to ionize and form a plasma emission (glow discharge) (Saksono et al., 2016)

Plasma electrolysis is an electrolysis process at a high voltage which causes the excitation of electrons so that electric sparks arise and generate plasma in an electrolyte solution. Plasma electrolysis combines the principle of plasma formation in the gas phase and electrolysis (Harianingsih et al., 2021). Plasma produces radicals that break the bonds in the electrolyte and nitrogen in the air. Direct current is generated between the electrode and the surface ofelectrolyte surface so that the glow discharge electrolysis occurs (Salsabila et al., 2020). The process of plasma electrolysis was first initiated by the Faraday electrolysis process at low voltage and then formed gas bubbles around the electrode. The reaction that occurs in the Faraday electrolysis is a oxidation-reduction (redox) reaction, as it can be seen in the following reaction equation (Rumbach et al., 2013):

Anode : $2H_2O + 2e^- \rightarrow H_{2(g)} + 2OH^-$ (1)

Cathode : $2H_2O \rightarrow O_{2(g)} + 4H^+ + 4e^-$ (2)

This electrolysis reaction is exothermic, where the amount of H_2 and O_2 produced in a redox reaction is directly proportional to the electric charge applied. This is following Faraday's law,as the voltage increases, the plasma begins to form in the solution. At high voltage, there is an anode with an oxidation reaction, a cathode with a reduction reaction, and a low voltage (Faraday Electrolysis). Plasma electrolysis decomposes water molecules into •OH and •H (Jiang et al., 2014). The discharge caused by the formation of plasma creates conductive channels so that electrons with high energy are generated in the electrolyte solution in the form of ionization, dissociation, and recombination of water molecules. The simple mechanism of the process is shown in the following equation (Bruggeman et al., 2016):

$$H_2O_{(g)} \rightarrow \bullet OH + \bullet H \quad (3)$$

The electrolyte solution consists of water and compounds with high conductivity values. Water has a relatively high dielectric constant (Ɛr = 81) and density (ρ = 1000 kg m^{-3}), while gas has a lower dielectric constant than water (Ruma et al., 2015). Therefore, a large amount of energy is required to initiate the discharge and formation of plasma in water. Air injection can reduce the power of plasma formation so that the initiation of plasma discharge formation becomes easier (Farawan et al., 2022). In addition, because the density of gas bubbles is smaller than that of water, the plasma formed in the presence of air injection appears to be larger than without air injection. Thus, adding gas injection into the solution can reduce the energy required for plasma formation. The direct injection mechanism at the electrode causes the plasma to dimmer and smaller so that it becomes less stable and fewer radicals are formed (Farawan et al., 2021).

The main problem that exists in plasma electrolysis research is the use of high voltages. Therefore, this study aimed to determine the effect of air injection flow rate to identify the formation of plasma zones on the current-voltage curve, as well as identify the presence of radicals that affect the formation of nitrate in the nitrogen fixation process.

In this study, observations were made on the improvement of the nitrogen fixation process using the anodic plasma electrolysis method either with or without air injection, which until the time this research was conducted had not been reviewed in previous studies. The focus of this research is on the formation of reactive species due to plasma discharge either with or without air injection. The reactive species formed will later show effectiveness in forming nitrogen fixation products, especially nitrate.

MATERIALS AND METHODS

Materials

0.02 M Potassium sulfate Merck 1.05153.0500 (Merck, Germany) was used as an electrolyte solution. Air injection (N_2/O_2=79/21) used was from the compressor with gas injection flow rate of 0, 0.2, 0.4, and 0.8 L.min^{-1} at 700 V. Tungsten EWTH-2 RHINO GROUND with size 1.6 mm x 175 mm was used as the anode and placed in a glass sheath and maintained by a length of tungsten immersed/contacted in the electrolyte solution at the end of the sheath at 5 mm long. Stainlesstel AS SUS 316 DIA, inert as a cathode (25 cm long with a diameter of 6 mm) is placed 5 cm from the tungsten.

Methods

A 1.5 L glass batch reactor, with diameter of 10 cm and 20 cm high, was used for the experiment. The reactor was equipped with a DC and condenser to maintain the operating temperature at 60 °C. The cathode and anode were separated by 2 cm apart and connected to an electrical equipment. The system had a total liquid volume of 1.2 L, consisting of 0.02 M K_2SO_4 as an electrolyte. A representative experimental setup is shown in Figure 1.

The effect of air injection flow rate on glow discharge plasma electrolysis was observed by measuring the current and voltages at flow rate variations of 0 L.min^{-1}, 0.2 L.min^{-1}, 0.4 L.min^{-1} and 0.8 L.min^{-1} with a voltages range of 200-750 V plotted in a plasma zone graph. The phenomenon of plasma formation can be explained using the relationship curve between currents and voltages (I-V curve). The I-V curve has a unique characteristic during plasma electrolysis. Radicals production using an air plasma electrolysis reactor was conducted in the following series of operating conditions: (1) the anode was 2 cm deep from the liquid surface with a constant electrical power of 400 Watt; (2) the flowrates pump circulation varied at 0 $L.min^{-1}$, 0.2 $L.min^{-1}$, 0.4 $L.min^{-1}$and 0.8 $L.min^{-1}$; (3) the operating temperature was maintained at 60 °C; (4) the pressure was maintained at 1 atm; and (5) the initial pH was maintained 6.

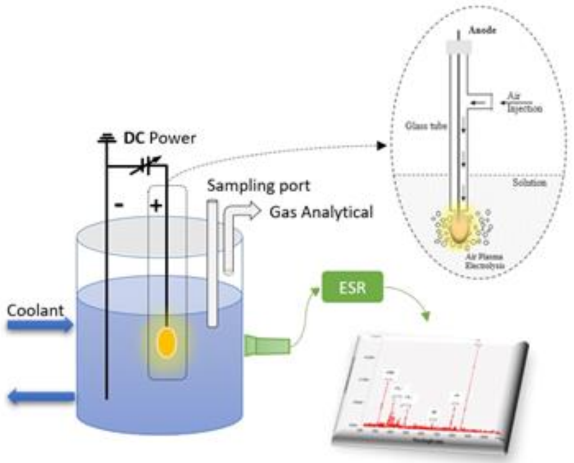

Figure 1: Experiment Apparatus

The intensities emission spectrum of radicals was tested with an analyzer connected to a personal computer. Electro spine resonance (ESR) spectroscopy was used to analyze the intensity of the emission spectrum, which was connected to an optical probe, carried out in a dark room to determine the gas formed in the reactor due to the release of plasma at the electrode and to eliminate other light waves received by the camera. The ESR has tuned at 200–1100 nm with a very high UV-NIR response sensitivity using an ICCD (Intensive CCD) camera placed perpendicular to the plasma splash, where the signal time remained at 1 ms (Luvita et al., 2022). Nitrate test was carried out using Merck HACH 2106169 (Merck, Germany). 1 ml of sample was dissolved in distilled water then added 1 g of reagent, stirred, and allowed to stand for 15 minutes. Nitrate was then measured using BEL Engineering UV-M51 Single beam spectrophotometer at a wavelength of 514 nm (Harianingsih et al., 2021).

RESULTS AND DISCUSSION

In observing the glow discharge of the plasma electrolysis process, the addition of a gas in the form of air with a direct injection mechanism into the anode area can assist in initiating the ionization process in plasma formation to reduce the amount of energy consumption needed for the evaporation process in Joule heating (Budikania et al., 2019). In addition, the gas injection mechanism and the gas flow rate can affect the physical and chemical characteristics of the formed plasma. Air injection in the plasma zone reduces the energy consumption of H_2O evaporation due to the effect of joule heating (Saksono et al., 2016).

The plasma glow discharge zone is very important in nitrogen fixation using the plasma electrolysis method. This zone is a location where plasma begins to have a stable form and the presence of radicals that affect the formation of nitrate begins to increase. In addition, nitrate products are produced in this zone. Meanwhile, we know that nitrate is currently being developed as a safe and environmentally friendly nitrogen fertilizer as well as a fixation process using plasma electrolysis that does not use natural gas and does not produce CO_2 emissions, such as making ammonia fertilizer using the Haber Bosch process.

Table 1 shows that plasma formation energy with air injection is lower than without air injection. In the air injection flow rate variation, the higher the flow rate, the lower the required plasma formation energy. This is due to the presence of oxygen in the air, where oxygen has a low ionization potential (Ahmed et al., 2016).Thus, the greater the flow rate of the air injected directly into the plasma, the more the ionization process during plasma formation requires energy. The addition of air injection into the plasma zone through the electrode sheath can reduce energy consumption in the H_2O evaporation process from the joule heating effect process. the energy consumption of plasma formation with air injection is lower than without air injection. In addition, plasma is more easily formed in gases that have a lower dielectric constant than water. At variations in the air volume flow rate, the higher the air volume flow rate, the lower the energy required for plasma formation. This is due to the presence of oxygen in the air where oxygen has a low ionization potential (Ahmed et al., 2016). So that the greater the flow rate of air injected directly into the plasma, the ionization process during plasma formation requires less energy. The mechanism for the formation of plasma electrolysis at each increase in voltage can be observed through the voltage and current characterization curve shown in Figure 2.

Figure 2 shows that the current-voltage characterization curve in plasma electrolysis using K_2SO^4 electrolyte and air injection can be identified in three zones formed as follows: (1) Ohmic zone; in this zone, the current increases linearly with increasing voltage. In this condition, conventional electrolysis occurs, characterized by the emergence of gas bubbles around the electrode. The reduction potential of H_2O is greater than that of the alkaline K^+ group, so reduction occurs at the cathode. On the anode side, the water is oxidized due to the reduction potential of H_2O being more significant than the SO_4^{2-} ion. This follows equations (1) and (2) (Rumbach et al., 2013).

The increase in voltage during conventional electrolysis is carried out until the highest breakdown voltage (V_B) 310 V and breakdown energy (E_B) 480 Watts are reached). (2) Transition Zone; there is a decrease in current with increasing voltage due to the formation of a gas envelope around the anode. The formation and breakdown of the vapour envelope give rise to plasma sparks and current instability.

Table 1: Effect of Air Injection Flow Rate on Voltages and Powers Values in 0.02 M K_2SO_4 Electrolyte Solution

Flowrate (L.min^{-1})	V_B (V)	I_B (A)	E_B (Watt)	V_D (V)	I_D (A)	E_D (Watt)
0	310	1.55	480	675	0.33	220
0.2	310	1.52	470	660	0.32	210
0.4	305	1.51	460	650	0.32	205
0.8	300	1.33	400	650	0.31	201.5

*V_B: breakdown voltage, I_B: strong breakdown current, E_B: breakdown energy, V_D: critical voltage, I_D: critical current strength, E_D: Critical energy

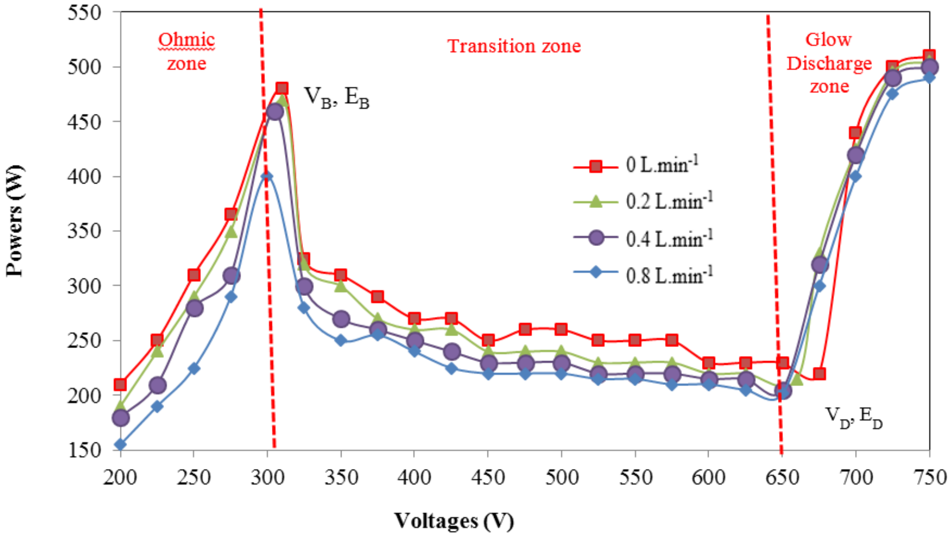

Figure 2: Voltage-Power Characterization at Different Air Injection Flow Rates

A vapour layer is formed at the anode, causing the anode and the solution not to contact, the current can no longer increase, and there will be a decrease in the increase in voltage. The lowest current of the transition zone is achieved at a critical voltage (V_D) of 650 V with a binding energy (E_D) of 201.5 watts. In this zone, the production of radicals is not maximized because the plasma formed is not stable. (3) Glow discharge zone; the current begins to increase with an increase in voltage which is indicated by bright and bright plasma on the end side of the tungsten anode contact with the electrolyte solution. The increase in critical voltage (VD) is directly proportional to the increase in binding energy (ED), and many radicals are formed. This is due to the low dielectric strength of the electrolyte, which causes higher discharge strength at higher flow rates and the formation of a lighter plasma (Ahmed et al., 2016).

After passing this voltage, the glow discharge state has been reached. However, even at almost the same voltage, the resulting current is different. It can be seen that the greater the air flow rate, the smaller the current value at the same voltage. This is because the presence of airflow at the anode can interfere with the current entering the electrolyte solution so that the measured value becomes smaller. In Figure 2, it can be seen that the current value at 0.8 L.min^{-1} is lower than 0.2 L.min^{-1}, 0.4 L.min^{-1} and 0 L.min^{-1}. A significant air flow rate will actually make the plasma size more significant because of the large air envelope. The difference in plasma visualization between the three can be seen in Figure 3. At the highest flow rate, the incoming current is effective for producing reactive species. A higher voltage or power has a higher effective air flow rate. The radicals formed without gas injection are only in the form of •OH, •H, and •O, as shown in Figure 4. Radicals are formed more due to the dissociation process of water molecules in the plasma zone. Air injection in the form of N_2 and O2 gases encourages the formation of •OH, •N, •N_2*, •N_2^+, •H and •O, as described in Figure 5, while the emission intensity values in Figures 4 and 5 are presented in Table 2. Table 2 shows the radicals generated by air injection at 0 L.min^{-1} and 0.8 L.min^{-1} plasma electrolysis. Without air injection (0 L.min-1), •N, •N_2*, •N_2^+ were not generated even though the emission intensities of •OH, •H and •O adsorbed were 20012 a.u, 10121 a.u and 10245 a.u, respectively. Whereas using air injection, radicals are in the form of •N_2*, •OH and •O were 28540 a.u, 30863 a.u and 49800 a.u, respectively. According to the research of Kučerová et al. (2020) if there is an O atom as a result of O_2 ionization by high-energy electrons, which then binds to •N from the solution, it will form NO and then oxidizes to NO_2 and NO_3 according to equations (4) and (5) (Saksono et al., 2023).

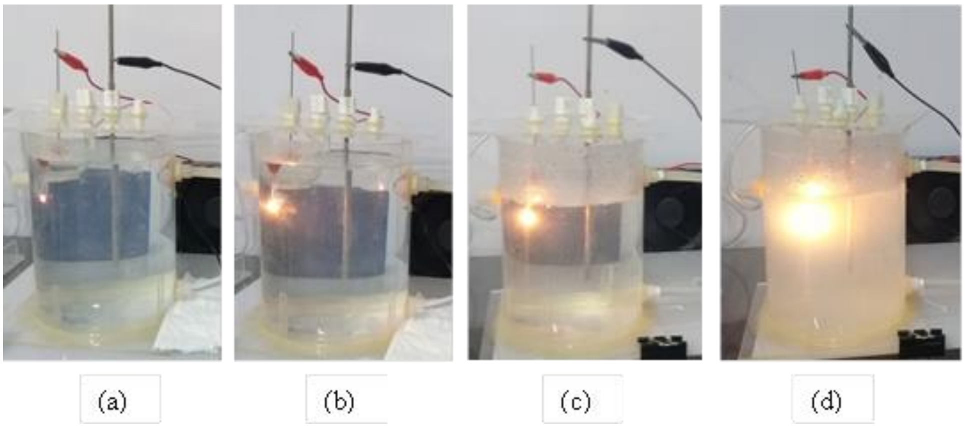

Figure 3: Plasma on Variation of Air Injection Flow Rate (a) 0 $L.min^{-1}$, (b) 0.2 $L.min^{-1}$, (c) 0.4 $L.min^{-1}$ and (d) 0.8 $L.min^{-1}$

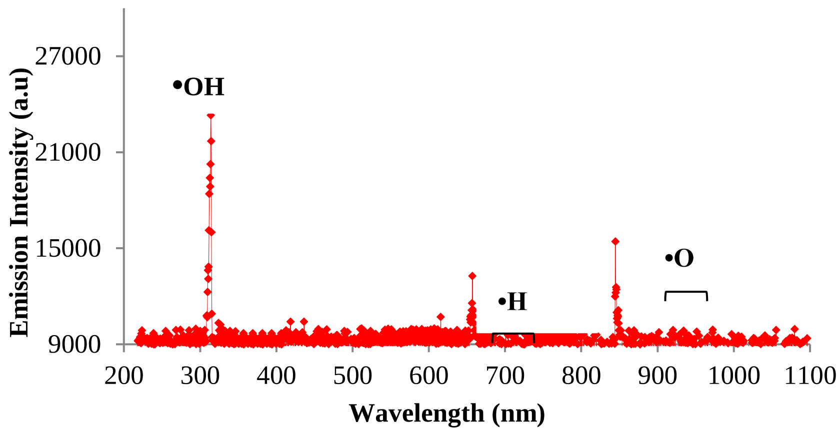

Figure 4: Radical Emission Intensity Without Air Injection

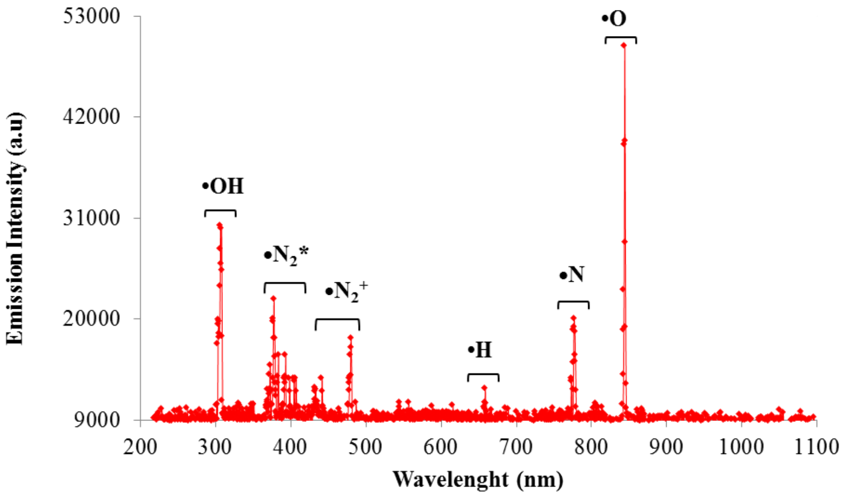

Figure 5: Radical Emission Intensity With Air Injection Flowrate 0.8 $L.min^{-1}$

Table 2 : Radical Emission Intensity without Air Injection and with Air Injection at 0.8 $L.min^{-1}$

Injected air flow rate ($L.min^{-1}$)	Radical emission intensity (a.u)						Nitrate (ppm)
	N	N_2*	N_2^+	•OH	•H	•O	
0 (non injected)	-	-	-	20012	10121	10245	-
0.8	20139	28540	18023	30863	12547	49800	1889

Wavelength: •OH (306 nm), •H (654 nm), •O (844 nm), •N_2 (317 nm), •N_2^+ (479 nm), •N (777 nm)

$$N_2 + e^- \rightarrow \bullet N + \bullet N + e^- \quad (4)$$

$$O_2 + e^- \rightarrow \bullet O + \bullet O + e^- \quad (5)$$

The addition of air injection produces optimal nitrate because it increases the input of nitrogen and oxygen into the plasma zone resulting in the formation of •O and $\bullet N_2^*$, which results in the addition of three •OH. The presence of $\bullet N_2^*$, •OH and •O also plays a role in the formation of NO, and it can be seen that high •O will produce nitrate, meaning that more NO is formed to be oxidized to NO_2, which eventually forms NO_3 (Saksono et al., 2023).

$$\bullet O + H_2O_{(l)} \rightarrow \bullet OH + \bullet OH \quad (6)$$

$$H_2O_{(l)} + e^- \rightarrow \bullet H + \bullet OH + e^- \quad (7)$$

$$N_{2(g)} + H_2O_{(l)} \rightarrow \bullet N_2^+ + \bullet OH + \bullet H \quad (8)$$

$$N_{2(g)} + \bullet O \rightarrow NO + \bullet N_2^* \quad (9)$$

$$\bullet N_2^* + \bullet O \rightarrow NO + \bullet N \quad (10)$$

$$2NO_{2(g)} \rightarrow N_2O_{4(g)} + H_2O_{(l)} \rightarrow HNO_{3(l)} + HNO_{2(l)} \quad (11)$$

$$NO_{2(g)} + NO_{(g)} \rightarrow N_2O_{3(g)} + H_2O_{(l)} \rightarrow 2HNO_{2(l)} \quad (12)$$

$$NO_2 + \bullet OH \rightarrow HNO_3 \quad (13)$$

At conditions of air injection rate of 0.8 $L.min^{-1,}$ there is a good balance between electric power and air injection rate which produces various radicals such as •OH, •N, $\bullet N_2^*$, $\bullet N_2^+$, •H and •O. Emission spectrum analysis was carried out in a dark room to determine the gases formed in the reactor due to the release of plasma at the electrodes to eliminate other light waves received by the camera. The emission spectrum analyzer is tuned at 200–1100 nm wavelength with a very high UV-NIR response sensitivity (Tanski et al., 2019). The camera is an ICCD (Intensive CCD) placed perpendicular to the plasma splash, with a fixed signal time of 1 ms. The light spectrum of the resulting plasma release is translated into a graph of 300–850 nm, containing •OH (308 nm), $\bullet N_2^*$(317, 337, 380, 399 nm), $\bullet N_2^+$ (427, 479 nm), •H (654 nm), •N (777 nm), •O (844 nm). For emission spectra emitted at various camera positions, the results are dominated by excited nitrogen molecules ($\bullet N_2^*$) due to the high-energy collisions of electrons with N_2 and O_2 molecules (Liu et al., 2012). The density of •OH increases with the electron density. A higher voltage induces a larger current and results in a higher electron density; However, the decomposition energy efficiency decreases at higher application voltages. High conductivity results in a broader release in oxygen bubbles, increasing the intensity of O emission (Saito et al., 2015). Species • OH is a radical with the highest oxidation state that can oxidize nitrogen from the air to nitrate. •OH species are widely produced by plasma due to gas ionization from the heating effect of joules of water from the solution (Sen Gupta, 2015). The water molecule will split into •OH because of its high energy electrons that are excited to the plasma. •OH has a short residence time of about 3.7 x 10-9 seconds (Jiang et al., 2014).

The formation of •OH during the process of plasma electrolysis comes mainly from the dissociation of water molecules caused by electron collisions. In this study, the injection of air containing oxygen and nitrogen can increase the formation of •OH. Dissociation of oxygen by electrons forms oxygen radicals which will then react with water to form •OH based on the reaction equation in the gas phase (Yasuoka & Sato, n.d.). Water molecules in the gas phase will dissociate to form •OH in the gas phase, which then diffuses into the solution. In addition, the electrons formed in the plasma discharge will also react with water molecules in the gas-liquid interface to form •OH. The formation of •OH can also come from the ionization of water molecules by electrons followed by the reaction of H_2O^+ ions with other H_2O molecules. However, the H_2O^+ reaction with H_2O molecules is unlikely to occur because it requires a more significant electron energy of 12.6 eV compared to the electron energy for H_2O dissociation of 6.4 eV. In addition to oxygen, excited nitrogen molecules will dissociate water molecules to form •OH and •H (Magureanu et al., 2008).

CONCLUSION

The air injection flow rate has a significant influence on the formation of glow discharge zones in nitrogen fixation using plasma electrolysis. At a flow rate of 0.8 L.min-1, plasma stability is achieved so that •OH, •N, •N2*, •N2+, •H, •O radicals are formed. In contrast, without injection of air (0 L.min-1) only •OH, •H, •O radicals are formed. So that with the injection of air, nitrate will be formed from the reacting radicals of 1889 ppm.

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