

## Study of Plasma Electrolysis in Water: Alternative Technologies for Hydrogen Production

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### Abstract

Plasma electrolysis has been studied as an alternative method for generating hydrogen. A plasma reactor is used in this electrolysis to produce glowing plasma and the reactive interface fluid layer has an asymmetrical spiral-rod electrodes configuration. Plasma electrolysis was carried out in 2 positions at the surface of the solution and at a depth of 5 mm. On the surface, it is divided into 4 regions of I-V characteristics, namely the conventional electrolysis area, the plasma formation region, the gas formation area that covers the anode, and the glowing plasma region. While at a depth of 5 mm, the formation of plasma with a voltage of 500 V takes 120 seconds to heat the solution with a conventional electrolysis mechanism around the anode. Glowing plasma at a depth of 5 mm resembles the shape of a ball with a larger diameter as the addition of stress and the addition of the concentration of NaCl solution. The mechanism for the formation of hydrogen molecules occurs in the reactive interfacial fluid layer. The plasma ball will increase with a greater voltage. The enlargement of the glowing plasma with increasing operating voltage shows that voltage is a very important factor in the generation of plasma in water. The diameter of the glowing plasma sphere produced for electrolysis at the surface of the solution and at a depth of 5 mm in the solution always increases with increasing voltage. Furthermore, the greater the molarity of the NaCl solution in water, the larger the glowing plasmasphere. The production of hydrogen increases as the voltage increases, this is indicated by the increase in spectrum intensity  $H_{\delta}$  and  $H_2$ .

**Keywords:** Plasma electrolysis, Contact glow discharge electrolysis, Hydrogen production, Plasma glow

### Introduction

Hydrogen is the most attractive alternative fuel because it is environmentally friendly with the ability to react with air 3 times compared to hydrocarbons. Various technologies have been tried to produce hydrogen. Plasma electrolysis is an efficient method for producing hydrogen [1,2]. To get hydrogen continuously with efficiency over conventional electrolysis efficiency, it is necessary to control the electrode surface conditions, plasma electrolysis temperature, current depth, and input voltage. Plasma electrolysis at a voltage of 120 V produces as much as 80 times more hydrogen than conventional electrolysis at 300 V [1]. If plasma electrolysis lasts for a long time, large amounts of thermal energy will be obtained [3].

Mizuno [4] conducted a study using a 700-cc electrolyte solution of 0.2 M  $K_2CO_3$  which was placed in a 1,000 cc pyrex beaker. The electrodes used were mesh made of platinum as anodes and tungsten with a diameter of 1.5 mm, a length of 29 cm as a cathode. Based on the data recorded during the process, the heat energy released is 800 times higher than the input energy.

Sharma [6] reports their research that plasma electrolysis is a suitable method for producing steam continuously. The electrolyte solutions used are  $NaHCO_3$  and NaCl. The thermometer is placed at a different depth in the electrolyte solution. As a result, there is a difference in the temperature distribution of the solution between the surface and the bottom of the reactor. The solution around the plasma has a high temperature compared to the solution's temperature at the bottom of the reactor. This shows that plasma electrolysis can be used to produce steam continuously with an 80 % steam generation efficiency.

The interaction of the plasma with the liquid will allow the formation of a highly reactive interfacial fluid layer. Plasma products such as electrons, ions, photons, and radicals will increase the reaction ability, as has been discussed by Bruggeman *et al.* [6]. The ability of these highly reactive plasma species also

makes it possible to decompose water to produce hydrogen. Jin *et al.* [7] using a thin platinum anode in contact with an electrolytic solution, contact electrolysis of light discharge (CGDE) occurs at a sufficiently high voltage. In this study, the researchers used a solution of  $\text{Na}_2\text{SO}_4$  and  $\text{NaCl}$ . Glowing plasma in solution is supported by a direct current between the electrode and the electrolyte surface.

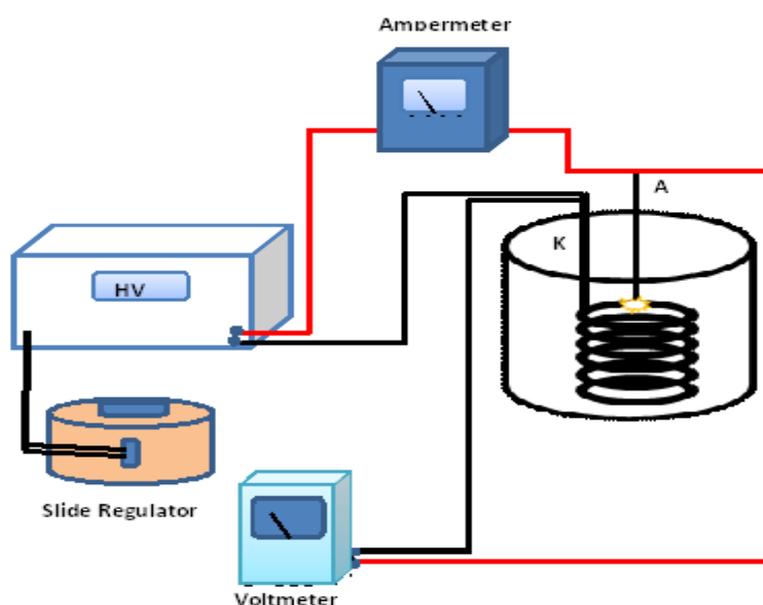
The review that has been done by Bruggeman *et al.* associated with the presence of a reactive interfacial fluid layer [6] and the results obtained by Mizuno *et al.* [1] that plasma electrolysis can produce much larger hydrogen than conventional electrolysis, as well as the results of research from Sharma *et al.* [5], has been used as a reference for using plasma electrolysis with the aid of  $\text{NaCl}$  solution [7] as a method for producing Hydrogen.

In this paper, the study of hydrogen production with plasma electrolysis technology using asymmetric electrodes in 3 different concentrations of  $\text{NaCl}$  solutions will be presented in this article. The study was conducted on 2 events, namely the formation of plasma on the surface of the solution and the formation of plasma at a certain depth of the solution.

## Materials and methods

The scheme of the experimental setup of on study plasma electrolysis in water can be seen in **Figure 1**. The plasma reactor is used in this electrolysis to produce glowing plasma and the reactive interface fluid layer in the form of 1,000 mL Pyrex glass. This reactor has an asymmetrical spiral-rod electrodes configuration. To produce plasma in solution, the anode was placed in the center of the spiral electrode circle. The anode was positioned perpendicular to the surface of the solution. Graphite anodes were made tapered at the ends, 8 cm long and 2 mm in diameter. Spiral cathodes used were made of stainless steel with a diameter of 4 mm, a length of 150 mm, and a spiral circle of 5 cm in diameter. We varied the concentration of  $\text{NaCl}$  in the solution including 0.3, 0.6 and 0.8 M. The increase of  $\text{NaCl}$  concentration aims to increase the conductivity of the solution. This difference in conductivity will result in a difference in the plasma formed, for example in the diameter of the glow plasma.

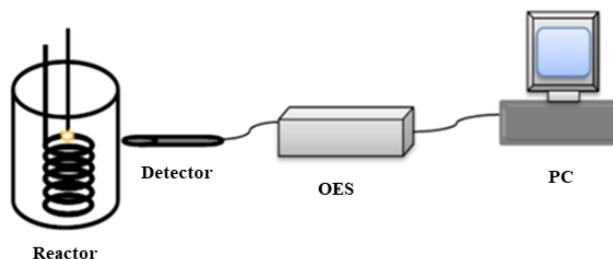
The voltage source used to generate plasma was a DC voltage source with a working area of 0 - 680 V, 2,000 VA produced by Matsunaga MFG and complementary by a slide regulator as a voltage divider. At the time of the characterization test, the stem electrode was installed at the conditions above the surface of the electrolyte solution. As for the depth test, the electrode will be immersed at a depth of 5 mm from the surface of the solution. The positive polarity of HV DC was connected with the Kyoritsu digital amperemeter to measure the current. Changes in electric current during the electrolysis process are measured as a function of time. The voltage measurement given from an HV DC source was done by a voltmeter Sanwa Japan YX360TRF.



**Figure 1** The experimental setup scheme for measurement and glow discharge plasma electrolysis reactor.

Glowing plasma in water due to the release of photons in the ionization and excitation process in the form of close to the “Plasma Ball” (then with the abbreviation PB), hereinafter referred to as PB. We measured the physical quantities consisting of PB such as the diameter of “ball”, electrolysis time, current, and voltage. By using the results of measurement, we make the comparison of physical properties before and after the formation of PB in a solution of Sodium Chloride (NaCl). Measurement of the time needed by the reactor in electrolyzing the solution using a timer on the Canon 20 MP digital camera. While the diameter measurement of PB has been produced using a ruler.

The emission spectrum of the PB was taken using Optical Emission Spectroscopy (OES). The CHEM4-UV-FIBER combines a USB4000 Spectrometer with modular accessories including an ISS-UV-VIS. The PB emission spectrum is used to identify what emissions occur as shown in **Figure 2**.

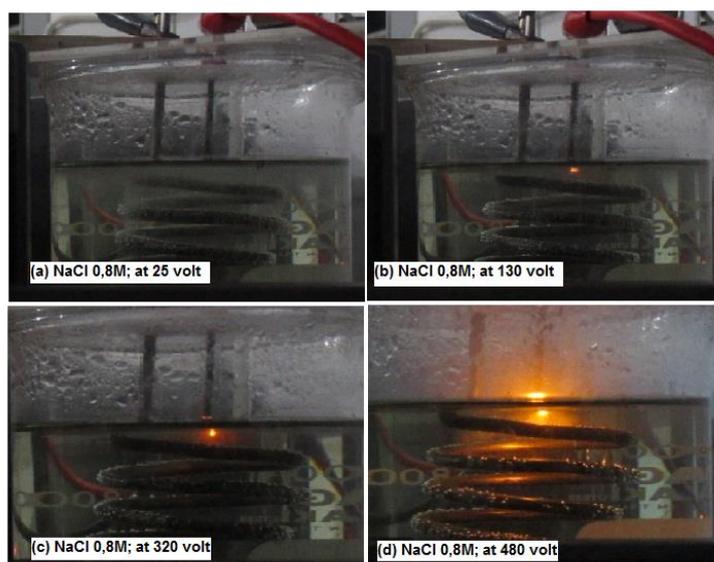


**Figure 2** Scheme of emission spectrum collection.

## Results and discussion

### Plasma formation in NaCl solution.

Plasma electrolysis occurs with the formation of plasma at the anode. From **Figure 3**, the greater the voltage, the greater the glowing plasma. When the voltage applied is 25 volts as shown in **Figure 3(a)**, there is a production of bubbles containing air. This bubble production is a result of the conventional electrolysis process occurring on the surface of the water given an electrolyte solution. If a lot of this gas bubble covers the anode, this causes the electric charge to be difficult to be transported to the cathode so that the current decreases dramatically. When the voltage continues to be increased, there is an electron avalanche or chain ionization (electronic avalanche) allowing the formation of plasma. This makes the charged particles in the gas collide with the surrounding particles and ionization and excitation occur following the release of photons, visible flares approaching the shape of the plasmasphere [6].



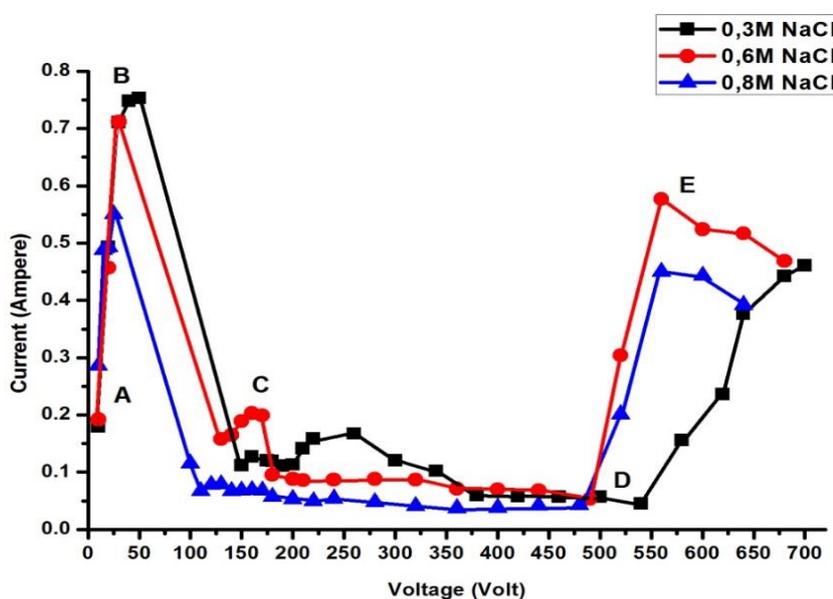
**Figure 3** Plasma formation for several applied voltage.

At a voltage of 130 volts for a 0.8 M NaCl solution, plasma flares occur at the end of the anode (see **Figure 3(b)**). The glowing plasma is still small and looks like a burning anode tip. Furthermore, photo in the **Figure 3(c)** shows plasma at 320 V, for a photo (d) glowing plasma at a potential of 480 V. Electrical arc discharge occurs continuously, and plasma enters a very unstable state. **Figures 3(c) - 3(d)** glowing plasma images increase in intensity along with the addition of voltage to the plasma reactor.

This intensity is the emission of an atom or molecule that experiences de-excitation.

#### Characterization of current as a function of voltage

The relationship curve between currents as a function of voltage for 3 variations of the concentration of NaCl electrolyte solution is 0.3, 0.6 and 0.8 M, shown in **Figure 4**. The electrodes used are graphite in the anode and stainless-steel spiral at the cathode. From the characteristic I-V relationship curve in **Figure 4**, there are several areas identified. Characteristics I-V can be classified into several regions. The first region is the area where conventional electrolysis (A-B) takes place. This area is indicated by the increasing current when the voltage is raised. This area is also called the ohmic region. In this area, small gas bubbles are formed due to the electrolysis process that takes place.

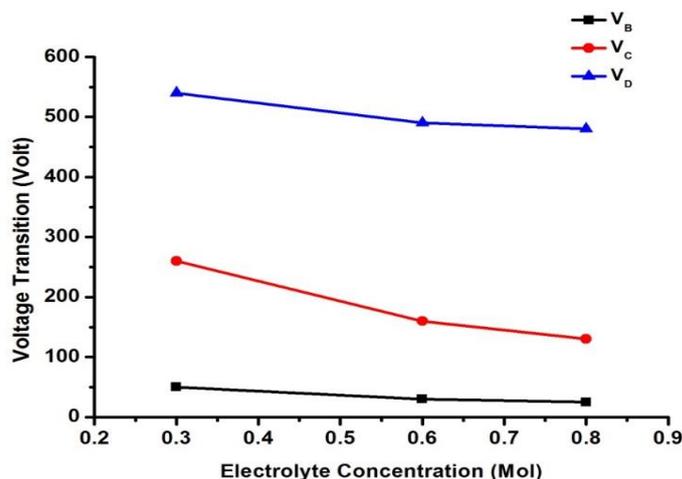


**Figure 4** Characterization of current as a function of voltage.

The next area is the plasma area that is formed, which is indicated by a sharp decrease in the current (B-C). Plasma is formed due to the transfer of energy due to collisions between charged particles with gas atoms and gas molecules from the vapor formed. Energy transfer can make the temperature of the system higher due to the Joule heating effect. Increasing temperature results in more evaporation in the anode region. Evaporation is what causes the electrode to be closed by water vapor bubbles so that electrical resistance increases and current decreases. The voltage is raised but the current does not change. This area reaches a critical voltage where the current increases sharply. The voltage at point B is a breakdown voltage, which is the voltage when the plasma begins to form. The next area is the gas casing stage covering the anode which causes the current to decrease (C-D). The current in this area will reach its lowest point, then rise again. The voltage when the current starts to rise sharply ( $V_D$ ) is called the midpoint voltage. This does not escape the presence of ions in electrolyte solutions which play a role in electron mobility towards the anode. With a greater number of electrons, relatively smaller energy is needed for excitation. The next area (D-E), with glowing plasma. The division of regions in the characteristics I-V above corresponds to those conducted by several previous researchers [7,8,10,11]. At the concentration of 0.3 M, the results were obtained as shown in **Figure 4**. The voltage return point ( $V_B$ ) is equal to 50 V with a current of 0.753 A. While the midpoint voltage ( $V_D$ ) is 540 V with a current of 0.054 Ampere. A variation of 0.6 M solution also shows similar characteristics. The turning point voltage ( $V_B$ ) is at a voltage of 30 V with a current of 0.712 A. The midpoint voltage ( $V_D$ ) is at a voltage of 490 V with a current of 0.053 A.

Based on the results obtained in this study, the higher the concentration followed by the higher the conductivity of the solution, the greater the turning point voltage ( $V_B$ ) shifts to the left or the smaller the turning point voltage (**Figure 4**). The reverse voltage point is obtained by the relationship that the higher the conductivity of the solution, the lower the voltage of the turning point. This is like what was stated by previous researchers [7,12]. High conductivity makes the current density also high at the same voltage and makes the energy dissipation at the anode higher. This causes collisions of ions around the anode to increase and the plasma will easily form. From the graph above, it is obtained that  $V_B$  for a concentration of 0.3 M is greater than  $V_B$  concentration of 0.6 M, and  $V_B$  concentration of 0.8 M is the lowest. The midpoint voltage ( $V_D$ ) characteristics of the 3 variations obtained  $V_D$  results for concentrations of 0.3 M greater than  $V_D$  for concentrations of 0.6 M, and  $V_D$  for concentrations of 0.8 M of the lowest magnitude (**Figure 5**). The pattern of midpoint voltage reduction is proportional to the increase in concentration and conductivity by what was obtained by previous researchers [7,8,12].

In his research, using platinum wire as an anode and stainless-steel rod as a cathode, [9] found that the midpoint voltage is getting smaller for NaCl solution with greater conductivity. Based on Debye-Huckel's theory that conductivity is directly proportional to the root concentration of electrolytes according to the formulation  $\sigma_t = \sigma_t^0 - (A + B \sigma_t^0) \sqrt{c}$  with  $\sigma_t$  is total conductivity,  $\sigma_t^0$  is the contribution conductivity of anions-cations, A and B are extension constants Dubye-Hukle's and c are electrolyte concentrations. In the same year with a different electrolyte solution,  $Na_2SO_4$ , [13] also found that the addition of electrolyte solution conductivity contributed to shifting the midpoint voltage to a lower voltage. This is because the high conductivity of the medium makes electrons move more easily which speeds up the formation of plasma.

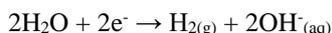


**Figure 5** Transition voltage as a function of electrolyte concentration.

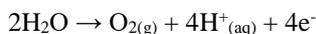
**The mechanism for the formation of radicals and hydrogen**

Electrolysis occurs in water given NaCl electrolyte solution there are 2 types of electrolysis. The electrolysis is conventional electrolysis or anion-cation separation that occurs in NaCl electrolyte solutions. After Na burns, heating occurs in water, forming water in the vapor phase, by energetic electrons produced in plasma flares will be able to decompose  $H_2O$  vapor. In the mechanism of water decomposition through plasma electrolysis, OH and H radicals will be formed. In decomposition of water through the plasma electrolysis process follows the mechanism that occurs among others [14,15];

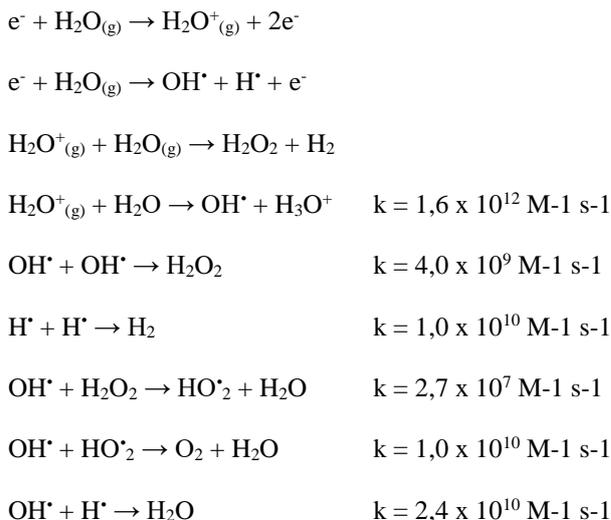
Reaction at the cathode



Reaction at the anode



$H_2O$  molecules in the gas phase are formed through the Joule heating effect which then triggers radical determination as the mechanism of the Hickling reaction due to the collision process [4,15,16];

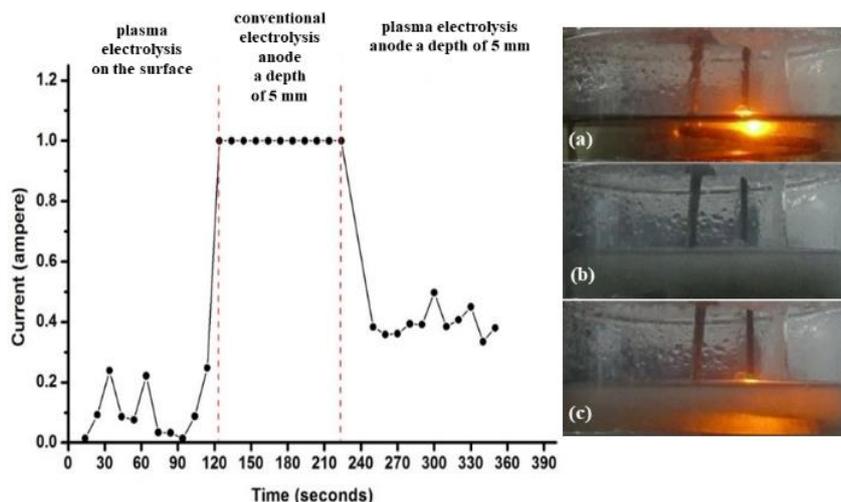


From the reaction equation above, OH<sup>•</sup> radicals have a short lifetime so it is easy to recombine to form longer-lived oxidizing molecules such as H<sub>2</sub>O<sub>2</sub> [16]. Furthermore, the OH<sup>•</sup> radical will also react with hydroxyl peroxide and hydrogen radicals to form water molecules again. The OH<sup>•</sup> radicals have a high oxidation potential of 2.80 eV. This is the reason for OH<sup>•</sup> radicals or hydroxyl can be used effectively to oxidize microorganisms and organic compounds that are difficult to decipher.

**Formation of plasma in the depth of the solution**

In **Figure 6** we can see, that when the anode was connected to a voltage of 500 V, the plasma directly forms on the surface of the solution. Within 120 s, the current generated shows an up and down rhythm with an average of 0.104 amperes. After the anode was inserted with a dipping depth of 5 mm, the plasma discharge is immediately lost and replaced with ordinary electrolysis with a constant current of 1 ampere. To bring up plasma glow, it takes time to heat the solution around the immersed anode for 120 s. After bubbles of gas bubbles are created and heated due to the heating joule effect, particles easily collide with energy transfer, which in turn results in subsequent excitation experiencing de-excitation by emitting emission, and the plasma glowing again. Starting seconds, the 250<sup>th</sup> current returns down and fluctuates up and down with an average of 0.394 amperes.

In plasma at a depth of 5 mm, the electric current is greater than the electric current in the plasma on the surface. Increasingly large currents are caused by the wider surface of the anode being immersed. With a larger immersed anode surface area, the energy needed to produce a gas envelope around the anode is higher [18,19].



**Figure 6** Current as a function of times and photograph of plasma electrolysis in solutions; a) plasma electrolysis on the surface, b) conventional electrolytic anode immersed to a depth of 5 mm, c) plasma electrolysis anode immersed to a depth of 5 mm.

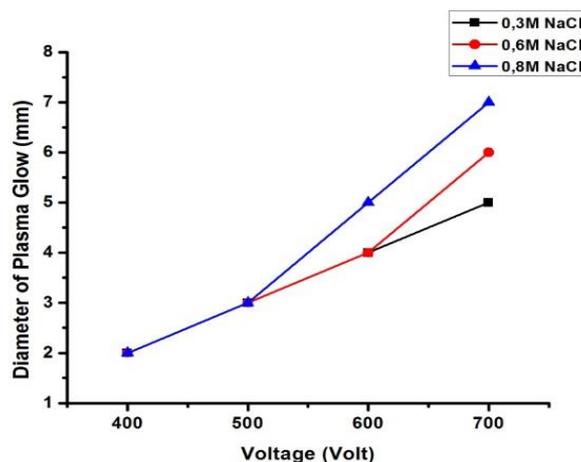
### Matrix plasma balls and plasma balls diameter

Discharge of gas into microplasma is formed around the active electrode. The heating process of gas incandescent (plasma glow) heats water around the plasma ball and produces more water vapor. The electrons from energetic ions undergo collisions with  $H_2O$  vapor and  $H_2O$  breaks into  $H^+$  and  $OH^{\cdot}$ . The process followed by plasma flares is getting bigger with the intensity getting brighter. After experiencing saturation, some species experience recombination again and are accompanied by a decreased inflow. Furthermore, it undergoes heating and will re-create radical species and ionize particles around the anode. The rising current and the plasma ball will be large glowing plasma along with higher light intensity. Enlargement of PB with increasing operating voltage can be seen visually in the photos shown in **Figure 7**. Based on these photos, it can be said that the operating voltage is very influential on the formation of a plasma in the water. According to Bruggeman *et al.* [6] associated with the presence of a reactive interfacial fluid layer. This interfacial fluid layer is where the chemical plasma occurs. The mechanism for the formation of hydrogen molecules occurs in the reactive interfacial fluid layer. The plasma ball will increase with the intensity of light getting higher, at the same concentration with a greater voltage. The enlargement of the glowing plasma with increasing operating voltage shows that voltage is a very important factor in the generation of plasma in water.



**Figure 7** Comparison of “plasma ball” glow for several applied voltages and NaCl concentrations.

**Figure 8** is a graph of plasma ball diameter as a function of voltage. The size of the PB’s diameter is influenced by the voltage and the concentration of the electrolyte solution. At a voltage of 400 - 500 volts, the diameter of the plasma ball is the same for all solution concentrations. At a voltage of 600 - 700 volts, the largest diameter of the spherical plasma is found at a concentration of 0.8 M, with smaller sequences for concentrations of 0.6 and 0.3 M. It can be concluded that in the condition that the plasma has been formed the greater the voltage given the greater the plasma zone formed, the greater the current produced. This increase in plasma ball diameter is strongly related to power. Electricity consumption used is multiplied by the current produced (in this case is a capacitive current).

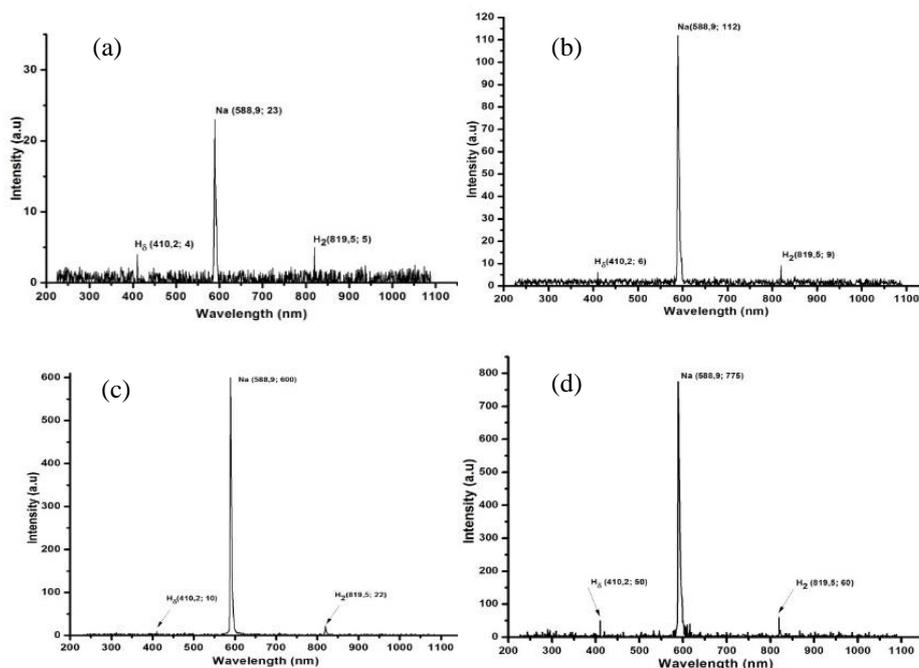


**Figure 8** Diameter of plasma ball incandescent as a function of voltage.

### Emission spectrum and function of the voltage function intensity

**Figure 9** shows the spectrum of emissions in the electrolysis of plasma in water with contamination of NaCl solutions. The emission spectrum at a voltage of 300 V shows similarities with the emission spectrum at 380, 420 and 640 V. The spectral lines at wavelength 410.2 nm are Balmer series of hydrogen atoms which show the transition from  $n = 6$  to  $n = 2$  ( $H_{\delta}$ ). Hydrogen in molecular form ( $H_2$ ) is detected at a wavelength of 819.5 nm. Sodium spectrum lines with a wavelength of 588.9 nm can be seen clearly and appear dominant. Sodium is excited by a mechanism:  $e + Na \rightarrow Na^* + e$ , transition  ${}^2P_{3/2} \rightarrow {}^2S_{1/2}$ ,  ${}^2P_{1/2} \rightarrow {}^2S_{1/2}$ , with an energy threshold for excitation of 2.1 eV [20].

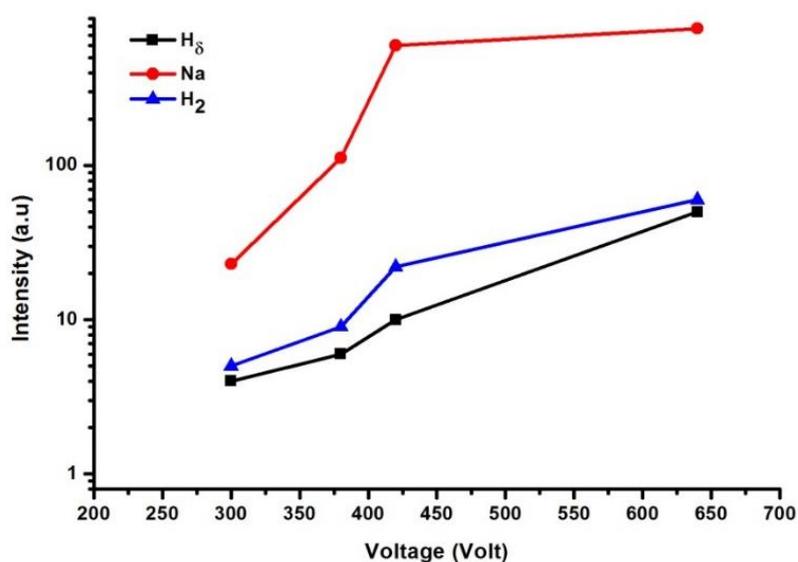
In the emission spectrum of a plasma ball for a voltage of 300 V, the intensity of the hydrogen atom spectrum is very weak at wavelength 410.2 nm with an intensity of 4 AU and a hydrogen molecule at wave 819.5 nm with an intensity of 5 AU. The Sodium spectrum line is visible at a wavelength of 588.9 nm with an intensity of 23 AU.



**Figure 9** Emission spectrum of 0.8 M NaCl plasma electrolysis at voltages; (a) 300 V, (b) 380 V, (c) 420 V, (d) 640 V.

The emission spectrum at 380 V shows a sodium spectrum that is even higher in intensity, which is 112 AU. The hydrogen spectrum line still appears thin with increasing intensity compared to the emission test for a voltage of 300 V, i.e. at a wavelength of 410.2 nm with an intensity of 6 AU and a wavelength of 819.5 nm with an intensity of 9 AU. The emission spectrum for voltage 420 V also shows a trend of increasing intensity. The hydrogen spectrum line appears with an intensity of 10 AU at a wavelength of 410.2 nm and an intensity of 22 AU at a wavelength of 819.5 nm. At a voltage of 640 V, the hydrogen spectral line is seen more clearly with an intensity of 50 AU at a wavelength of 410.2 nm and an intensity of 60 AU at a wavelength of 819.5 nm. In **Figure 10** the intensity of Na, H $\delta$ , and H $_2$  can be seen increasing with increasing voltage applied to the reactor system. These results indicate the production of H $_2$  molecules with plasma electrolysis increases with increasing voltage. An increase in voltage results in an increase in the intensity of the electric field. A strong electric field will accelerate electrons and with sufficiently high energy pounding H $_2$  molecules cause the molecule to dissociate, followed by excitation or ionization. Hydrogen molecules have bonding energy of 436 kJ/mol, meaning that to break the covalent bonds in one mole of hydrogen gas molecules requires 436 kJ of energy. So, the electrons involved in collisions with H $_2$  molecules have enough energy so that H atoms are formed.

Emissions of hydrogen atoms are detected with the intensity that is always below the intensity of H $_2$  molecules because the basic material to produce H atoms requires H $_2$  molecules which are then dissociated. The addition of voltage to the reactor system makes collisions that occur more and more so that the production of H $_2$  molecules and hydrogen atoms increases too.



**Figure 10** Graph of emission intensity as a function of voltage.

#### Full width half maximum (FWHM) of H $\delta$

From the hydrogen atom emission H $\delta$  spectrum that is at wavelength 410.2 nm, full width half maximum (FWHM) analysis is carried out. FWHM is determined by using a Gaussian profile, this is done because the intensity of the spectrum is not too large. The determination of FWHM is intended to obtain information on energetic influences on the broadening of the H $\delta$  transition spectrum. Obtained that there is a tendency for the greater the input voltage the greater the FWHM of the spectrum. Examples of determining FWHM with a Gaussian profile are shown in **Figure 11**. The effect of the input voltage on spectrum widening can be seen in **Figure 12**.

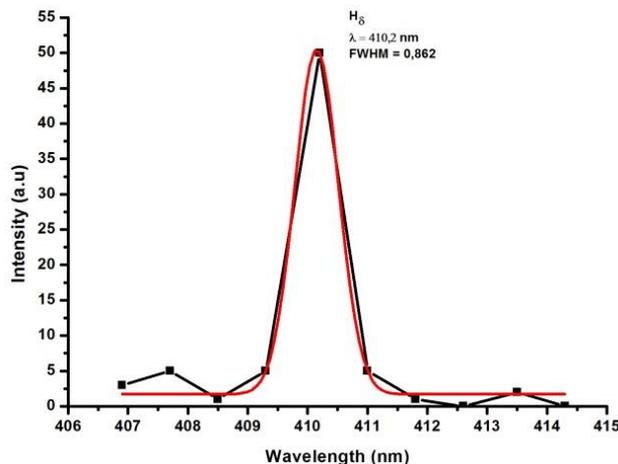


Figure 11 FWHM spectrum of the hydrogen atom.

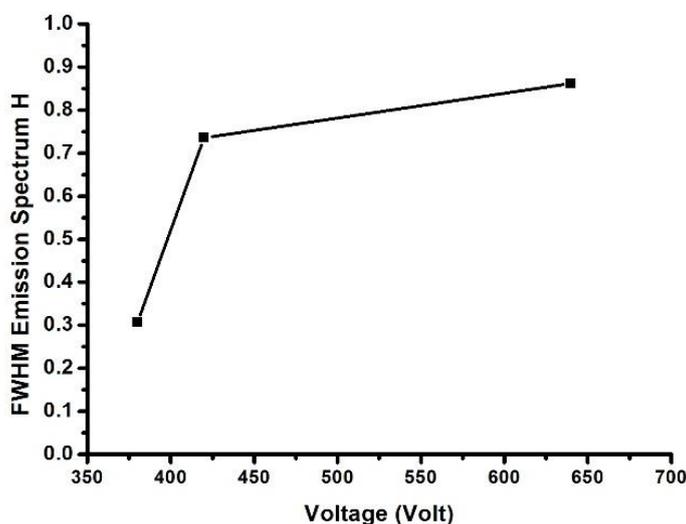


Figure 12 Graph of FWHM of atomic emission H<sub>δ</sub> as a function of applied voltage.

The results and previous discussions found a tendency that is related to 1 another. To draw a scientific conclusion, a discussion is conducted simultaneously on the electrolyte concentration of NaCl of 0.8 M. The current-voltage characteristics (Figure 4) produce several regions of current. When a plasma ball occurs (Figure 7), the electric current rises sharply, and power is calculated using the input voltage multiplied by the current produced ( $P = V I$ ) also surges sharply. The formation of the plasma ball that occurs can be seen in the photos in Figure 7. Changes in plasma ball diameter as an input voltage function are shown in Figure 8. Plasma ball formation with its incandescent light emits areas ranging from infrared to ultra-purple. H<sub>2</sub> spectrum emissions with wavelengths of 819.5 nm and H<sub>δ</sub> with wavelengths of 410.2 nm and spectrum of Na emissions at a wavelength of 588.9 nm.

Of all the results shown, there is a stress area where the ionization process transition from non-ionization to independent ionization. The electrolysis process can also be clearly distinguished by conventional electrolysis (producing anion-cation), electrolysis mixture between conventional and plasma electrolysis, and followed by pure plasma electrolysis.

Saito *et al.* [9] with wire and semicircular configuration made of platinum mesh, there is also an electrolysis area. According to Saito *et al.*, there is a partial electrolysis area of plasma that is characterized by an active electrode containment tube partially filled by plasma. The next area is the full plasma area, in the treatment without the active electrode enclosure after exceeding 173 volts. Slightly different naming

given in this study is conventional electrolysis area, conventional electrolysis area, and partial plasma and plasma electrolysis area. Of all the results there is a change from non-self-plasma discharge to independent plasma discharge in water at a critical voltage of 480 volts with a current of 0.043 A.

## Conclusions

In this study, a plasma electrolysis reactor with a spiral-rod electrodes configuration can be used to produce hydrogen by detecting the emission of hydrogen atoms and hydrogen molecules. The I-V characteristics in this study have found in 4 regions (**Figure 4**). The first region is the area where conventional electrolysis (A-B) takes place. This region is also known as the ohmic region. The next region is the plasma region formed, which is indicated by a sharp decrease in the current (B-C). the connection at point B is the breakdown voltage, i.e. the voltage at which plasma begins to form. The next area is the gas casing stage which covers the anode causing reduced current (C-D). the connection where the current starts to rise sharply ( $V_D$ ) is called the midpoint voltage. With a large number of electrons, the energy required for excitation is relatively smaller and is continued in the next area (D-E), with plasma flares. Plasma balls observed on the surface of the air and in the air are still dominant due to the NaCl solution contained in the water. Plasma spherical formations with their emission light areas ranging from infrared to ultra-violet. The emission spectrum of  $H_2$  with a wavelength of 819.5 nm and  $H_\delta$  with a wavelength of 410.2 nm and the emission spectrum of Na at a wavelength of 588.9 nm. The emission intensity of Na is still very large, exceeding the emission from  $H_\delta$  atoms and  $H_2$  molecules. All the results obtained regarding the diameter of the “plasma ball”, the expansion of the emission of  $H_\delta$  atoms, and the emission intensity of Na,  $H_2$  and  $H_\delta$  are strongly affected by the applied voltage. The method used to produce hydrogen in this research only gives an indication that  $H_2O$  decomposition occurs. This research needs to be continued with more precise measurement systems and more robust cells.

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