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Generation of High Frequency Pin-hole Discharge in Water Solutions

Abstract: This paper presents results on electric discharge generation by high frequency high voltage (15–100 kHz) in NaCl solutions with different initial conductivity (100–1300 μS cm⁻¹), and compares them with DC discharge in the same electrode configuration. A batch plasma reactor in the pin-hole configuration contained a ceramic dielectric barrier separating two planar stainless steel electrodes; barrier thickness of 0.6 mm and pin-hole diameter of 0.6 mm was used. Lissajous charts were evaluated from electric measurements for different discharge phases (electrolysis, bubble formation and discharge regular operation). Breakdown moments for different solution conductivities were determined from discharge power evaluation as a function of applied frequency. Breakdown voltage amplitude was decreased by the increasing conductivity in both regimes while frequency and current decreased. Changes of physical parameters (temperature, solution conductivity and pH) as well as production of hydrogen peroxide at different solution conductivities were compared. Solution conductivity was increased in both discharge regimes and with the initial conductivity value. Solution temperature was increased by the discharge in both regimes and with the increasing initial conductivity, too. Solution pH dropped to acidic conditions when HF or DC positive regime was applied while it was enhanced by DC negative regime.

Keywords: discharges in liquids, electric measurements, breakdown parameters, solution conductivity, hydrogen peroxide generation

1 Introduction

Electrical discharges generated directly in liquids or in the direct interaction with the liquid phase are one of the hot topics of the contemporary plasma research. These systems can be applied for example in water treatment [1,2], sterilization [3], and surface treatment of various temperature sensitive materials [4] as well as for nanoparticle production [5]. The plasma generated directly in water solutions can be obtained by various electrode configurations, and it can be supplied by many kinds of applied voltages from DC up to MHz frequencies using pulsed as well as non-pulsed regimes.

The pin-hole discharge is one of several suitable configurations allowing electrical discharge generation in liquids. We use the term of the “pin-hole” discharge is the general expression for the discharge created in a small orifice in the dielectric barrier separating two electrode parts of the plasma reactor. According to the orifice dimensions, we further use the term “diaphragm” (for the ratio of the barrier thickness to the orifice diameter approximately equal to 1) or capillary (the ratio substantially higher than 1) studied for example in [6-8]. Our paper is focused on results obtained in the diaphragm configuration however our further experiments are dealing with the transition between the diaphragm and capillary configuration and these results will be submitted for publication in another paper soon. Typically, DC or low frequency (50 Hz) high voltage sources are used in a pulsed or continuous regime [9]. Two of initiated processes, electrolysis and Joule heating, induce a decrease of energy efficiency of such systems [10]. Moreover, the applicability of such power supplies seems to be rather dangerous for handy operating devices. Thus, we have focused on the pin-hole discharge generation using an audio frequency power supply (from 1 to tens of kHz).

For plasma diagnostics of the pin-hole discharge, electrical measurements of the main electric values (discharge current, voltage, power and frequency) as well as other important additional parameters (light...
emission and sound records) are commonly employed in order to determine particular processes during the discharge ignition in water solution. Increasing the input energy into the system, following dominant phases can be consequently distinguished: electrolytic reactions, liquid evaporation into bubbles, random discharge breakdown in these bubbles accompanying by light flashes and finally, regular discharge operation with intensive light emission as well as bubble formation [11]. Besides these macroscopic phenomena, other physical and chemical processes are also initiated by the discharge generation in water solutions. Due to the ionisation and dissociation of water molecules, various reactive species such as radicals, ions, excited particles and molecules with a high oxidation potential are formed [12]. Among them, hydroxyl, hydrogen and oxygen radicals as well as hydrogen peroxide belong to the most studied particles because of their usability in further plasma applications. Physical processes are represented by UV radiation, intensive electric field and formation of shockwaves in highly conductive solutions [13]. Simultaneous action of these processes can substantially affect solution physical properties which are important to adjust properly for an optimal discharge operation and its utilisation in further applications. To control the main solution parameters (conductivity, pH, temperature, etc.) effectively, it is necessary to have an idea about their evaluation during the discharge operation. Therefore, we monitored these parameters continuously during the discharge operation and with respect to particular discharge configurations and conditions, too.

2 Experimental Procedure

A batch plasma reactor (Fig. 1) with total volume of 100 ml was used in experiments observing discharge breakdown. It was divided by a dielectric barrier into two electrode parts. The dielectric barrier was made of 0.6 mm thick Shapal-M™ ceramics with one central pin-hole with the diameter of 0.6 mm (details in Fig. 1). One planar electrode (48x40 mm²) made of stainless steel was installed in each part of the reactor in the distance of 45 mm from the dielectric barrier. Alternatively, a larger batch discharge reactor with total volume of 4.5 L was used for experiments focused on observation of physical and chemical processes during longer discharge operation. The same pin-hole configuration using the dielectric barrier of the same parameters was used in this device. Additionally, a mixing system was installed in the larger reactor in order to keep the bulk solution homogeneous during the whole experiment. Moreover, the pin-hole in the dielectric barrier was flushed at least ten times by the used electrolyte solution before each experiment in order to ensure the same conditions and obtain reproducible data.

A specially designed power supply based on a resonance circuit gave sinusoidal high voltage up to 5 kV. Its energy was set using frequency in the range of 15–100 kHz. The supplied energy was calculated using Lissajous charts [14] which were evaluated from electric measurements for different discharge phases (electrolysis, bubble formation and discharge regular operation). Subsequently, discharge power was estimated and plotted.
as a function of applied frequency in order to determine breakdown moments for particular discharge conditions. For comparative measurements, a DC power supply was used giving non-pulsing high voltage up to 5 kV. A four-channel oscilloscope Tektronix TDS 2024B was used for instantaneous records of voltage, current, light and sound before, during and after the discharge ignition. High voltage was measured by a Tektronix P6015A high voltage probe with the attenuation 1:1000, current was determined from voltage measured on a 4.1 µF capacitor. A simplified scheme of the plasma reactor and its integration into the circuit with the power supply and the oscilloscope is given by Fig. 1.

NaCl solutions with initial conductivity in the range from 100 to 1300 µS cm⁻¹ were used as a conductive liquid in the presented study. Physical parameters (conductivity, pH and temperature) of experimental solutions were monitored before, during and after the discharge operation. Solution conductivity was measured by a portable conductometer GRYF 107L. Temperature and pH were recorded by a portable pH-meter WTW Inolab. Detection of hydrogen peroxide produced by the discharge in water solutions was realised by a colorimetric method using a titanium reagent. This analysis was based on a selective reaction of hydrogen peroxide with the reagent forming a yellow complex of pertitanic acid which absorbance at 407 nm was directly proportional to H₂O₂ concentration in the complex [15]. Complex absorption was detected by a standard UV–VIS spectrometer Unicam Helios Alfa.

### 3 Results and Discussion

Instantaneous values of current and voltage were recorded in several steps with the increasing applied voltage. Records of sound and emitted light were also observed in order to estimate bubble formation and discharge breakdown in the plasma reactor. Subsequently, Lissajous charts [14] were constructed from data combining high voltage amplitude and voltage measured on the capacitor (4.1 µF). Typical examples of Lissajous charts for four different phases during the pin-hole discharge ignition are presented in Fig. 2.

Both high voltage amplitude as well as voltage measured on the capacitor increased with the increasing applied voltage. During the phase when only electrolytic reactions proceed in the reactor (no sound or light records were observed), high voltage between electrodes oscillated up to 300 V. When the applied voltage amplitude was increased up to 800 V, the first bubbles appeared in the pin-hole vicinity due to the solution overheating and

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**Figure 2:** Lissajous charts for four processes occurring in the pin-hole reactor with the increasing applied voltage: a) electrolysis (frequency of 50.5 kHz), b) bubble formation (30.2 kHz), c) discharge operation (22.0 kHz), d) maximal discharge operation (20.8 kHz); NaCl solution (500 µS cm⁻¹), dielectric barrier thickness of 0.6 mm, pin-hole diameter of 0.6 mm.
evaporation. Discharge breakdown in the pin-hole was observable when high voltage reached amplitudes over 1000 V. Maximal source operation gave the amplitude around 4000 V. Simultaneously with the increasing applied voltage, discharge frequency decreased into the audio range. In order to evaluate discharge power, an integral area \((A)\) of the curve in each Lissajous chart was calculated. Subsequently, discharge power was calculated according to the following equation:

\[
P = A \cdot C \cdot f
\]

where \(A\) is the integral area of the curve (V\(^2\)), \(C\) is capacitance of the capacitor (4.1 µF) and \(f\) is adjusted frequency (Hz).

Average values obtained from ten consequent periods were used in the calculation because of random discharge operation and induced electric instabilities in the system.

The values of discharge power were evaluated as a function of the high voltage amplitude (Fig. 3, left) or frequency (Fig. 3, right). Presented data were obtained in NaCl solution (conductivity of 500 µS cm\(^{-1}\)). The left part of the Fig. 3 represents an example of the discharge generation characteristic. The initial part (I) shows a very slow increase of energy dissipated in the system on applied voltage, and it corresponds to ohmic character of the interelectrode system. This means that there is no discharge created (no light emitted from the discharge area is recorded), and only electrolytic reactions can proceed in the system. Also sound records are on a minimal level. The second part (II) is characterized by significantly higher power consumption which corresponds to the bubble formation. Presence of bubbles was also confirmed visually, and significant sound generation caused by their cavitation was recorded. However, still no light emission appears during this part which indicates no discharge creation. Finally, the third part (III) represents the discharge operation accompanied by intensive light emission increasing with the increasing applied high voltage. A chaotic character of experimental points reflects the fact that the discharge is not operating continuously. It is generated inside the bubbles that cavitate and thus the power consumption is strongly dependent on the bubbles dimensions, mainly. Transitions between these determined parts expressed by the applied high voltage magnitude are as follows: 500 V for the start of bubble formation and 1600 V for the discharge breakdown. These transition ranges are in a good agreement with recently published works focused on DC diaphragm [16,17] or DC capillary discharges [18]. Dependence of discharge power on applied frequency is shown in Fig. 3, right. Energy consumption more or less exponentially decreases with the increasing frequency in the audio and higher frequency range (20–72 kHz) from 44 to less than 1 W.

As it was already published in our previous papers [11,19,20], generation of electric discharge in water solutions by the application of DC non-pulsing high voltage is essentially dependent on solution conductivity. Therefore, this paper presents results concerning breakdown conditions for high frequency discharge generation as a function of solution conductivity as well. Based on current-voltage characteristics (Fig. 3, left) obtained in NaCl solutions with initial conductivities in the range from 100 to 900 µS cm\(^{-1}\), breakpoints representing the start of bubble formation and breakdown moment of the discharge itself were identified. Each breakpoint was characterised by the specific value of high voltage amplitude, discharge power and applied frequency. All these parameters are plotted as a function of initial
solution conductivity for both processes (bubble formation and discharge breakdown) in Figs. 4 and 5.

In the case of the applied high voltage amplitude and its relation to the breakdown moment (Fig. 4, left), it is decreased by the enhanced solution conductivity and thus the discharge is ignited by substantially lower voltage when higher conductivity is used. Particularly, high voltage amplitude of almost 3000 V is needed for the discharge breakdown when solution conductivity of 100 μS cm⁻¹ is used. On the other hand, nine times higher conductivity (900 μS cm⁻¹) decreased the breakdown high voltage amplitude to 1000 V, only. A similar decreasing tendency caused by the increased conductivity was also achieved for the values of discharge power at the breakdown moment (Fig. 4, right). Its values drop from 17 to 10 W in the studied range of conductivities. However, breakpoint values of both parameters (high voltage amplitude and discharge power) representing appearance of the first bubbles in the system seem to be independent on solution conductivity, except the very low conductivity of 100 μS cm⁻¹. This higher energetic requirement is probably caused by higher resistance of the system due to lower amount of charged particles (ions) in the solution. The mean value of the high voltage amplitude needed for the bubble formation is around 500 V while energy up to 5 W should be supplied into the reactor.

Contrary to the previously analysed parameters, the situation with breakdown frequency dependence on solution conductivity is opposite (Fig. 5). Values of applied high frequency representing the discharge breakdown slightly increase with the increasing solution conductivity. Particularly, breakdown frequency increases from 22 to 27 kHz in the studied range of conductivities from 100 to 900 μS cm⁻¹. On the other hand, bubble formation starts at higher applied frequency and it is increased by the increasing conductivity. However, this dependence is not clearly linear as in the case previously analysed parameters.

The comparison of the breakdown conditions in different high voltage regimes, i.e. when high frequency or DC non-pulsing HV source was used for the discharge generation, gives slightly different results. The dependence of minimal voltage and current required for the DC discharge breakdown in NaCl solutions with conductivity varied from 300 to 1300 μS cm⁻¹ and is shown in Fig. 6. Accordingly to the high frequency discharge, breakdown voltage is also substantially decreasing with the enhanced solution conductivity. Particularly, the voltage magnitude drops from 1350 V to less than 900 V in the studied

![Figure 4](image1.png)  ![Figure 5](image2.png)

**Figure 4:** Minimal applied high voltage amplitude (left) and discharge power (right) of bubble formation and discharge breakdown as a function of solution conductivity; NaCl solution, dielectric barrier thickness of 0.6 mm, pin-hole diameter of 0.6 mm.

**Figure 5:** Minimal applied frequency of bubble formation and discharge breakdown as a function of solution conductivity; NaCl solution, dielectric barrier thickness of 0.6 mm, pin-hole diameter of 0.6 mm.
conductivity range. On the other hand, breakdown current is increased from 40 to 110 mA in the same range due to the enhanced amount of charged particles supplied into the solution by higher concentration of the dissolved electrolyte. However, total discharge power calculated by a simple multiplication of both parameters (voltage and current) in the DC regime is more or less constant. But it gives two values within the studied conductivity range: power of about 60 W is needed for the discharge breakdown in solutions with conductivity from 300 to 800 µS cm⁻¹ while higher power of about 85 W is required for higher conductivities from 800 to 1300 µS cm⁻¹.

Comparing to the high frequency regime (Fig. 4, right), lower discharge power around 15 W is needed and it is decreasing with the increased solutions conductivity. Our results dealing with the breakdown conditions in the DC non-pulsing regime and their dependence on solution properties were published in [11,19,20]. Although the breakdown parameters are substantially influenced by electrolyte concentration in the solution (i.e. by solution conductivity), they seem to be independent on the electrolyte kind itself [9,19] (the comparison is given for NaCl, NaNO₃, Na₂SO₄, and Na₃PO₄ electrolyte solutions). On the other hand, the process of bubble formation in the DC regime is affected by both electrolyte kind as well as its concentration [19]. In general, lower voltage is sufficient for the first bubble appearance in the case of smaller electrolyte molecules (NaCl) because their mobility is higher and thus the solution can evaporate into bubbles easily by more intensive Joule heating [19]. Moreover, higher concentration of electrolyte and its ions in the solution also decreases voltage needed for the bubble formation due to higher current density in the system [20]. Due to this fact, NaCl solutions were selected for most of experiments in order to observe intensive bubble formation and its dependence on experimental conditions.

As it had been mentioned above, generation of electric discharge in water solutions initiates various physical and chemical processes which influence physical properties of the solution such as its conductivity, pH or temperature. Due to the formation of reactive species such as ions and radicals by the dissociation and ionisation of water molecules, the total amount of charged particles is changing during the discharge operation. Therefore, solution conductivity is not stable due to changing concentration of these particles and their proceeding reactions. Conductivity changes of NaCl solutions after 40 minute discharge operation in the DC non-pulsing and high frequency regimes are demonstrated in the Fig. 7, on the left, with respect to the initial solution conductivity (in the range from 300 to 900 µS cm⁻¹). Due to substantial effect of electrolytic reactions taking place in the DC discharge without mixing solution volumes at the anode and cathode parts of the reactor, final solution conductivity is significantly higher in both electrode parts similarly, and it is more or less linearly dependent on the initial solution conductivity. The same conductivity increase is observed in the HF regime as well. The highest absolute enhancement of about 200 µS is reached in the case of the highest initial conductivity of 900 µS cm⁻¹.

Formation of reactive particles and their subsequent reactions in the solution also affects pH of the solution, i.e. concentration of hydrogen cation. Comparison of final pH reached after 40 minute discharge operation at different voltage regimes is given in Fig. 7 right. Initial pH of used NaCl solutions was 6.0 (marked by the dash line in the graph) and initial conductivity was varied from 300 to 900 µS cm⁻¹. It is evident that there is an opposite effect of the discharge operation on solution pH in both electrode parts when the DC regime with separated electrode parts is applied. Due to electrolytic reactions and formation of hydroxyl anions, higher pH values of about 10−11 are obtained in the cathode part of the reactor. These values are more or less independent on the initial solution conductivity in the studied range. On the other side of the dielectric barrier, the situation is reverse. Due to the increased amount of hydrogen cation and related electrolytic reactions, solution pH drops to strongly acidic conditions with values about 3−4 in the anode part. Although we had expected that the application of the high frequency voltage regime would not affect solution pH because of simultaneous action of antagonistic electrolytic reactions, the final pH values obtained after the HF discharge operation reached acidic conditions. The decrease of pH values is not so dominant as in the case...
of the anode part in the DC regime and final pH reaches values around 5 independently on the initial solution conductivity.

Observation of both solution conductivities as well as pH during the discharge operation is important due to the proper control of processes taking place in the plasma reactor. As it has been discussed above, solution conductivity significantly influences the discharge breakdown and operation itself. On the other hand, solution pH affected mainly chemical reactions initiated by the discharge in various solutions and liquids in general, as it was already proved in [21]. Besides solution conductivity and pH, temperature is also one of the most important physical parameters that should be checked during various plasma treatments. Extremely high temperature in the plasma reactor could lead to lower energetic efficiency, undesirable decomposition of discharge products or destruction of treated materials. It also plays an important role during plasma sterilisations. Therefore, changes of temperature during the discharge operation in the different high voltage regimes were also studied. For these experiments, a larger plasma reactor with volume of 4.5 L was used because this device was further tested for special discharge applications. Results of temperature changes reached after the 40 minute discharge operation in NaCl solutions with different solution conductivities (300–900 µS cm⁻¹) are shown in Fig. 8, left. The initial solution temperature was around 21°C. In the studied range of solution conductivities, temperature is enhanced by the discharge operation more or less linearly with the increasing initial conductivity, but independently on the applied high voltage regime. When the discharge is applied for 40 minutes, the enhancement is up to 10°C for all tested initial conductivities. This means that the final temperature after the 40 minute treatment does not exceed 30°C which is a crucial value for thermal decomposition of hydrogen peroxide. This compound formed by the discharge in water solutions is one of important parameters indicating the discharge effective operation and thus its thermal decomposition should be prevented for the proper study. Comparing both high voltage regimes applied, the DC regime should induce intensive heating of the solution due to higher mean current density in the system while the high frequency regime is more thermally stable. However, the final heating is strongly dependent on the total volume of the treated solution as well. Concerning experiments in the plasma reactor with larger volume (4.5 L), it seems that the heat dissipation of the solution is sufficient to prevent the system against higher overheating in both discharge regimes.

As it has been already mentioned above, formation of hydrogen peroxide belongs to crucial comparative parameters characterising every plasma device based on different configurations of the discharge in water solutions (or liquids containing OH groups). Due to an easy and fast detection of hydrogen peroxide by the colorimetric method using a specific titanium reagent, its evaluation for different discharge conditions is one of the standard chemical analyses serving for wide comparative studies. Thanks to linear production of hydrogen peroxide during the discharge operation, a net production rate of hydrogen peroxide formation \( k_{H_2O_2} \) can be evaluated. Results of hydrogen peroxide formation obtained by the discharge operation in NaCl solutions with different initial conductivity (300–900 µS cm⁻¹) are demonstrated in Fig. 8 right with respect to applied voltage regimes. The highest production rates are achieved in the cathode part of the DC plasma reactor. Formation of hydrogen peroxide is directly proportional to the initial solution conductivity. Contrary to the cathode part, production of hydrogen peroxide in the anode part is almost negligible independently on solution conductivity. This difference in \( \text{H}_2\text{O}_2 \) formation in opposite polarities of the DC regime is caused by different energy dissipation around the pin-hole in the dielectric barrier. More details about hydrogen peroxide formation initiated by the DC non-pulsing discharge at various conditions were published in our several papers [10,22,23]. In case of the high frequency discharge, production rates of hydrogen peroxide are almost the same at lower conductivities up to 500 µS cm⁻¹ as in the cathode part of the DC discharge, but they reached lower values when higher conductivities are used. Unfortunately, obtained data are charged by a relatively high uncertainty due to low values of determined rate coefficients. On the other hand, these results indicates that application of high frequency voltage is more efficient from the viewpoint of the \( \text{H}_2\text{O}_2 \) formation because in the DC discharge, energy applied into the anode part of the reactor is not consumed for this process and thus it is wasted.

4 Conclusions

The paper presents results on the pin-hole discharge generation by high frequency high voltage in water solutions, and compares them with the results obtained in the DC non-pulsing voltage regime. Energetic conditions related to the discharge breakdown are evaluated by Lissajous charts and frequency-voltage characteristics in order to determine breakdown parameters for particular
discharge configurations, especially with respect to solution initial conductivity. Macroscopic phenomena in the plasma reactor when increasing high voltage is applied can be distinguished as three main phases: electrolytic reactions, solution evaporation into bubbles and regular discharge operation (primary ignited in these bubbles) accompanied by intensive light emission. Breakdown moment characterised by minimal values of high voltage amplitude, discharge power and frequency is dependent on solution conductivity. While the first two parameters are decreased by the enhanced conductivity, lower applied frequency is needed when higher conductivity is used. A similar effect of solution conductivity is observed for the first appearance of bubbles in the vicinity of the pin-hole. Comparing the breakdown parameters in the DC non-pulsing regime, the minimal values of voltage are decreased as well as current decreased with the increasing conductivity. However, total discharge power required for the discharge ignition is constant in the studied range of solution conductivities.

Generation of electric discharge in water solutions initiates physical and chemical processes that affect physical properties of the solution (conductivity, pH and temperature) as well as chemical reactions leading to the formation of hydrogen peroxide, for example. Monitoring and control of these parameters is crucial for the discharge optimal operation as well as its utilisation in further plasma applications. Solution conductivity is increased
by the discharge operation when both voltage regimes are applied. Moreover, conductivity enhancement is directly proportional to the initial conductivity value. Solution pH is substantially influenced by the discharge operation in both voltage regimes. However, change of pH is opposite in the anode and cathode part of the DC plasma reactor with separated electrode volumes. Due to electrolytic reactions, pH drops to acidic conditions in the anode part and arises to basic conditions in the cathode part. Concerning the high frequency regime, pH also drops to acidic conditions. In all cases, pH changes are more or less independent on initial solution conductivity. Solution temperature is increased by the discharge operation at both voltage regimes. Its change is also increasing with the increasing solution conductivity. The enhancement after the 40 minute discharge operation is similar when DC non-pulsing or high frequency voltage is applied into 4.5 L of the solution. Hydrogen peroxide production is increased by higher initial solution conductivity when high frequency and DC negative voltage is used. Slightly higher net production rates are reached in the DC negative regime in both voltage regimes. Its change is also increasing with the increasing solution conductivity. The enhancement after the 40 minute discharge operation is similar when DC non-pulsing or high frequency voltage is applied into 4.5 L of the solution. Hydrogen peroxide production is increased by higher initial solution conductivity when high frequency and DC negative voltage is used. Slightly higher net production rates are reached in the DC negative regime compared to the high frequency regime. In case of the DC positive regime, hydrogen peroxide formation is almost negligible.

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