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# Contribution to the study of the stainless steel electrodes dissolution under bipolar pulsed polarization

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## Abstract

In systems of biocide treatment of liquid solutions that use pulsed electric fields to inactivate micro-organisms, tanks are made of stainless steel. The application of a voltage involves oxidation/reduction reactions at the interface material/electrolyte, and steel can dissolve; it follows then a change in the chemical composition of the solution near the electrode. In this process, the use of high intensity voltage and short pulses provides better inactivation of bacteria without varying significantly the temperature of the environment. The purpose of this work is to show that the use short pulses allow also a significant limiting of the dissolution of the tank. For that, we submitted two stainless steel grades to a pulsed bipolar tension and define their character of dissolution in these conditions according to the applied tension and the frequency of pulse.

## 1. Introduction

The biocide and antifouling treatment is a current practice in the chemical, petrochemical and food-processing industries. In the chemical industry, sea water pumping stations are endowed with a chemical system of biocide treatment; it results in this practice the discharge of toxic compounds in the sea which cause a considerable damage to the environment. Economic and ecological concerns and food safety has led to the search for an alternative to the usual techniques. The biocide effect of the pulsed polarization was the object of the study of numerous works dedicated to the use of the electric processes of pasteurization in the food-processing industry [1-7]. The liquid food is exposed to short pulses of high intensity voltage (10 to 80KV/cm) and varying duration from 1 to 10 microseconds. The lethal and the no-thermal effect of pulsed voltage on different microorganisms were demonstrated by several authors [8-13]. Various mechanisms have been proposed to explain the phenomenon of inactivation of biological cells by pulsed polarization [14-17]. Among these mechanisms, dielectric breakdown and electroporation are often implicated in bioelectrical inactivation.

For the first one and from an electrical point of view, the cell membrane can be likened to a capacitance whose walls are separated by a dielectric. The natural transmembrane potential of biological cell is of the order of 50 to 250mV. The exposure of such cells to an external electric field causes changes in the membrane structure. When the applied electric field is very high, it induces an increase in membrane potential and the reduction of the thickness of the membrane. For many cells, the electrical breakdown occurs when the transmembrane potential reaches a critical value of 1V [12] which matches in the case of E.coli to an external electric field of 10KV/cm [18].

For the second mechanism, the electroporation is assigned to the destabilization of the bilayer of the cell membrane under the effect of the electric field. The repulsion phenomena between charged molecules lead to the formation of pores in the cell membrane, thus increasing its permeability [17, 19]. When the external electric field is high, the irreversible process of formation of pores leads to the outward migration of cytoplasmic contents and cell death.

The biocide effect of pulsed polarization has also been observed at very low potential values and the electric field (~1V/cm) [20, 21]. This inactivation was attributed to a mechanical disruption of the bacterial membrane as a result of a fatigue phenomenon induced by bipolar system of polarization.

In the antifouling domain, applying a pulsed electric field to opportunistic species can prevent biofouling in cooling systems that use sea or river water.

The principle is to kill or to put to sleep species before entry and during their stay in the cooling process.

Thus, Schoenbach and Abou-Ghazala [3, 13, 22], have been able to control, by varying the intensity and frequency of the electrical pulse, the immobilization of certain species, in the larval state, such as Hydrozoa or the Brine shrimp in a period ranging from minutes to hours. The temporary immobilization is assumed to be caused by a reversible disruption of the cell membrane.

The pulsed electric field method of high intensity involves the application of a pulsed voltage to a liquid disposed between two electrodes. This electrical energy is discharged into the treatment tank in pulses. Electrical pulses can be of various shapes: square or exponential decay and the regime may be mono or bipolar. The shape of the pulse depends on the electrical system configuration. On the one hand, the importance of inactivation depends on the processes, such as the applied voltage, the shape of the pulse and the duration of treatment [23-25]. The duration of treatment is shorter as the applied voltage is important [9, 26]. In some cases, optimum frequencies for inactivation were observed and assigned to resonance phenomena [13, 20]. The use of a bipolar pulse allows a better inactivation and prevents fouling of the electrodes [23, 27]. On the other hand, the inactivation depends on the species to be inactivated and also its size, its growth phase and its concentration in the solution [27-29], as well as the composition of the solution to be treated, such the pH or conductivity [30-34]. This method would be ideal if it allowed a liquid decontamination with greater efficiency than commonly used methods. For liquid foods, it has an advantage in the heat treatment, since the non-thermal method, can better preserve the olfactory, gustative and nutrition qualities of food [18, 35].

During treatment, the solution is in direct contact with the electrodes which constitute the treating tank. These electrodes are made of stainless steel. Large electric current flows through the solution, electrochemical reactions can occur at the interface of the two electrodes that will become a cathode for the one and anode for the other [4, 36, 37]. These reactions concern the solution to be treated and the electrodes.

In the work on the biocide and antifouling treatment by this process, the electrodes used are generally made by noble and expensive materials [38] and the corrosion behavior is rarely studied [36]. The disadvantage in this procedure is that the electrode reactions can lead to a change in the chemical composition of the liquid in the vicinity of the electrode surface. In aqueous solution, these reactions come from the electrolysis of water and the dissolution of the electrodes. Indeed, it occurs at the anode oxidation respectively, of water and the alloying metals forming the electrode which may be iron, chrome, nickel, etc…, according to the following reactions:

$$2\,H_2O_{(l)} \leftrightarrow O_{2\,(g)} + 4H^+_{(aq)} + 4\,e^- \qquad (1)$$

$$M \leftrightarrow M^{n+} + n\,e^- \qquad (2)$$

When the medium to be treated contains chlorides, the result is the oxidation of chloride ions

$$2Cl^- \leftrightarrow Cl_{2\,(gaz)} + 2\,e^- \qquad (3)$$

The order in which the species will oxidize can be obtained from the table1 [36, 38] representing the oxydo-reduction standard potential. One might expect that $H_2O$ is preferred, because its reduction potential is lower than $Cl^-$ (1.23V and 1.36V respectively), but the chloride reaction, whose potential is close to that of water, is much faster and will occur before the oxidation of water [40].

The reduction of water ($H_2O$) or $H^+$ ions if the solution occurs at the cathode, and the products formed are hydrogen gas and hydroxyl ions ($OH^-$).

$$2\,H_2O_{(l)} + 2\,e^- \leftrightarrow H_{2\,(gaz)} + 2OH^-_{(aq)} \qquad (4)$$

The hydroxyl ions alter the pH of the solution in the vicinity of the electrode. The presence of gas bubbles ($H_2$) in the solution reduces the intensity of the applied voltage [40]. When the medium is aerated, the oxygen reduction can also be considered. According to [41], for $5 < pH < 12$, oxygen reduction obeys the following two step mechanism:

First step:

$$O_2 + 2H_2O + 2e^- \leftrightarrow H_2O_2 \quad 2OH^- \qquad (5)$$

Second step:

$$H_2O_2 + 2\,e^- \leftrightarrow 2\,OH^- \qquad (6)$$

The overall reaction is given by:

$$O_2 + 2H_2O + 4\,e^- \leftrightarrow 4OH^- \qquad (7)$$

Since reaction (5) is generally faster than reaction (6), hydrogen peroxide is a detectable and relatively stable intermediate.

**Table 1:** Reduction half reactions with their standard potentials $E°_{RED}$ in volt with respect to SHE [37, 39].

| $E°_{RED}$ | Reactant Product |
|---|---|
| 1.36 | $Cl_{2(g)} + 2é \leftrightarrow 2Cl^-$ |
| 1.23 | $O_{2(g)} + 4H^+_{(aq)} + 4é \leftrightarrow 2H_2O_{(l)}$ |
| 0.40 | $O_2 + 2H_2O + 4é \leftrightarrow 4OH^-_{(aq)}$ |
| -0,83 | $2H_2O_{(l)} + 2é \leftrightarrow H_{2(g)} + 2OH^-_{(aq)}$ |
| 0.77 | $Fe^{3+}_{(aq)} + é \leftrightarrow Fe^{2+}_{(aq)}$ |
| 0 | $2H^+_{(aq)} + 2é \leftrightarrow H_{2(g)}$ |
| -0.04 | $Fe^{3+}_{(aq)} + 3é \leftrightarrow Fe_{(s)}$ |
| -0.28 | $Ni^{2+}_{(aq)} + 2é \leftrightarrow Ni_{(s)}$ |
| -0.41 | $Cr^{3+}_{(aq)} + é \leftrightarrow Cr^{2+}_{(aq)}$ |
| -0.44 | $Fe^{2+}_{(aq)} + 2é \leftrightarrow Fe_{(s)}$ |
| -0.74 | $Cr^{3+}_{(aq)} + 3é \leftrightarrow Cr_{(s)}$ |
| -0.91 | $Cr^{2+}_{(aq)} + 2é \leftrightarrow Cr_{(s)}$ |
| -2.71 | $Na^+ + é \leftrightarrow Na_{(s)}$ |

The main chemical compound that can be formed at the interface of the cathode is hydrogen peroxide ($H_2O_2$) [4, 20, 41, 42]. This is a biocide compound and its antibacterial effect may improve the process, but it is also toxic; in the case of food processing, contamination by a toxic compound is not acceptable.

Formed compounds can in turn react at the electrode, or give secondary reactions with the electrolyte, chlorine disproportionates in neutral pH water according to reaction (8): [20, 42, 43].

$$Cl_2 + H_2O \leftrightarrow HClO + Cl^- + H^+ \qquad (8)$$

In neutral or slightly acid solution, hypochlorous acid (HClO: pKa = 7.5) is essentially in its undissociated form (based on the potential-pH, said diagram of Pourbaix of chlorine element) [39]. Hypochlorous acid and hydrochloric acid are both toxic and are therefore not desirable [9]. Hypochlorous acid and hydrogen peroxide were respectively dosed in a neutral pH solution with and without chlorides according to the frequency and the pulsed voltage applied (less than 2V) in bipolar regime and time constant. The electrodes used in this case were made of graphite [20]. It was shown in this study that the formed amount of these compounds decreased when the pulse frequency was increased. This amount was of the order of one (1) ppm at an optimal inactivation frequency of 1KHz. In fact, the use of short duration pulses reduces the risk of electrolysis and temperature changes [13]. Chlorine production during the treatment can also be avoided by using non-chloride media such as phosphate or sulphate environments [9]. Regarding the electrodes, this process causes their fouling and particularly their corrosion which shortens their lifetime. Fouling is the formation of a layer of food or other substances resulting from the electrolysis reactions that can be deposited on the electrode surface [10]. The dissolution of the electrodes occurs by the passage in the medium of ions derived from the oxidation of the main elements constituting these electrodes, (see the reaction (2)). It concerns especially the anode in the case of monopolar system [40]. The electrolysis effect in question in this study indicates that the dissolution of the electrode can occur at the anode while at the cathode there is reduction of water and oxygen. For this, we treated two stainless steel grades by a pulsed bipolar polarization of ± 10V and ± 20V with square shape at different frequencies.

## 2. The experimental conditions

### *2.1 Experimental setup*

A two-electrode assembly has been designed especially for these tests. This arrangement allows us to control various experimental parameters necessary for the germs inactivation process; these parameters include frequency, the voltage between the electrodes and the pulse shape. The experimental setup consists of an electronic circuit and an electrolysis cell.

### *2.1.1 Electronic setup*

The currents from laboratory pulse generators are not powerful enough to excite the proposed solution. To overcome this difficulty, the idea was to take a voltage from a DC source at the rhythm of the displayed frequency. For this purpose, a simple electronic setup was performed with the following blocks:

- A pulse generator delivering centered square signals of variable frequency.
- A trigger made of bipolar transistors allowed to realize the inversion and the gap between the trigger pulses

- A block constituted by Triacs assembled in parallel
- The Ecc source constitutes the power supply

The assembly is directed around four blocks:

1. Generator of function: allows delivering square signals of variable amplitude frequency. The signal stemming from the generator is rectified through a conventional diode of recovery 1N 4007.
2. A trigger constituted by 02 complementary transistors BC141 type N and BC 161 type P working in switching mode, the set is polarized by a supply + 5V through two resistors of 150 Ω. Two equal and moved tensions are presented on the collector of T1 and the emitter of T2
3. The power section is made of two triacs whose anode A1 is in the ground; A2 anode is connected to positive terminal of a variable DC supply (0-30V) through two variable potentiometers 1k Ω (270 Ω typical value). In doing so, this value allows to maintain a strongly negative polarity between anodes (A2, A1). Indeed, the positive pulse on the collector of T1 and the tension of anode (A21, A12) is positive and the triac TR1 enters conduction. A21 and A12 are in the ground, while the trigger G2 of the triac TR2 is in zero and A22 and A21 are in the air (open circuit). A Current circulates in the solution between electrodes in the direction (2) towards (1) or negative current. At a negative pulse on the emitter of T2 and the positive tension of anode (A22, A12), the triac TR2 enters conduction and the triac TR1 remains blocked. Then, the current circulating in the solution in the direction (1) towards (2) is established. Thus the solution is requested alternately by a positive and a negative current to the rhythm of the frequency displayed by the pulse generator.
4. Finally, the amplitude is adjustable from the continuous supply Ecc. and the solution is compensated due a capacity of C = 0.01µF

### *2.1.2. Sample preparation and electrolysis cell*

We used two stainless steel grades whose composition is given in Table 2, are cut into bars, then coated in resin and polished with abrasive paper of grain size ranging from 22 to 10 µm. The active sample surface is 3.14 cm$^2$. These samples were then washed with water, then with ethyl alcohol before being dried; they were then introduced into the treatment medium.

**Table 2**: The chemical composition of steel in weight percentage.

| **grade** | **C** | **Si** | **Mn** | **S** | **P** | **Ni** | **Mo** | **Cu** | **Cr** | **Fe** |
|---|---|---|---|---|---|---|---|---|---|---|
| **18-8** | <0.14 | 0.69 | 1.42 | <0.003 | 0.034 | 8.62 | <0.021 | 0.19 | 18.16 | 70.72 |
| **28-20** | 0.099 | 1.56 | 1.95 | <0.003 | 0.019 | 20.18 | 0.287 | 0.12 | 27.93 | 47.85 |

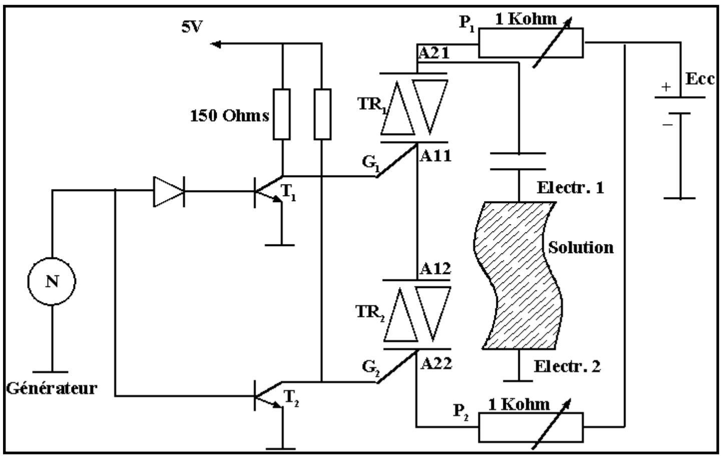

**Figure 1:** The experimental setup.

The cell consists of a beaker containing 200 ml of an aqueous solution containing 32 g of sodium chloride (NaCl) per liter into which plunge two electrodes: the first electrode is made of graphite and has a large surface. The second electrode is made of stainless steel. Finally, the two electrodes were placed one opposite the other

and spaced 1 cm in the solution. The solution was periodically stirred using a magnetic stirrer and the treatment time was 4 hours. The system operated in static mode and at ambient temperature (20 ± 02) ° C.
The experimental setup is shown in figure1**.** In this study we used bipolar pulses square, two voltages ± 10V and ± 20V and frequencies from 1Hz to 10 kHz.

*2.2 Analysis of the solution*
Atomic absorption spectroscopy was used to quantify the metal cations in solution.

## 3. Results and discussions

The system functions as an electrolytic cell. Since the voltage is applied in the form of bipolar pulses, the electrodes alternately act as anode and cathode. The electrode 1 is in graphite and the electrode 2 is made of stainless steel. The main species in the experimental liquid were $Na^+$, $Cl^-$ , $H_2O$ and dissolved oxygen. During the anodic pulse treatment, electrode 1 was the anode and the electrode 2 was the cathode. At the anode, there were two substances that can be oxidised, namely $Cl^-$ and water molecules. As we have stated previously, the oxidation of chloride ions is faster than that of water. At the cathode there were three substances that can be reduced, namely Na+, water molecules and oxygen dissolved. Table1 shows that water molecules reduce easier than sodium ions, the reduction potential are -0.83V and -2.71V, respectively. The ions $Na^+$ are more difficult to reduce and require special operating conditions [40].
During the cathodic pulse treatment, anode and cathode are changing. Electrode 1 is the cathode and 2 is the anode, initially the stainless steel electrodes are probably inert because of their oxide skin, but the experiments showed that parts of the stainless steel alloy have been found in the liquid, which indicates that electrodes do not remain inert. If the oxide skin is damaged, the metal is more favorable to oxidation at the anode than the $Cl^-$ or water. The most favourite reaction from thermodynamic point of view is (2) which is the oxidation of metal constituting the alloy. At the cathode, reduction of water molecules and/or oxygen occurs in reactions (4), (5) and (6).
- *Dissolution of the material*

The main elements constituting the study steel oxidize at the anode. Analysis of the reactional medium reveals the presence of iron, chromium and nickel. After 4 hours of polarization at frequencies above 1 KHz, and for the two voltage applied, the electrode surface remains clean and has few pitting. At low frequencies (≤1KHz), the surface of the electrode is covered with a layer of corrosion products of dark-green color that turns yellow-brown after a few minutes of exposure to air. The addition of a few crystals of potassium ferricyanure in solution during the experiment makes it possible to highlight the presence of $Fe^{2+}$ ions by forming a blue color complex. We also measured the pH of the solution in the immediate vicinity of the electrode; it was between 7 and 8.6 while it remains generally constant and equal to 6.8 in the solution. The $OH^-$ ions present in the immediate vicinity of the electrode were derived from the reactions (4) because the voltage applied is in the water decomposition area, and probably from the reactions (5 and 6**)** during the cathodic pulse, and they locally alkalinized the medium [40]. In neutral or slightly alkaline media, the ferrous iron ($Fe^{2+}$) forms with $OH^-$ ions, the green ferrous hydroxide. This compound is very sensitive to oxygen, and oxidizes to rust brown [44].
Iron, chromium and nickel were each analyzed by atomic absorption spectroscopy, as a total component present in the solution. The results are presented in tables 3 and 4 and are the average of obtained results of several tests.

**Table 3:** Concentration in mg / l of elements analyzed in function of the frequency after treatment for 4 h at ± 10V.

| Frequencies In Hz | Steel 18-8 | | | Steel 28-20 | | | Current [mA] |
|---|---|---|---|---|---|---|---|
| | [Fe] | [Cr] | [Ni] | [Fe] | [Cr] | [Ni] | |
| 1 | 1.26 | 0.62 | 10.43 | 0.91 | 0.47 | 5.65 | 28 |
| 10 | 1.19 | 0.60 | 8.98 | 0.76 | 0.30 | 4.84 | 28 |
| 100 | 1.00 | 0.52 | 7.66 | 0.61 | 0.26 | 4.21 | 28 |
| 1000 | 0.72 | 0.35 | 5.59 | 0.30 | 0.14 | 2.40 | 28.6 |
| 10000 | 0.57 | 0.28 | 5.40 | 0.24 | 0.12 | 2.01 | 28.6 |

**Table 4:** Concentration in mg /l of elements analyzed in function of the frequency after treatment for 4 h at ± 20V.

| Frequencies in Hz | Steel 18-8 | | | Steel 28-20 | | | Current [mA] |
|---|---|---|---|---|---|---|---|
| | [Fe] | [Cr] | [Ni] | [Fe] | [Cr] | [Ni] | |
| 1 | 2.18 | 0.99 | 13.53 | 0.97 | 0.62 | 8.60 | 29 |
| 10 | 1.9 | 0.90 | 12.23 | 0.92 | 0.51 | 7.66 | 29.6 |
| 100 | 1.82 | 0.72 | 12.10 | 0.88 | 0.47 | 6.71 | 30 |
| 1000 | 1.12 | 0.40 | 8.01 | 0.54 | 0.21 | 3.36 | 31.6 |
| 10000 | 0.94 | 0.23 | 6.91 | 0.49 | 0.12 | 2.95 | 32 |

The figures 2 (a, b, c, d, e, f) show that the dissolution of the main alloying elements is a function of the voltage and of the frequency applied. For an applied constant voltage, the steel dissolution decreases as frequency increases. However, the amount of nickel found in the solution was relatively more important than other elements.

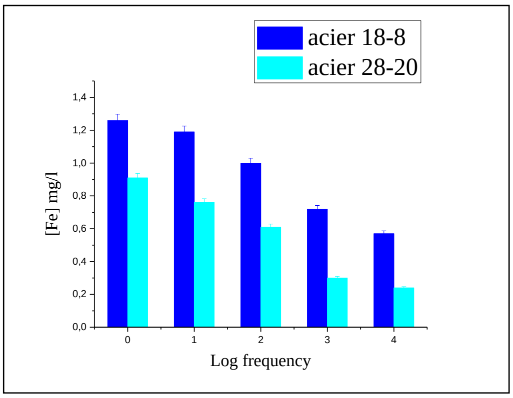

**Figure 2 (a):** Dissolution of iron in chloride medium at ±10V

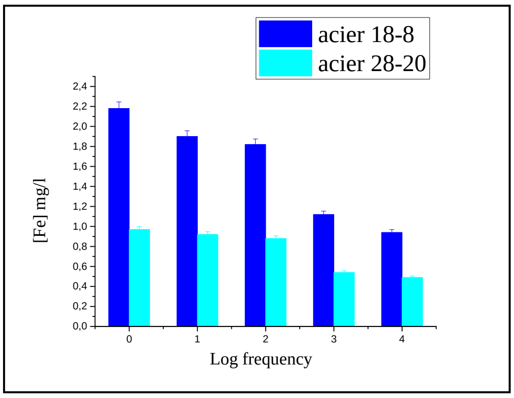

**Figure 2 (b):** Dissolution of iron in chloride medium at ±20V

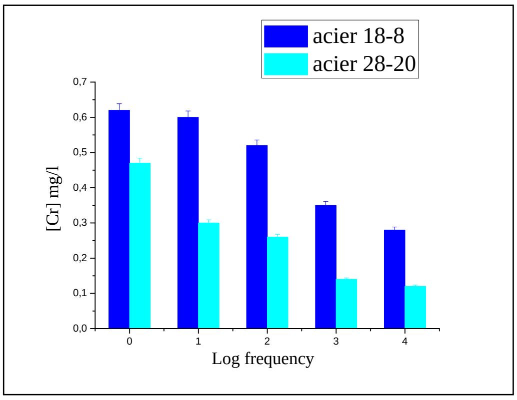

**Figure 2 (c):** Dissolution of chromium in chloride medium at ±10V

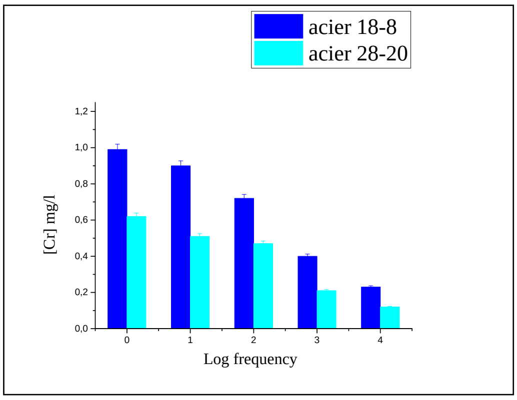

**Figure 2 (d):** Dissolution of chromium in chloride medium at ±20V

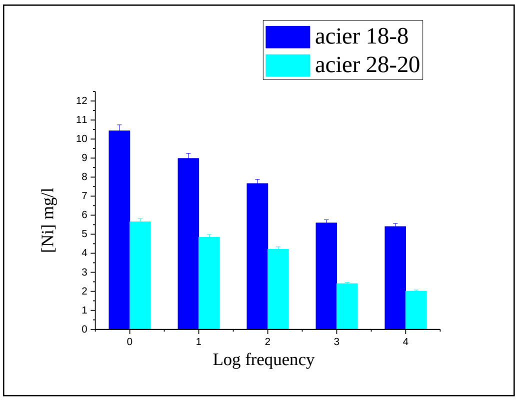

**Figure 2 (e):** Dissolution of nickel in chloride medium at ±10V

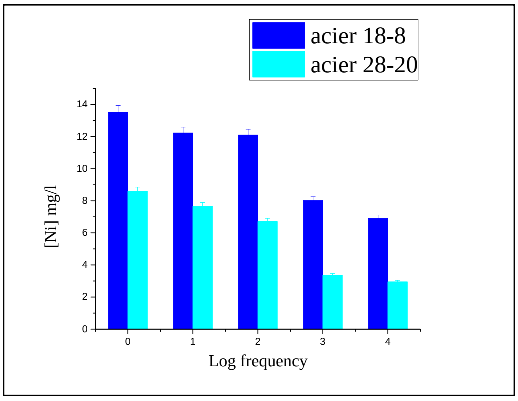

**Figure 2 (f):** Dissolution of nickel in chloride medium at ±20V

In the methods of biocide liquids treatment and in the food sector in particular, the applied voltages are of the order of 100 kV and the treatment time is very short, of the order of some microseconds. The use of short pulses is necessary because the biocide effect occurs at high frequency as we mentioned in the introduction; it is also important for preventing the temperature variation of the solution [8,9,13,43]. At low voltages, the effectiveness of the treatment increases with the time of contact; in these conditions, the electrical signal is low and it is effective only on medium frequencies, which are in the range of 1kHz [20, 21].

Thus, J. Moran and al.[36] have shown that the material constituting the electrode dissolves more significantly at frequencies below 1 kHz for voltages of 15V and 150V.

The work on the oxidation-reduction reactions that may occur at the interface of the electrodes, and the corrosion behavior of the material forming the treatment tank in the conditions of pulsed voltages used, are not sufficient to define operating conditions as they would reduce the dissolution of the tank and prevent the change by electrolysis of the reaction mixture [4, 20, 36, 37].

## Conclusion

The behavior of the stainless steel electrodes subjected to pulsed voltages of bipolar system was determined by the change in their dissolution as a function of the voltage intensity and frequency.

The results obtained by analyzing the solution allowed us to observe, for both imposed tensions and for both steel grades, a net decrease in the dissolution of the main alloying elements when we used high frequencies.

Further studies should be undertaken to define the acceptable limits amounts of compounds resulting from the dissolution of electrodes and electrolysis reactions in products treated by the method of pulsed voltages.

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