



Metal release from stainless steel electrodes of a PEF treatment chamber: Effects of electrical parameters and food composition



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ARTICLE INFO

Article history:

Received 12 March 2013

Accepted 10 October 2013

Editor Proof Receive Date 8 November 2013

Keywords:

Pulsed electric fields (PEF)

Electrode corrosion

Metal release

Electrical parameters

Product composition

ABSTRACT

The effects of electrical parameters (field strength E , total specific energy input W_T and pulse frequency) and product composition on the release of the main metallic elements (Fe, Cr, Ni and Mn) of stainless steel (type 316L) electrodes of a continuous flow parallel plate PEF chamber into the treatment medium were investigated. Experiments were carried out by subjecting two different buffer solutions (McIlvaine and Trizma-HCl) with the same values of pH (7) and electrical conductivity ($\sigma = 2$ mS/cm) to PEF treatments (mono-polar exponential decay pulses, lasting $3.1 \mu\text{s}$) at different intensities ($E = 12\text{--}21\text{--}31$ kV/cm, $W_T = 20\text{--}60\text{--}100$ J/mL) and flow rates ($2\text{--}3\text{--}4$ L/h). The results showed that, for each field strength applied, the concentration of metallic elements increased upon increasing the total specific energy input. At constant total energy input, it was noticed that the metal concentration decreased upon increasing the field strength applied. These results were mainly attributed to the key role played by the pulse frequency in the charging process of the double layer capacitors at the electrode–solution interface. Moreover, it was shown that the amount of metal released from the electrodes markedly depended on the presence of halides in the composition of the processed product.

Industrial relevance: Electrochemical reactions at the electrode solution interfaces are unavoidable when typical conditions for PEF processing are applied. For the acceptability of PEF processing as a non-thermal method for liquid food pasteurization, the occurrence of these reactions should be avoided or minimized since they may determine undesired phenomenon of contamination of the food product, electrode-fouling and electrode corrosion. In this paper, electrode corrosion was studied, for the first time, in a continuous flow parallel plate PEF treatment chamber. The present investigation contributes to clarifying the effects of some electrical parameters and food composition on electrode corrosion or release of electrode materials under real PEF treatment conditions.

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1. Introduction

Pulsed electric fields (PEF) are a non-thermal technology for the cold pasteurization of pumpable food products. The PEF process involves the application of a high intensity electric field ($10\text{--}50$ kV/cm) as a train of short duration pulses (of the order of microseconds) to a liquid food which is placed between or flowing through two electrodes of the treatment chamber (Pataro, Senatore, Donsi, & Ferrari, 2011).

The electroporation phenomenon, which drastically increases the permeabilization of the cell membrane, has been universally recognized as the main cause determining the microbial inactivation. In several studies it was demonstrated that, PEF allows to achieve a sufficient reduction of the initial microbial load in liquid food pasteurization of different products with minimal losses of nutrients and flavor (Aronsson, Lindgren, Johansson, & Ronner, 2001; Barsotti & Cheftel, 1999; Mosqueda-Melgar, Elez-Martinez, Raybaudi-Massilia, & Martín-Belloso, 2008; Sale & Hamilton, 1967a,b). Despite the extensive

knowledge in the field of food preservation by PEF, the definition of optimal treatment conditions, electrode geometry and electrode material able to avoid or minimize the occurrence of undesirable electrochemical reactions at the electrode surface is still a matter of concern.

In fact, from an electrochemical viewpoint, a PEF treatment chamber which consists of two metal electrodes (typically made of stainless steel) placed in direct contact with an electrolytic solution and electrically connected to a high voltage pulse generator, acts as an electrolytic cell (Roodenburg, Morren, Berg, & de Haan, 2005a). As a result, the passage of current through the PEF chamber leads to the formation of a charged double layer at each electrode–solution interface which behaves as a capacitor (Morren, Roodenburg, & de Haan, 2003).

During the charging process of the double layer capacitors, as long as the voltage across them remains smaller than a certain threshold value typical of the reaction potential of the electrode's material (~ 1 V), the current flow would be purely capacitive, avoiding oxidative and reductive reactions at the electrode–solution interfaces. Once the voltage across the double layer overcomes the threshold voltage, electrochemical reactions will occur leading to undesired contamination phenomenon of the food

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product, as well as the electrode-fouling and electrode corrosion (Bushnell, Clark, Dunn, & Lloyd, 1995; Góngora-Nieto, Sepúlveda, Pedrow, Barbosa-Cánovas, & Swanson, 2002; Morren et al., 2003; Roodenburg et al., 2005a).

The effects of electrochemical reactions in PEF food processing systems have been recognized and discussed by several research groups even though most of the papers are mainly descriptive. In addition, most of the results published so far are dealing with the problems of the production of toxic compounds, such as H₂O₂ and chlorine, as well as the formation of a film of food particles at the electrode surfaces (electrode-fouling), due to the electrophoretic process, which can cause local electric field distortion and arcing (Bushnell et al., 1995; Hulshager & Niemann, 1980; Jayaram, 2000; Sale & Hamilton, 1967a,b; Toepfl, Heinz, & Knorr, 2007).

As far as the problem of the electrode corrosion, which is the main topic of this contribution, it is related to the dissolution of the anode material due to the oxidation of the metal of the electrode at the anode–solution interface and to the deposition of part of the dissolved metals on the surface of the cathode due to reduction reactions. For these reasons, electrode corrosion is undesirable and must be minimized from the viewpoint of safety and quality of food, fouling and electrode lifetime (Evrendilek, Li, Dantzer, & Zhang, 2004; Morren et al., 2003; Roodenburg, Morren, Berg, & de Haan, 2005b; Roodenburg et al., 2005a; Saulis, Rodaitė-Riševičienė, & Snitka, 2007).

A method to avoid or reduce electrochemical reactions and the fouling of electrodes in a PEF processing system was patented by Bushnell et al. (1995). Based only on a theoretical analysis, the authors suggested a method to both prevent the double layer capacitor from charging to a potential greater than the reaction potential as well as avoid the cumulative build-up of charges that occur, by removing all the residual charge from one electrode during the discharge period, so that a “zero net charge” is delivered.

On the basis of the principle of avoiding full charging of the double layer capacitors, other authors found that the extent of the electrochemical reactions may be limited by either using electrode materials featuring higher resistance to electrochemical reactions such as titanium and platinized-titanium (Samaranayake & Sastry, 2005), or by using bipolar pulses (Kotnik, Miklavčič, & Mir, 2001).

However, the first attempt to study the release of electrode material during PEF treatment was presented by Morren et al. (2003). The authors exposed stainless steel electrodes in contact with an aqueous solution of NaCl to sinusoidal waveforms with a maximum electric field of 0.15 kV/cm and a maximum current of 480 mA, conditions which were far below those typical of PEF treatments. This notwithstanding, the results have a certain relevance showing that at each voltage level, different elements of the stainless steel electrodes appeared in the liquid below a certain critical frequency (and above a certain pulse width). Lowering the frequency at the same peak–peak voltage leads to an increase in charge and thus an increase in voltage at the double layer.

In spite of these preliminary studies, an extremely limited number of data is available on electrode corrosion in real PEF treatment conditions.

With respect to the safety and quality of food products, it was found that stainless steel electrodes of a co-field chamber produced significant release of Fe, Cr, Zn, and Mn in the PEF-treated beer samples and caused a significant loss in flavor acceptance and mouth feeling of the products (Evrendilek et al., 2004). Moreover, when orange juice was treated with PEF treatment in similar PEF chamber configuration, the metal concentrations detected did not exceed the limit set in the European legislation for fruit juices and drinking water for human consumption (Roodenburg et al., 2005b).

Furthermore, corrosion can cause serious damages to the electrodes, whose surface roughness can increase as a consequence of the metal release or deposition (Saulis et al., 2007). This, in turn, can cause local electric field distortion and arcing drastically limiting the lifetime of the electrodes to few hours (Roodenburg et al., 2005b; Saulis et al., 2007).

The extent of all the above undesired effects related to the electrode reactions depends on many factors such as chamber design (Master, Schuten, & Mastwijk, 2007) and electrode material (Góngora-Nieto et al., 2002; Kotnik et al., 2001; Samaranayake & Sastry, 2005), electrical parameters such as pulse shape (Roodenburg et al., 2005a), peak voltage, total specific energy input, polarity, and pulse duration (Gad & Jayaram, 2011, 2012a; Kotnik et al., 2001; Moonesan, George, Aucoin, & Jayaram, 2012), as well as the composition and chemical-physical properties of the treated medium (Gad & Jayaram, 2011, 2012b; Moonesan et al., 2012; Roodenburg et al., 2005b).

However, due to the highly limited literature data as well as the large number of parameters relevant to the electrochemical phenomenon during PEF treatment, and the wide range of experimental conditions utilized and differences in the equipment adopted from the several research groups, no clear relationship between the release of electrode components and the operating conditions has been derived up to now.

This paper, presents the results of an extended study of the electrode corrosion in a continuous flow parallel plate PEF treatment chamber. The effects of electrical parameters (E, W_T, pulse frequency) and product composition on the metal release from the stainless steel electrodes in the treatment medium were investigated in experimental conditions commonly adopted in PEF processing microbial inactivation.

2. Materials and methods

2.1. PEF treatment system

PEF experiments were carried out in a laboratory scale with a continuous flow unit of their own construction previously described by Pataro, Ferrentino, Ricciardi, and Ferrari (2010) and Pataro et al. (2011).

Briefly, the system consisted of a high voltage pulse generator able to deliver short mono-polar exponential decay electric pulses to a PEF treatment chamber through which the liquid product was pumped. The voltage and frequency of the pulses were adjustable and the device was capable of generating up to 20 kV, 5kA and 400 Hz pulses with a pulse width depending on the resistance of the treatment chamber load, and hence on the electrical conductivity of the processed liquid.

The treatment mediums were processed in a continuous flow self-designed treatment chamber, equipped with two polished parallel flat stainless steel electrodes (type AISI 316L) with an effective area (A) of 2.6 cm² and kept at a distance (*d*) of 0.25 cm with a Teflon insulator.

A peristaltic pump equipped with a food grade Tygon tube was used to pump the liquid product through the system at controlled flow rate in the range from 0.5 to 10l/h. A stainless steel (type AISI 316L) coiled tube (4 mm diameter, 1.3 m length) submerged into a circulating water heating bath made it possible to control the product temperature before entering the treatment chamber. Two T-thermocouples were used to measure the product temperature at the inlet and outlet of the PEF chamber. A high voltage probe and a Rogowsky coil connected to a 300 MHz digital oscilloscope measured voltage and current signals at the treatment chamber. A PC connected via GPIB made possible data acquisition and process control, utilizing an interactive program based on Labview 6i (National Instruments™).

The typical output waveforms (voltage, current and electric field) of the generator for a pulse width of 3.1 μs are shown in Fig. 1.

The maximum electric field intensity (E, kV/cm) was evaluated as the peak voltage divided by the inter-electrode gap. The pulse width, τ, defined as the time needed to decrease the voltage to 37% of its peak value, was 3.1 ± 0.2 μs for an electrical conductivity of the treatment medium of 2 mS/cm. The specific energy input per pulse (W, J/pulse) was calculated according to Eq. (1):

$$W = \int_0^{\infty} U(t) \cdot I(t) dt \quad (1)$$

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