



Scale-up of high voltage electrical discharges for polyphenols extraction from grape pomace: Effect of the dynamic shock waves

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ABSTRACT

This work aims at producing high dynamic shock waves by high voltage electrical discharges (HVED) in a water suspension in order to increase polyphenols extraction from grape pomace. Experiments at the laboratory (1 L) and pilot (35 L) scales were compared. The total specific energy input was varied up to 800 kJ/kg. The intensification of the extraction of total polyphenols was increased 7 times from grape pomace, seeds, skins and stems treated by HVED at both laboratory and pilot scales. However, higher treatment energies are required at the pilot scale to obtain equivalent polyphenols rates. The pressure of the shock wave generated during HVED was measured. When applying the same specific energy input per pulse ($E_{bm} = 0.53$ kJ/kg) at both scales, the entire volume of both treatment chambers is treated by shock waves of similar pressures values (≥ 100 bars). Below this pressure value, the shock waves seem to have no effect on the polyphenols extraction.

Industrial relevance: This paper presents relevant information for the design of generating electrical discharges treatment. The study also addresses a specific case of use of by-products and shows the effectiveness of such technology at the laboratory and pilot scale.

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1. Introduction

Grape pomace is a rich source of polyphenols, which can determine the sensory and health useful properties of juices and wines (Kammerer, Schieber, & Carle, 2005; Kondakova et al., 2009). The major part of polyphenols remains after pressing of grapes in the press residues, or pomace. The grape pomace reaches 20% of the weight of processed grapes. Upgrading of this typical low-value food by-product can be useful for production of polyphenol extracts, functional food components, useful health ingredients and antioxidant additives (Makris, Boskou, & Andrikopoulos, 2007).

The methodologies employed for polyphenols recovery may use organic solvents, like methanol or acetone (Pinelo, Rubilar, Jerez, Sineiro, & Nunez, 2005). Alternative methods like enzymes, supercritical fluid extraction or pressurized liquid extraction can also be used (Corrales, Toepfl, Butz, Knorr, & Tauscher, 2008; Pinelo, Arnous, & Meyer, 2006). Nowadays the safe and simple extraction protocols without employment of organic solvents and chemical additives become most interesting.

Recently, electrical treatments that provoke electroporation and biological tissue damage have also been used for compounds extraction (Balasa & Knorr, 2007; Grimi, Lebovka, Vorobiev, & Vaxelaire, 2009; Loginova, Shynkaryk, Lebovka, & Vorobiev, 2010; López, Puértolas, Condón, Álvarez, & Raso, 2008; Schilling et al., 2007; Toepfl, 2006; Vorobiev & Lebovka, 2008).

Electrical discharges have been studied as a means for cell disruption in biochemistry, biology, medicine and drug delivery (Chen, Zhang, Dai, & Yuan, 2004; El-Aragi, 2009; Mikula, Panak, & Dvonka, 1997). Electrical discharges have been used for bio-compounds extraction from different products (Barskaya, Kuretz, & Lobanova, 2000; Boussetta, Lanoisellé, Bedel-Cloutour, & Vorobiev, 2009; Gros, Lanoisellé, & Vorobiev, 2003; Moubarik, El-Belghiti, & Vorobiev, 2010; Negm, Vorobiev, & Sitohy, 2009; Vishkvatzev, Kuretz, Lobanova, Filatov, & Barskaya, 1998). The mechanisms of formation of the electrical discharge in water are insufficiently delighted. Two types of processes may lead to establishment of a conductive channel in water (Klimkin, 1990). The first hypothesis assumes development of a gaseous phase first, in which electronic avalanches take place. The second hypothesis posits that a gaseous phase is not required. It assumes that breakdown is governed by multiplication of the charge carriers caused by ionization of the liquid. The confrontation between the so-called bubble theory and direct impact ionization model is ongoing. The electrical discharge leads to the generation of hot, localized plasmas that strongly emit high-intensity UV

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light, produce shock waves, and generate hydroxyl radicals during water photodissociation.

One of the most important features of the underwater electrical discharge is the generation of strong dynamic shock waves. During the formation of the plasma channel (1–2 ms) in an electrohydraulic discharge reactor, an intense 5–20 kbar shock wave is generated due to the rapidly expanding plasma channel (Starikovskaia, Anikin, Pancheshnyi, Zatspepin, & Starikovskii, 2001; Touya, Reess, Pecastaing, Gibert, & Domens, 2006). The resulting shock wave can induce pyrolytic and free-radical reactions indirectly via electrohydraulic cavitation (Joshi, Locke, Arce, & Finney, 1995). The pressure shock wave is followed by a rarefaction wave that produces cavitations. The collapsing cavitations create strong secondary shocks with very short duration (≈ 60 ns that sometimes result in sonoluminescence (excitation of light spikes)), and these shocks can interact with structures on the size of cells (Locke, Sato, Sunka, Hoffmann, & Chang, 2006). Shock waves are known to mechanically rupture cell membranes (Howard & Sturtevant, 1997). The effect of the pressure shock wave on biological cellular damage has been studied previously by (Vogel, Busch, & Parlit, 1996). After formation of the plasma channel between electrodes, biological cells are first exposed to the passage of a shock wave. The contact time depends on the distance of biological cells from the electrodes. At the front of the shock wave, pressure gradients of about 600 MPa can be reached after only a few micro- or nanoseconds. Cells that are located in this area are submitted to a high compression by a factor of 1.22 in a very short time (only $20 \cdot 10^{-12}$ s). Taking into account the speed of these events, the effects of shock waves on tissues are located and visible at the microscopic level (cellular or subcellular level).

The aim of this paper is to study the HVED effect at laboratory and pilot scales and to analyse the impact of the pressure waves generated during electrical discharges on the extraction of polyphenols from grape pomace. The experiments carried out at laboratory and pilot scales will be compared; the extraction efficiency will be determined via the contents of total polyphenols.

2. Materials and methods

2.1. Biological material

Fresh industrial grape pomace and its individual components (skins, seeds and stems) from red and white grapes (*Vitis vinifera* L., cultivars “Pinot Meunier” and “Chardonnay”, vintage 2009) from Epernay (France) was obtained as the residue of pressed grapes. The dry matter content in the grape pomace was 30.0 ± 0.1 wt.%.

2.2. High voltage electrical discharges experiments

Extraction experiments were carried out at both laboratory and pilot scales.

For experiments at the laboratory scale, the experimental apparatus (Toms Polytechnic University, Russia) was used. It consisted of a pulsed high voltage power supply (200 nF) and a laboratory one-liter treatment chamber. The treatment chamber contained two electrodes (Fig. 1a). The first one was a stainless steel needle 10 mm in diameter. The second was a stainless disk grounded electrode 35 mm in diameter. A positive pulse voltage was applied to the needle electrode. The high voltage pulse generator provided 40 kV–10 kA discharges during few microseconds.

For experiments at the pilot scale, the high voltage generator (Pau University, France) was composed of a capacitor (200 or 5000 nF) that discharged towards a triggered air spark gap (Fig. 1b). The capacitor was charged with a maximum voltage of 40 kV. The 35 L treatment chamber consisted of a needle electrode of 10 mm in diameter and a grounded electrode of 120 mm in diameter. Measurement of the shock wave pressure was done with a pressure sensor developed by Bauer (1995). It was made of a piezoelectric polymer material (polyvinylidene fluoride PVDF). It had a high natural frequency of

100 MHz. In both cases, the electrodes geometry was the same. The inter-electrode space was 5 mm. The pulse repetition rate was fixed at 0.5 Hz. The elevation temperature due to the treatment was < 7 °C.

2.3. Extraction experiments

The treatment chamber was initially filled with grape pomace, seeds, skins or stems which were further on mixed with distilled water (the liquid–solid ratio, w/w, was fixed at the level of 5) at 20 °C. Note that each product (whole grape pomace, seeds, skins and stems) was studied separately. The total product masses used for laboratory and pilot experiments were respectively 0.3 kg and 7.5 kg. Electrical discharges (up to 1000 pulses) were applied to the aqueous suspension. For control experiments, the extraction was performed in a cylindrical cell under agitation (160 rpm) without electrical treatment. The concentrations of total polyphenols were controlled every 5–10 min during the extraction process.

2.4. Extract analysis

To characterize the electrical treatment efficiency, the extraction rate was quantified by the contents of total polyphenols. The total polyphenols amount was measured spectrophotometrically by the Folin–Ciocalteu method based on a colorimetric oxidation/reduction reaction of phenols (Singleton, Orthofer, & Lamuela-Raventos, 1999). Note that the Folin–Ciocalteu method is not phenol-specific but it can provide a good estimation of the polyphenols content in the extracts. A volume of 0.2 mL of diluted extract and 1 mL of Folin–Ciocalteu reagent (Sigma-Aldrich, France) (diluted 1:10 with water) were mixed. 0.8 mL of Na_2CO_3 (75 g/L) (VWR, France) was then added. The sample was incubated for 10 min at 50 °C and then cooled at room temperature. For the control sample, 0.2 mL of distilled water was taken. The absorbance was measured at 750 nm by the UV/Vis spectrophotometer (Libra S32, Biochrom, France). Gallic acid (Sigma-Aldrich, France) was used for the calibration curve. Results were expressed as mg gallic acid equivalent per liter (mg GAE/L). The analyses were performed in triplicate and average deviation was calculated.

2.5. Simulation of peak pressure and shock wave energy

The generation of shock waves requires the occurrence of a dielectric breakdown in water, after which heat and expansion of plasma channel between the two electrodes produce a dynamic wave. The peak pressure P_0 (Pa) depends on the energy E_B (J) remaining at the time of breakdown and can be approximated by Eq. (1) (Touya et al., 2006):

$$P_0 = k \cdot E_B^\alpha \quad (1)$$

where $E_B = \frac{1}{2} \cdot C \cdot U_B^2$ and U_B (V) is the breakdown voltage value, C (F) is the capacitor capacity, k (m^{-1}) and α are parameters depending on the inter-electrode geometry and the distance d_s between the pressure sensor and the needle electrode. Note that the specific energy input per pulse E_{Bm} (J/kg) is defined as the ratio of the energy input per pulse at the moment of breakdown E_B and the mass of total treated product.

It is important to distinguish the maximum energy stored in the tank capacitor: $E_M = \frac{1}{2} \cdot C \cdot U_M^2$ (where U_M (V) is the charging voltage) from the remaining energy E_B (J) at the moment of breakdown t_B (s).

Before the moment of breakdown t_B , ionic conduction develops through the water to the ground electrode. Then, microdischarges occur inside the bubbles, thus reducing the energy available for dissipation in the plasma channel at the moment of breakdown. Consequently, even for a high E_M value, a breakdown occurring after a long time t_B would lead to a low energy E_B and faint peak pressure values. For a given U_M value, enhancement of the peak pressure is associated with reduction in the time t_B . One can decrease the time of

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