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Combined densification and pulsed electric field treatment for selective polyphenols recovery from fermented grape pomace

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ABSTRACT

The aim of this study is to assess a new process for the valorization of fermented grape pomace using pulsed electric fields (PEF). The combination of densification and PEF treatment was applied on grape pomace of low relative humidity, without any addition of conductive liquid. The kinetics of extraction and the composition of polyphenols were evaluated throughout the subsequent hydro-alcoholic extraction at different temperatures. Optimal parameters of PEF treatment (field strength $E = 1.2$ kV·cm^{-1}; energy input $W = 18$ kJ·kg^{-1}; density $\rho = 1.0$ g·cm^{-3}) increased the content of total polyphenols regardless of the temperature of extraction. The ratio of total anthocyanins to total flavan-3-ols at 20 °C was equal to 7.1 and 9.0 for control and PEF treated modalities, respectively. These results demonstrate the selective nature of PEF treatment in anthocyanin extraction, and thus reveal new possibilities to produce extracts with different biochemical compositions.

Industrial relevance: This study examines the feasibility of densification combined with PEF pre-treatment of relatively low humidity grape pomace for the enhancement of bioactive compounds extraction. The concentration of total phenolic compounds obtained after PEF treatment showed that the use of this technique is relevant for an industrial use, since solvent amount and extraction time can be reduced. Moreover, the selective nature of PEF opens the opportunity to produce extracts of different biochemical compositions. This process is an alternative to conventional pre-treatments of raw material (e.g. dehydration and grinding), which have impacts on product quality and are more energy consuming.

1. Introduction

The re-use of agricultural residues has become a worldwide concern over the last few years, due to the need for environmental protection and the shortage of natural resources. In this line, several investigations have been carried out by different research groups with the aim of valorizing the plant biomass waste (Devesa-Rey et al., 2011; Toscano et al., 2013). The valorization of red grape waste-products is very promising, since grape is one of the most largely produced fruit crops with an annual world production of 58 million tons in 2012 (FAOSTAT, 2012), of which about 80% is used for winemaking (Mazza & Miniati, 1993). The red grape residues are rich in polyphenols, which are natural antioxidants with high added value (Teissedre, 2007; Xia, Deng, Guo, & Li, 2010).

Phenolic compounds from grape pomace are usually extracted by classical extraction techniques using solvents and heating. The selectivity of the extraction processes mainly depends on the molecular affinity between solvent and solute (Cowan, 1999). However, environmental safety, human toxicity and financial feasibility should also be considered in the selection of a solvent for the extraction of bioactive compound. Moreover, the natural variability of raw material and the pre-transformation processes (drying, grinding, etc.) could be determinants for the quantity and composition of extract (Meireles, 2008). For instance, high temperatures can lead to denaturation of targeted compounds and grinding causes significant increase of undesired components extraction. Thus, conventional pre-transformation processes decrease the selectivity and/or the efficiency of the extraction process.

To enhance overall yield and selectivity of bioactive components from plant materials, microwave (Liazid, Guerrero, Cantos, Palma, & Barroso, 2011; Pérez-Serradilla & Luque de Castro, 2011), ultrasound (Vilkhu, Mawson, Simons, & Bates, 2008), and high hydrostatic pressure (Corrales, García, Butz, & Tauscher, 2009; Prasad, Yang, Yi, Zhao, & Jiang, 2009) have been studied as non-conventional methods.

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Pulsed electric field (PEF) treatment is an innovative and non-conventional method that increases the permeabilization of cell membranes due to the electroporation phenomenon (Kotnik, Kramar, Pucihar, Miklavcic, & Tarek, 2012). PEF has gained interest in food processing for the inactivation of microorganisms (Castro, Barbosa-Cànovas, & Swanson, 1993; Heinz, Alvarez, Angersbach, & Knorr, 2001; Mosqueda-Melgar, Raybaudi-Massilia, & Martín-Belloso, 2012) and improving the efficiency of extraction of intracellular compounds (Maskooki & Eshtiaghi, 2012; Vorobiev & Lebovka, 2008) and water (Ade-Omowaye, Angersbach, Taiwo, & Knorr, 2001) from plant materials, amongst which we may also find grapes (Delsart et al., 2013; López, Puértolas, Hernández-Orte, Álvarez, & Raso, 2009; Puértolas, López, Saldaña, Álvarez, & Raso, 2010). PEF has recently shown good results for the enhancement of polyphenols extraction from grape wastes (Boussetta, Vorobiev, Le, Cordin-Falcimaigne, & Lanoisellé, 2012; Boussetta et al., 2009; Corrales, Toepfl, Butz, Knorr, & Tauscher, 2008). The electrical pre-treatment of unfermented grape pomace at high electric field strengths ($E = 20$ kV·cm^{-1}) and delivered energy ($W = 212$ kJ·kg^{-1}) increased the content of polyphenols in the extract during aqueous extraction (Boussetta, 2011). Even fields of moderate strength and time of application, i.e. $E = 1300$ V·cm^{-1} and $t_{PEF} = 1$ s, proved to be an efficient method for the recovery of polyphenols from unfermented grape skins (Boussetta et al., 2009), where a 12% increase in total polyphenols yield was observed. Moreover, the extraction of anthocyanins had been increased by 17% when PEF treatment of field strength 3.0 kV cm^{-1} delivering specific energy of 10 kJ·kg^{-1} was applied to red grape skins (Corrales et al., 2008). These studies were carried out on wet material – relative humidity (RH) ≅ 75% – or with the addition of liquid to the treatment chamber.

Red wine making includes a pressing step that separates the grape pomace from the grape juice after the alcoholic fermentation. This step leads to fermented pomace of low relative humidity, RH ≅ 50%. To the best of our knowledge, no published information in the available literature about PEF effect on the polyphenols extraction from fermented red grape pomace of low RH yet exists or the information is extremely scarce.

This work discusses the effect of the combined use of densification and moderate PEF treatment for the dry pre-treatment of fermented grape pomace on the kinetics of polyphenols extraction. The electric treatment of field strength $E = 1.2$–3.0 kV·cm^{-1} was applied without the addition of any liquid and the index Z of tissue permeabilization induced by PEF was estimated. PEF efficiency for polyphenols recovery was assessed at different densities of raw material. The extraction kinetics of polyphenols was then analyzed at different extraction temperatures (20° C< T <50 °C). The composition of anthocyanins and flavan-3-ols was compared for the different pre-treatments.

2. Material and methods

2.1. Raw material

Dunkelfelder is an early red grape cultivar from Switzerland. It was used for red wine processing, including a maceration–fermentation step. The pomace was collected immediately after pressing at 2 bars (Sutter EPC 50, Bucher Vaslin SA, France) and was treated with 50 mg of SO_2 per kg of raw material (RM). Samples were stored at 4 °C under vacuum until further processing. The dry matter was determined by the measurement of the mass of grape pomace before and after drying the samples at 105 °C overnight and was equal to 44.8%. The pH was 3.76.

In order to avoid complications with repeatability due to the heterogeneity of the raw material, the skins and the seeds were separated with a vibrating sifter (Retsch GmbH, Germany). Afterwards, small fractions (diameter Ø < 2.8 mm) and large fractions (Ø > 5.5 mm) were removed. Then, the two standardized fractions were manually and homogeneously mixed (49% of seeds and 51% of skins fresh weight).

2.2. PEF treatment for cell permeabilization

For the estimation of cell permeabilization index Z, a sample of reassembled grape pomace (150 g ± 1 g) was placed in a treatment chamber. The treatment chamber is a polypropylene container in which two stainless electrodes are inserted and separated by a distance of 1.25 cm. The PEF-treatment cell was placed under a texture analyzer (models TA-XT Plus and TA-HDi, Stable Microsystems, Surrey, UK) to monitor the compression parameters of grape pomace. Different pressures in the range of 0–10 bars were applied to the grape pomace in order to assess the effect of the density ρ, ranging from 0.6 to 1.3 g·cm^{-3}, on PEF efficiency. Electrodes were connected to the PEF generator (Hazemeyer, Saint Quentin, France) capable of delivering a maximum voltage of 5 kV at 1 kA current, which provided unipolar pulses of rectangular shape (Fig. 1).

A series of N trains were applied until the desired energy delivery was attained, after which the PEF treatment was stopped. Each series consisted of n pulses with pulse duration t_i, and time interval Δt between the start of two consecutive pulses. The following parameter values (varied within a range, where a range is given) were used in PEF experiments: $E = 0$–3.0 kV·cm^{-1}, $n = 200$–2000, $N = 20$–200, $t_i = 100 \pm 1$ μs, $\Delta t_i = 100$ ms.

Experimental data (current (I), voltage (U), t_i, Δt_i, n) were collected using a data logger (Nemo, IME Messgeräte GmbH, Germany) and a dedicated software developed locally by our electronic department (Service Electronique, UTC, Compiègne, France) using HPVee software (Agilent VEE Pro, Agilent Technologies).

The electrical energy consumption (W, kJ·kg^{-1}) of the PEF treatment was calculated as:

$$W = \frac{\sum_{i=1}^{n} U \cdot I_i \cdot t_i}{m} \quad (1)$$

where U is the PEF voltage (V), I is the current intensity (A), t_i is the pulse duration (s) and m is the mass of wet raw material (kg).

The temperature was measured with a Teflon-coated thermocouple (Thermocoax, Suresnes, France) inserted into the sample (measurement precision ± 0.1 °C). The increase of temperature was less than 3 °C after the PEF treatment. The conductivity was measured during two consecutive series of pulses with an LCR meter U1733C (Agilent technologies) at a frequency of 1 kHz. The degree of tissue damage was estimated from the electrical conductivity-based permeabilization index Z (Lebovka, Bazhal, & Vorobiev, 2001,2002):

$$Z = \frac{\sigma - \sigma_u}{\sigma_d - \sigma_u} \quad (2)$$

where σ (μS·cm^{-1}) is the periodically measured electrical conductivity of sample, and the subscripts 'u' and 'd' refer to the electrical conductivities of the untreated and completely damaged samples, respectively. The value of σ_d was estimated from the measurements of electrical conductivity of tissue damaged by PEF treatment at $E = 3.0$ kV·cm^{-1}, repeatedly applied until constant conductivity of the sample was reached.

2.3. Extraction experiments

After the application of PEF treatment, polyphenols extraction from grape pomace (100.0 ± 0.1 g) was carried out in a mixture of ethanol and water (50/50, v/v), maintained at the desired temperature (20 °C, 35 °C and 50 °C) in a cylindrical extraction cell. The liquid-to-solid ratio was maintained at the value of 5. A gentle agitation at 160 rpm (16.8 rad·s^{-1}) was provided using a round incubator of 12.5 mm shaking throw (Infors HT Aerotron, Bottmingen, Switzerland). For untreated samples, the same protocol of extraction was used. Regular sampling was carried out during 420 min of extraction. At the end of extraction,