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Effect of alternative physical pretreatments (pulsed electric field, high voltage electrical discharges and ultrasound) on the dead-end ultrafiltration of vine-shoot extracts

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ABSTRACT

This work is devoted to the dead-end ultrafiltration of vine shoot extracts; these extracts were obtained after a previous step of solid–liquid extraction assisted by different physical pretreatments. Grinding, high-voltage electrical discharges (HVED), pulsed electric fields (PEF) and ultrasounds (US) were studied. The highest cellular damage (Z) was provoked by grinding (360 kJ/kg; $Z = 1$) then HVED (242 kJ/kg; $Z = 0.71$). The resulting protein and polyphenol concentrations were 0.097 and 0.4 mg/mL for grinding and 0.082 and 0.3 mg/mL after HVED pretreatment. A relationship between the extraction process and the unstirred dead-end ultrafiltration parameters was found. The highest cellular damage obtained with grinding ($Z = 1$) gave the greatest specific cake resistances ($\alpha c \approx 4.8 \times 10^{13}$ m/kg). A correlation was therefore observed between cellular damage resulting in a higher polyphenol concentration and the membrane fouling phenomenon. HVED treatment (242 kJ/kg) gave a lower specific cake resistance ($\alpha c \approx 4 \times 10^{13}$ m/kg) than grinding. Dynamic ultrafiltration was also conducted to decrease membrane fouling and study polyphenol purification and concentration in retentates. The experimental conditions leading to the highest filterability (highest flux J) and polyphenol retention conducted on HVED (242 kJ/kg) extracts were a stirring velocity of 150 rpm, a pressure value of 4.5×10^5 Pa and a nominal molecular weight cut-off equal to 50 kDa for the polyethersulfone membrane. Under these conditions, the filterability of HVED extracts (2.7×10^{-6} m^3/m^2 s) was higher than that of grinding (2×10^{-6} m^3/m^2 s). Up to 8% of polyphenols from the grinding extract were shown to deposit on the membrane. A lower percentage was found for HVED (7%). Polyphenols were therefore shown to be implicated in membrane fouling and pore blocking for unstirred and dynamic ultrafiltration processes.

1. Introduction

Growing interest in winemaking and viticulture waste valorization has been observed [1–8]. Vine shoots are grape industry wastes resulting from cane pruning, generally composted or burned. Vine shoots contain high amount of polyphenols [2] and the recovery of these compounds can increase the economic value of this by-product. New environment-friendly technologies have been proposed to improve the extraction process reducing chemical use and operational time [9]. Among them, three physical pretreatments have been shown to be efficient for the intensification of polyphenol extraction from vine shoots: high voltage electrical discharges[1] (HVED), pulsed electric fields[2] (PEF) and ultrasounds[3] (US). The specific energetic threshold was determined for each treatment (HVED (10 kJ/kg), PEF (50 kJ/kg) and US (1010 kJ/kg)). For the same energy input of the treatments (254 kJ/kg), polyphenol diffusion coefficient was the highest for HVED

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[1] High voltage electrical discharges: HVED.
[2] Pulsed electric fields: PEF.
[3] Ultrasounds: US.

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(9.4×10^{-12} m^2/s) followed by PEF (9.88×10^{-13} m^2/s) then US (6×10^{-13} m^2/s) [2]. HVED is based on the electrical breakdown phenomenon in water. Sufficient electrical field intensity is able to induce an electron avalanche responsible for the starting point of streamer propagation from the positive to the negative electrode. Secondary phenomena, such as bubble cavitation, liquid turbulence and high-amplitude pressure shock waves contribute in enhancing cellular damage and biomolecules extraction from cell cytoplasm [10,11]. PEF induces cell membrane electroporation. It creates high electrical potential difference (exceeding the threshold of 1–2 V for plant tissues) throughout the cell membrane, causing loss in its semipermeability [12]. US generates air bubbles that propagate in the liquid medium with expansion and compression cycles. A local pressure inside of air bubbles can attain 50 MPa and a local temperature up to 5000 °C [13]. The cavitation phenomenon resulting from bubble collision is capable of damaging proximate cell membranes [14]. HVED, PEF and US were shown to improve polyphenol extraction from different matrices [15–17]. Membrane technologies such as ultrafiltration and microfiltration have been widely used for the subsequent step of polyphenol purification and concentration [18,19]. Loginov et al. [20] used ultrafiltration process to purify polyphenol flaxseed hull extracts by separating them from proteins. Liu et al. [21] used dead-end ultrafiltration to concentrate polyphenol extracts resulting from HVED-treated grape pomace. The fouling process decreases filtration flux and affects filtrate quality by modifying membrane permeability and molecular selectivity [22]. To our knowledge, no study has been conducted on the dead-end ultrafiltration process of vine shoots extracts. The filterability of shoot extracts, the implication of polyphenols in membrane clogging and their purification by ultrafiltration are not yet well understood. In this study the effect of the extraction parameters on unstirred and stirred dead-end ultrafiltration was studied. Three alternative physical pretreatments: PEF, HVED and US were used to enhance polyphenol extraction from vine shoots and were compared to conventional grinding. The relationship between the pretreatments and the membrane fouling process was investigated. The most adequate pretreatment was chosen in terms of polyphenol recovery (extraction process), extracts quality (purity toward polyphenols) and filterability (ultrafiltration process).

2. Materials and methods

2.1. Raw material

Vitis vinifera var. Grenache Blanc (France) vine shoots were used. Vine shoots were pruned in 2012, and stored in a cold room at 4 °C until used. For HVED, US, PEF and control extracts, vine shoots were cut into cylinders of 1 cm height and a mean diameter of 5 mm.

2.2. Extraction experiments

2.2.1. Grinding pretreatment

Fifteen grams of vine shoots were grinded by means of a coffee grinder (SEB, France). Powdered samples (<1 mm) were then used for extraction experiments. The specific energy of the grinding process[4] E_{gr}, was 360 kJ/kg, and was calculated as following

$$E_{gr} = \frac{W_{gr} \times t_{gr}}{m} \quad (1)$$

where t_{gr} is the total grinding duration (s), m is the product mass (kg), and W_{gr} is the specified power of the coffee grinder (0.18 kJ/s). This energy consumption corresponds well to the data published in [23] for the grinding of wood wastes.

2.2.2. High voltage electrical discharges pretreatment

As shown in Fig. 1a, the HVED apparatus was a treatment chamber with stainless electrodes and a high voltage power supply (Tomsk Polytechnic University, Tomsk, Russia). The electrodes consisted of a stainless steel needle with a 10 mm diameter and a grounded plate electrode with a 35 mm diameter. The electrodes were distant of 5 mm and a positive pulse voltage of 40 kV was applied to the needle electrode. The electrical breakdown in water generated electrical discharges with a duration of approximately 10 μs for each discharge. The generator imposed a discharge repetition rate of 0.5 Hz. Cylindrical vine shoots (30 g) and water (300 g) at 50 °C were introduced between the electrodes. The temperature elevation of the suspension during the treatment was controlled every 100 pulses, limiting thus the heating process to less than 2 °C for the whole treatment duration.

The specific HVED energy input[5] E_{HVED} (kJ/kg) was calculated from Eq. (2)

$$E_{\mathrm{HVED}} = \frac{E_p \times n}{m} \quad (2)$$

where E_p is the energy of one pulse delivered by generator (0.160 kJ), n is the number of pulses (n = 100, 250, 350 and 500) and m is the mixture (shoots + water) mass (kg). Four different treatment energies (49, 121, 170, 242 kJ/kg) were then compared in this study.

2.2.3. Pulsed electric field pretreatment

The PEF apparatus consisted of the same generator as that used for HVED; however the PEF treatment chamber had an electrode area of 95 cm^2, consisting of two stainless steel parallel disks (Fig. 1b). The high voltage power supply provided 40 kV to 10 kA pulses. The inter electrode gap was fixed at 3 cm thus resulting in an electric field strength E of 13.3 kV/cm. The duration of each pulse was approximately 10 μs, and the pulse repetition rate of 0.5 Hz was imposed by the generator. The temperature of the suspension (50 °C) was controlled by a series of 1 min pauses every 100 pulses, limiting thus the temperature elevation to less than 5 °C for the whole treatment duration. The specific PEF energy input[6] E_{PEF} (kJ/kg) was calculated from Eq. (3)

$$E_{PEF} = \frac{E_P \times n}{m} \quad (3)$$

where the value of E_P (0.160 kJ) was the same for PEF and HVED treatments, the number of pulses was fixed (n = 1000), and the mixture (shoots + water) mass m (kg) was the same as for the HVEF treatment (30 g of vine shoots 300 g of and water). This mixture was introduced at 50 °C between the electrodes and a total energy input of 484 kJ/kg was delivered.

2.2.4. Ultrasound pretreatment

As shown in Fig. 1c, the ultrasound treatment chamber ($190 \times 200 \times 130$ mm^3) contains a titanium ultrasound probe (H14 Hielscher GmbH, Germany), with a length of 100 mm and a diameter of 14 mm, connected to the ultrasonic generator (Hielscher GmbH, Stuttgar, Germany) with a maximal power of 400 W and a maximal frequency of 24 kHz. In this study, the power and frequency regulators were both fixed to 100% (400 W and 24 kHz respectively). The probe was submerged (length of 90 mm) in a mixture containing 300 g of water (50 °C) and 30 g of cylindrical vine shoots in a narrownecked glass flask

[4] Energy of the grinding process: E_{gr}.

[5] HVED energy input: E_{HVED}.

[6] PEF energy input: E_{PEF}.