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Shelf-life evaluation of virgin olive oil extracted from olives subjected to nonthermal pretreatments for yield increase

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ABSTRACT

Critical process parameters for the yield and quality of olive oils are the temperature and treatment duration of the malaxation process. A balance between oil yield and quality must be achieved. Novel technologies such as High Pressure (HP) and Pulsed Electric Fields (PEF) that cause alterations to cell permeability, may promote the olive oil extraction process during malaxation, resulting in higher oil yields.

Three different varieties of Olive fruits (*Tsounati*, *Amfissis* and *Manaki* var.) were subjected to different HP (200 and 600 MPa, 25 °C for 1 and 5 min) and PEF (1.6–70.0 kJ/kg) process conditions before malaxation (30 min at 30 °C). The extraction yield was increased up to 18 and 16% for PEF and HP treated olive fruits, respectively. The shelf-life tests indicate that the oil quality from nonthermally pretreated olives is dependent on the technology and process conditions used. An increase of oil oxidative stability for PEF and HP treated olives was shown, compared to control samples.

The results show that HP and PEF could be applied for the production of superior quality virgin olive oil with increased yield.

1. Introduction

Olive oil is a high-value edible oil which is appreciated for its sensorial characteristics as well as its high nutritional value (Visioli, Bellomo, & Galli, 1998). It is extracted from the fruit of *Olea europaea* L. using mechanical or physical procedures (Uceda, Gabriel, & Jimenez, 2006). For olive oil extraction, the conventional procedure includes a malaxation process for yield increase. The application of malaxation results in an approximately 5% yield increase compared to non-malaxated olives, which is a significant percentage for the industries. Novel technologies such as High Pressure (HP) and Pulsed Electric Fields (PEF) that cause alterations to cell permeability may promote the olive oil extraction process during malaxation, resulting in higher yields. Critical process parameters are the elevated temperature having a negative effect on the extracted oil and the treatment duration. A balance between oil yield and quality must be achieved.

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HP processing not only is being applied as a pasteurization process to extend the shelf life and control safety risk in a wide range of food products but also it can be used for the extraction of active ingredients from plant sources (Zhang et al., 2004). In particular, HP may lead to structural changes in foods such as cellular deformation, cellular membrane damage and protein denaturation (Zhang, Li, Tatsumi, & Isobe, 2005). It can also improve the mass transfer rate, enhance solvent permeability in cells as well as secondary metabolite diffusion (Dörnenburg & Knorr, 1993 and Ahmed & Ramaswamy, 2006). Recent studies have indicated that HP may reduce the processing time and obtain higher extraction yield of several compounds from plant sources (Zhang et al., 2004, 2005 and Corrales, Toepfl, Butz, Knorr, & Tauscher, 2008). Such examples are the extraction of flavonoids from propolis (Zhang et al., 2005), anthocyanins from grape skin (Corrales et al., 2008) and ginsenosides from the roots of *Panax ginseng* (Zhang, Tan, Zhao, & Wang, 2007).

Pertaining to PEF technology, it has been proven as an effective technique for irreversible permeabilization of cell membranes in plant tissues without a significant temperature increment of the sample, with low operation cost (Toepfl, Heinz, & Knorr, 2006 and Grimi, Mamouni, Lebovka, Vorobiev, & Vaxelaire, 2011). PEF technology has been already applied to enhance the extraction yield of juices from fruits and vegetables, reducing the drying times or improving the extraction of intracellular valuable compounds such as colorants, sucrose, or polyphenols

(Vorobiev & Lebovka, 2008; Donsi, Sessa, & Ferrari, 2010; Knorr et al., 2011). Published data related to the application of PEF for assisting olive oil extraction are promising. Guderjan, Topfl, Angersbach, and Knorr (2005) demonstrated firstly the potential of PEF for increasing oil extraction yield up to 7.4% from fresh olives. Abenoza et al. (2013) pointed out the benefits of this technology on olive oil extraction yield as well as its impact on product quality, using a laboratory-scale olive oil extraction system. Other researchers maintained that PEF treatment not only improves the oil extraction yield, but also increases the content of several compounds such as polyphenols, phytosterols and tocopherols, maintaining the EU legal standards of highest quality olive oil (Puértolas & Martínez de Marañón, 2015). When the cell is exposed to an external electric field, pore formation of cytoplasmic membrane takes place, due to the accumulation and attraction of oppositely charged ions at both sides of the nonconductive membrane (Tessie et al., 2002). After PEF treatment, when the critical field strength is excessive, the electroporation can be irreversible leading to leakage of intracellular compounds and improving the mass transfer. Similarly, High pressure can cause some structural changes in foods, such as cellular deformation and cellular membrane damage (Zhang et al., 2004). High pressure may also improve the mass transfer rate and enhance solvent permeability in cells as well as secondary metabolite diffusion (Dörnenburg & Knorr, 1993 and Ahmed & Ramaswamy, 2006). The differential pressure between the cell interior and the exterior of cell membranes is big enough to lead to rapid permeation and improvement of the mass transfer, as a result of their different compressibility. Consequently, equilibrium in the concentration between the cell interior and the exterior of cell membranes is achieved in short time.

Oxidative stability is an important parameter in evaluating the quality of oils, as it gives a good estimation of their susceptibility to oxidative degeneration, the main cause of their alteration. The greater or lesser stability of an oil means the conservation or not of the so-called dynamic parameters during the useful life of the product. In the course of the autoxidation reaction a series of compounds are formed, causing off-flavors and rancidity, loss of nutritional value and finally consumer rejection of the food product. Autoxidation is therefore the main cause of olive oil quality deterioration and its reaction rate determines the shelf-life of this product (Frankel, 1998). Oxidation is a process which occurs fairly slowly at room temperature, so an accelerated shelf-life test is necessary to choose what conditions are to be used to increase the oxidation rate and the analytical determinations to follow the progress of the reaction, preferably by measuring both primary and secondary oxidation products. In choosing the method used to determine the induction period of the reaction, which should correspond to level of rancidity detectable by the consumer, a specific value of an oxidation index or a sudden change in the rate of oxidation must also be selected. In the case of virgin olive oil, the EU regulation (EC 796/2002) establishes upper limit values for different oxidation indexes (peroxide value 20 meq/kg; K_{232} 2.50 and K_{270} 0.20). In the literature, there are limited works on the effect of PEF treatment on oil yield increase (Guderjan et al., 2005; Abenoza et al., 2013), but no references on the effect of HP technology on yield increase, either on the shelf-life stability of oils from olives pretreated with novel technologies.

The objective of this work was to correlate and comparatively evaluate the potential benefit of the olive oil yield increase applying novel technologies such as PEF and HP with the oxidative stability of the oils through shelf-life tests.

2. Materials and methods

2.1. Plant material

The study was carried out with olive fruits from three different varieties (*Tsounati*. *Amfissis*. *Manaki* var.), which were harvested in October. The olive fruits were at the 1st degree of ripening (green skin-color) (Aparicio, Roda, Albi, & Gutiérrez, 1999). All varieties immediately were transported to the laboratory for olive oil extraction. After grinding the olive fruit with the mill, 900 g of the olive paste were placed into a stainless-steel mixing container for malaxation. The malaxation was conducted at 30 ± 0.2 °C for 30 min. After malaxation, the olive paste was centrifuged at 3.000 ×g for 4 min and then the oil was collected. After a filtration process to remove water waste and solid impurities, final oils were bottled and subsequently, analyzed. For the investigation of the effect of PEF and HP technology, the olive fruits were accordingly treated before milling.

2.2. High pressure treatment

Olive fruits were washed, cleaned of leaves, weighed and finally, processed with HP. HP treatment was conducted at the following combinations of pressure and time: 200 MPa for 1 min, 200 MPa for 5 min, 600 MPa for 1 min and 600 MPa for 5 min at 25 °C. The conditions were selected based on preliminary experiments conducted targeting in as milder as possible treatment conditions in parallel with significant yield increase. High pressure treatments were performed using a laboratory scale HP equipment with a maximum operating pressure and temperature of 1000 MPa and 90 °C, respectively (Food Pressure Unit FPU 1.01. Resato International BV. Roden. Holland) consisting of a high pressure unit with a pressure intensifier and a vessel of 1.5 lt.

2.3. PEF treatment

As in the case of HP processing, olive fruits were washed, cleaned of leaves, weighed and finally, processed with PEF. The PEF treatment involved 5 conditions at electric field strength of 1.8 kV/cm, 15 μs pulse width and a frequency of 300 Hz with total amount of energy 1.6, 13.3, 20.0, 50.0 and 70.0 kJ/kg. The selection was based on preliminary experiments and works cited in the literature (Puértolas & Martínez de Marañón, 2015 and Abenoza et al., 2013). The initial temperature of the samples was around 20 °C and never exceeded 27 °C. Pulsed electric field process was conducted in a versatile pilot scale system for food processing (Elcrack-5 kW. DIL. Quakenbruck. Germany). Olives were treated in a 80 × 100 × 50 mm (gap × length × depth), 400 ml volume stainless steel batch chamber for cell disintegration. Food is capable of transferring electricity because of the presence of several ions, giving the product in question a certain degree of electrical conductivity.

2.4. Olive oil analysis

2.4.1. Determination of olive oil extraction yield

Olive oil extraction yield was calculated taking into account the mass of the olive paste (kg) after grinding and the final olive oil recovered after natural decantation (ml). The extraction yield was expressed in percentage (%) of olive paste to final mass of olive oil.

2.4.2. Chemical analysis of olive oil

General chemical parameters, free acidity (% of oleic acid), peroxide value (meqO_2/kg), K_{270} and K_{232}, were determined according to the analytical methods described in the Regulation EEC/2568/91 of the European Union Commission and later modifications.

2.4.3. Determination of shelf life of olive oils

Several samples (15 g) of HP or PEF pretreated and untreated olive oils were stored in darkness at different temperatures (25, 35, and 45 °C) in amber glass bottles. One bottle was taken from the incubator for analysis at predetermined time intervals.

2.4.4. Chemical analysis of olive oil

Oxidative stability was studied through shelf-life study of the oils measuring appropriate indices such as peroxide value (PV). Oxidative stability was evaluated by the Rancimat method (Gutierrez, Albi, Palma, & Rios, 1989) and was expressed as the oxidation induction