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Combining high power ultrasound pre-treatment with malaxation oxygen control to improve quantity and quality of extra virgin olive oil

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ABSTRACT

The objective of this study was to evaluate high power ultrasound (HPU) treatment before malaxation and control of oxygen during malaxation on extraction efficiency and quality of extra virgin olive oil (EVOO). HPU treatment (150 W and 20 kHz, 13.5 kJ kg^{-1}) was followed by controlling headspace oxygen concentrations (2%, 5%, 10%, and 21% (control)) during malaxation. Oil yield and various oxidative quality parameters were evaluated. The results showed that HPU treatment significantly ($p < 0.05$) increased oil yield and oil extractability. Significant differences ($p < 0.05$) between the treatment oxygen concentrations and control were found for the quality parameters measured. Oxygen concentration of 5% in the malaxation headspace appears to be the best balance between oil extraction and quality. Combining these technologies presented potential advantages for EVOO production.

1. Introduction

Extra Virgin Olive Oil (EVOO) is the natural olive fruit juice extracted from fresh and healthy fruit (Olea europaea L.) by mechanical methods only (European Communities, 1991; International Olive Oil Council, 1996), and is a preferred vegetable edible oil by consumers in terms of health benefits and nutritional value (Abenoza et al., 2013; Clodoveo et al., 2014; Covas et al., 2006; Inglese et al., 2011; Pastore et al., 2014; Snchez-Ortiz et al., 2016). The extraction process of olive oil has gained increasing attention in recent years, and several studies confirmed that malaxation is a fundamental step in this process because physical, chemical and biochemical phenomena result (Frankel et al., 2013; Inarejos-García et al., 2009; Inglese et al., 2011; Leone et al., 2014; Parenti et al., 2006). Effective malaxation (optimal condition for extraction) extracts the maximum oil yield of high-quality EVOO while maintaining antioxidant compounds and favorable sensory characteristics (Clodoveo et al., 2013; Iqdiam et al., 2017). Malaxation is a mixing operation, where time, temperature, and headspace conditions during malaxation are key factors in determining the efficiency of extraction, and resulting quantity and quality of EVOO (Angerosa et al., 2001; Catania et al., 2013; Clodoveo et al., 2014b; Kalogeropoulos and Tsimidou, 2014; Kalua et al., 2006; Migliorini et al., 2006; Parenti et al., 2006; Tamborrino et al., 2014). Previous studies reported improved oil yield when mixing time was extended and olive paste temperatures exceed 29 °C (± 1 °C), (Angerosa et al., 2001; Clodoveo, 2012; Clodoveo et al., 2014). However, this practice in the presence of oxygen is known to activate the lipoxygenase pathway (LOX), thereby generating volatile compounds that affect the flavor of EVOO, as well as altering desirable sensory properties associated with the final product (Angerosa et al., 2001; Clodoveo, 2012; Clodoveo et al., 2013; Puértolas and de-Marañón, 2015; Selvaggini et al., 2014). Moreover, mixing olive paste for long periods of time in non-hermetic headspace malaxers (globally 95% non-hermetic) often involves a decrease in EVOO's polyphenol content and related quality parameters, such as oxidative stability and bitterness (Clodoveo et al., 2015; Gallina et al., 2005). Regarding malaxation headspace conditions, previous investigations confirmed that control or reduction of oxygen concentration in the malaxation headspace can inhibit endogenous enzymes, such as polyphenoloxidase (PPO) and peroxidase (POD), which are primarily responsible for the enzymatic degradation of phenolic compounds, affecting product stability and favorable sensory characteristics (Catania et al., 2013; Kalogeropoulos and Tsimidou, 2014). Several studies confirmed that hermetically sealed malaxers and/or control of oxygen concentration in the headspace during malaxation produced less

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oxidized oils and showed higher concentrations of antioxidant compounds than the control environment (open malaxation) (Catania et al., 2013; Clodoveo et al., 2015; Leone et al., 2014; Migliorini et al., 2006; Servili et al., 2008). However, controlling or reducing the oxygen concentration of malaxation headspace significantly decreased oil yield because of postulated reduction in endogenous enzyme activity (Leone et al., 2014). The rationale behind this is described by Migliorini et al. (2006), who reported that low malaxation headspace oxygen reduces the hydrolytic activity of lipase responsible for rupturing vacuole cell membranes. However, only a few papers have reported the relationship between low malaxation headspace oxygen concentration and efficacy of malaxation in regard to oil yield. Since previous information does not establish the optimum headspace O_2 concentration during malaxation, this research focuses on using and measuring constant O_2 concentrations in the headspace during malaxation.

Different approaches have been used to enhance malaxation efficiency with less mixing time (Abenoza et al., 2013; Amirante et al., 2006; Leone et al., 2014b; Leone et al., 2016; Puértolas and de-Marañón, 2015), one of them being high power ultrasound (HPU) processing (Amirante and Clodoveo, 2017; Bejaoui et al., 2016; Clodoveo et al., 2013, 2017; Iqdiam et al., 2017; Jiménez et al., 2007; Márquez et al., 2006). HPU induced cavitation helps to form gas pockets at high negative pressure in the olive paste, which disrupts cell walls, and assists in the removal and additional accumulation of oil droplets from tissue cells, which are then easily separated by centrifugation (Clodoveo et al., 2015, 2017). Moreover, several studies confirmed that high-frequency ultrasound maximized the sonic separation of the components (Juliano et al., 2017; Leone et al., 2017, 2018). The aim of this study is to assess the potential benefits of combining HPU pre-treatment on olive paste before malaxation while controlling oxygen concentration in the malaxation headspace for increased oil yield, extraction efficiency, enhanced quality, and sensory characteristics of EVOO. Furthermore, this study will provide unique data by combining HPU pre-treatment with different constant levels of oxygen in the malaxation headspace to assist the EVOO industry by optimizing extraction procedures without influencing EVOO quality.

2. Materials and methods

2.1. Fruit collection and properties

Fresh drupes of Arbequina cultivar (*Olea europea L.*) were manually harvested during 2016 from olive growers in Ocala, FL. Olive fruit were transferred to 26 L plastic cooler boxes, and sent to the food processing laboratory at the University of Florida (Gainesville). Olive fruit characteristics commonly measured are maturity index (Beltran et al., 2003), and moisture and fat content according to the Official Method 925.10 (AOAC, 2000). The results are presented as the mean value of triplicate samples.

2.2. Pilot plant

The extraction system was designed and built to study the extraction process by simulating a real industrial process. The extraction system was tested through experiments carried out in the pilot plant at the Food Science and Human Nutrition Department, University of Florida (Gainesville). The system is composed of three units: a stainless-steel hammer mill crusher (Conair™ SS115, Fisher Scientific, NY, USA), a malaxer (Abencor system, MC2, Seville), and a centrifuge (Allegra X-15 centrifuge, USA). Olive fruit processing is presented in the diagram (Fig. 1).

2.3. High power ultrasounds (HPU)

HPU treatments consisting of 150 W/cm^2 and 20 kHz (Sonics and Materials Inc., CT, USA) were performed before malaxation in the pilot plant. A Sonics (Model No. VCX 1500 CT) system was used to generate ultrasound by a power supply (net power output: 1500 Watts. frequency: 20 kHz) with dimensions (H = 178 mm, W = 380, D = 463.5 mm), and a standard titanium alloy probe (part No. 630–0617, tip diameter = 25 mm, solid length = 230 mm, weight = 680 g). The system was designed to provide 1500 W of maximal output power at 20 kHz (Fig. 2). Olive fruits were washed and crushed by a stainless-steel hammer mill (Conair™ SS115, Fisher Scientific, NY, USA). The olives were introduced into a stainless-steel container with a three-arm hammer rotating at a speed of 2200 rpm. Once properly crushed at room temperature, the ground olives fell through a 0.7 cm grid and collected as a paste. The crushed pastes were divided into triplicate samples (4000 g/each), and placed in a stainless-steel container (diameter: 17 cm, height: 22 cm, volume: 6 L) that was open to atmospheric oxygen, and treated directly with HPU (150 W and 20 kHz, 13.5 kJ kg^{-1}) with stirring for 6 min. The specific energy generated by the ultrasound transducers was measured according to Clodoveo et al. (2017) (Eq (1)):

$$Li = Ps * t / m \tag{1}$$

Where Li is the energy provided per kg of olive paste in static conditions, Ps (150 W) and t (6 min) are the delivered power and the operating time of the transducer, respectively; m denotes the mass of the specimen (4 kg olive paste).

The olive paste container was placed in the center of the stainless-steel water bath (diameter: 32 cm, height: 30 cm, volume: 12 L) that was open to atmospheric oxygen at 20 ± 1 °C (temperature of water tap) during the HPU experiment. The ultrasonic probe was immersed directly into the olive paste to a depth of 8 cm (middle of the olive paste total depth (16.5 cm) in the stainless-steel container) for all HPU experiments.

2.4. Temperature measurement

A thermocouple (Type-T, Omega. com. USA) connected to a thermometer (Model 199 Celsius Digital Thermometer, Omega Engineering ING. CT, USA) was used to measure the olive paste temperature before, during and after HPU treatment. To ensure accurate measurement, three thermocouples were used during the experiment, the first was submerged in the bottom of the container (16.5 cm), the second was placed in the center of the container (8 cm), and the third was found near the surface of the olive paste. The results represent the mean value for temperatures recorded from the three thermocouples at each time.

2.5. Malaxation experiment

The malaxation experiments were performed using a laboratory scale stainless-steel malaxer (7 kg capacity) (Abencor system, MC2, Seville) modified to be gas-tight. A sample (4 kg) of the crushed olive paste was placed in the malaxer and mixed for 35 min. The malaxer contains a reel with three spiral blades rotating at 18 rpm to homogenize the olive paste. Four headspace conditions were applied to olive paste in the malaxer with and without HPU treatment: 21% oxygen using an open malaxer (control/normal atmosphere 30 kPa); and 10% oxygen; 5% oxygen; and 2% oxygen at pressure (70 kPa) using the malaxer hermetically sealed. The malaxer was equipped with two valves, the first was an inlet valve for incorporating the headspace mixture of air and HPLC-grade nitrogen (Multi-Component Gas Mixing System Series 4000, Environics, Inc. CT, USA) into the malaxer chamber while the second valve was the outlet valve for removing the excess air mixture from the malaxation chamber. To prevent the gas mixture from back flowing into the malaxation headspace through the outlet valve, the gas outlet was discharged through water placed in a glass beaker (diameter: 10 cm, height: 14 cm, volume: 1 L) using a plastic tube connected to the outlet valve and immersed inside the water. Olive