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# Continuous conditioning of olive paste by high power ultrasounds: Response surface methodology to predict temperature and its effect on oil yield and virgin olive oil characteristics



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#### ABSTRACT

The purpose of this work is the study of the continuous conditioning of olive paste using High Power Ultrasounds (HPU) previous to malaxation, and its effect on extraction yield and Virgin Olive Oil (VOO) characteristics. For this purpose a laboratory scale device for HPU treatment experiments was developed. The HPU induced an instantaneous heating of olive paste until 28 °C whereas it took 20–30 min in the

traditional malaxation condition. The temperature increase depended on olive characteristics, olive paste flow rate and HPU intensity. A response surface model was carried out to predict the olive paste temperature for the variables identified previously. The sonication treatment improved the oil yield and gave higher extractability than conventional malaxation. The HPU treatment did not cause alteration on VOO quality indexes and composition. Olive oil volatile compounds related with oxidation mechanisms showed lower concentration for VOOs from HPU treated olive paste.

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#### 1. Introduction

The olive paste malaxation is a fundamental step of the Virgin olive oil (VOO) extraction procedures, since let to reach high and satisfactory extraction yield (Aguilera, Beltran, Sanchez-Villasclaras, Uceda, & Jimenez, 2010). Also, it has several effects on the VOO quality parameters, nutritional and sensorial characteristics (Aguilera, Jimenez, Sanchez-Villasclaras, Uceda, & Beltran, 2015). The main objective of kneading is the coalescence of oil drops, formed during olive crushing, to form the oily continuous phase. This phenomenon depends on olive paste rheological characteristics, kneading operating conditions (time and temperature) and addition of technological coadjuvants. Previous studies showed that to obtain higher oil yield it was necessary to perform the olive paste kneading at higher temperature and prolonged time (Aguilera et al., 2010).

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However, in order to avoid any undesirable changes in oil composition and quality, it was recommended malaxation at softer conditions than 28 °C and 60 min. The main factor leading to increase the kneading time is because heating up olive paste to 28 °C requires from 15 to 20 min in the thermo-malaxer (Jimenez, Beltran, & Uceda, 2007). The olive paste heating in the malaxer takes place from the wall to the central axe. This energy transfer is strongly influenced by the characteristics of paste (rheological properties and composition which depend on the olive varieties and the fruit ripeness) and operating conditions (geometry of malaxer, blade rotation speed and olive paste residence time in the malaxer).

Recently High Power Ultrasounds (HPU) were proposed for olive paste preparation (Jimenez et al., 2007; Jimenez, Beltran, Uceda, & Aguilera, 2006). Ultrasounds are defined as the sound waves frequencies, not audible for human ear, from 20 Hz up to 20 KHz (Mason, 1998). These ultrasonic waves are generally classified in power ultrasounds (20 kHz–1 MHz) and diagnostic ultrasounds (higher than 1 MHz). There are two principal effects resulting of high power ultrasounds (HPU) propagation through a medium: physical and chemical (Mason, Paniwnyk, & Lorimer, 1996). The main physical effects of HPU application was the mechanical movement generated by high and low pressure cycles. The

Abbreviation: VOO, virgin olive oil; HPU, high power ultrasounds; NMR, nuclear magnetic resonance; RSM, response surface methodology; Q, paste flow rate in the pipeline; W, high power ultrasounds intensity; OT, fruit temperature; OM, olive moisture; OF, olive fat content; IY, industrial yield; OE, oil extractability index.

resulting mechanical and shear forces help to increase mass transfer and can also break the cell walls (Ashokkumar, 2015). The power ultrasounds are applied in various areas in the food industry as surface cleaning, production of emulsion, acceleration of chemical reactions, extraction of aromas, marinating, drying and dehydration, microbial and enzyme inactivation, extraction of bioactive compounds, oil and protein extraction and some other applications (Chemat, Zill-e-Huma, & Khan, 2011; Mason et al., 1996; Patist & Bates, 2008).

First works on HPU application to the virgin olive oil extraction process were carried out by Jimenez et al. (2006) and consisted in the ultrasounds application, under discontinuous conditions, during the olive paste malaxation describing two major effects: a rapid heating of olive paste and the improvement of the oil extractability. However, the effect of HPU treatment on virgin olive oil quality parameters (free acidity value, peroxide value, K270 and K232) was not significant. Nevertheless higher concentration of tocopherols, chlorophylls and carotenoids were observed in the oils from ultrasound treatment, whereas lower levels of bitterness and polyphenols were obtained. Most recently Clodoveo, Durante, and La Notte (2013), reported two different HPU treatments: at kneading, as olive paste pretreatment, and to the whole olives before crushing during washing. For both treatments, a quick olive paste heating and higher oil extractability were described. Oil quality parameters were not affect as observed (Jimenez et al., 2007; limenez et al., 2006). All these previous experiments were applied in batch conditions, under non continuous process, and performed in open conditions with atmosphere contact.

The aim of this manuscript was studying and modeling the olive paste heating using HPU continuous conditioning, previous to malaxation, at laboratory scale. Response surface methodology was applied for optimization and prediction of the olive paste heating and how it was affected by the variables tested. Furthermore, the effect of HPU on process yield and VOO quality and composition was determined. This study is part of the previous works to the development of a HPU treatment device at pilot plant scale.

#### 2. Materials and methods

#### 2.1. Plant material

Fresh and healthy olives (*Olea europea* L.) of the olive cultivar '*Picual*' were harvested from trees grown under irrigation in the experimental olive orchard of IFAPA Centro Venta del Llano in Mengibar, Jaén. Four harvesting dates were used, from November to February, at different olive ripening stages. Olive fruit characteristics (moisture and fat content), maturity index (Beltran, Uceda, Jimenez, & Aguilera, 2003) and harvesting dates are shown in Table 1.

#### 2.1.1. Olive moisture

The olive fruit was crushed and the milled paste was desiccated at 105 °C after weighted, this two later operation was repeated until

Table	1	
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Effect of high power ultrasound	treatment on o	live paste	temperature.
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Date	Olive paste composition			$\Delta T^{a}$ (°C)	
	MI <sup>b</sup>	Fat (%)	Moisture (%)	Test	HPU
09/11	2.7	15.61	51.54	2.5	15.3
10/12	3.8	24.74	48.56	3.6	23.2
15/01	4.6	24.45	44.29	3.4	11.6
10/02	5.2	21.63	49.72	5.4	16

<sup>a</sup> ΔT temperature increase for treated and untreated paste.

<sup>b</sup> MI: Olive fruit maturity index.

obtain constant weight. The results were expressed in weight percentage.

#### 2.1.2. Olive fat content

Oil content was measured, for the dried olive paste obtained from the olive moisture determination, using a Nuclear Magnetic Resonance (NMR) fat analyzer Minispec mq 20 (Bruker Analytik Gmbh). The NMR was previously calibrated and validated with Soxhlet extractor. The results were expressed on weight percent (on a fresh matter basis).

#### 2.2. VOO extraction and HPU treatment apparatus

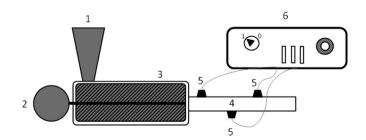
A laboratory scale device for HPU treatment was implemented as described in Fig. 1. This device was composed by three units: a rollers mill, a rectangular pipeline for olive paste transport equipped with three 40 KHz frequency piezoelectric transducers and a 150 W ultrasonic generator with an intensity regulator to power the transducers.

The olives were added in the roller mill and then flowed through the pipe until the output where the olive paste was collected for processing. The olive paste flow rate through the device was established by the olive fruit feed rate in the mill and measured for each conditions. After olive paste flowed through the device, it was taken at the output and VOO was extracted immediately at laboratory scale using the thermo-malaxer and the centrifuge of an Abencor system (MC2, Seville). Around 700–800 g of the olive paste were kneaded in the thermo-malaxer for the experimental conditions described for each experiment. After kneading, the olive paste was immediately centrifuged to separate oily must from solids. The liquid phases were recovered in a laboratory graduated cylinder and after settling, the volume of the oily phase was measured. The oil was filtered and stored at -24 °C until analyses.

#### 2.3. Experimental

#### 2.3.1. HPU treatment for olive paste heating

In order to test the HPU device ability for olive paste heating the experiments were carried as follows: the paste flowed through the pipeline when the piezoelectric transducers reached 60 °C using 100% of the generator intensity. The olive paste flow rate required to reach a paste temperature around 28 °C was comprised between 20 and 25 kg h<sup>-1</sup>. The variation of temperature between olive fruits and the paste (treated and untreated) was taken at the device output (Fig. 1). The temperatures were measured for the olive fruit before device entrance and the olive paste after treatment through the device (Fig. 1). For these experiments the olives harvested at the four harvesting dates were used.



**Fig. 1.** High Power Ultrasound device for olive paste continuous treatment composed of: (1) Olives reception hopper, (2) mill motor, (3) rollers mill, (4) rectangular pipeline, (5) piezoelectric transducers and (6) ultrasonic generator.

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