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Low-frequency, high-power ultrasound treatment at different pressures for olive paste: Effects on olive oil yield and quality

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ABSTRACT

Ultrasound technology was employed to test its action on the extraction of olive oil at the industrial scale. Because of its mechanical effects, ultrasound waves were applied to the olive paste, between the crushing and malaxing operations. Comparative experiments were performed between traditional extraction processes and the innovative extraction process, with the addition of the ultrasound treatment. Different levels of pressure were tested on olive paste, using four different olive cultivars. Pressure level played an important role in olive oil extractability. When ultrasound was subjected to olive paste with a pressure of about 3.5 bar, there was a significant increase of extractability compared to the traditional process. On the other hand, there was no significant effect between ultrasound treatment and traditional technology on extractability when ultrasound at a pressure level of 1.7 bar was used.

1. Introduction

Ultrasound technology is used in the food industry to develop various effective and reliable food processing applications. The most common applications in the food industry include cell destruction and extraction of intracellular material. Ultrasound (US) waves are mechanical waves having a frequency range of 20 kHz − 10 MHz. They produce mechanical effects that increase the modifying properties of certain solids and alter liquid–liquid and gas–liquid systems [1,2]. Further subdivisions within this range have been identified [3], and they have substantially different characteristics, depending on the frequency at which they are generated and on the amount of energy generated by the acoustic field [3]. In particular, high-energy US characterized by low frequencies (20 kHz–100 kHz) are implicated in the cavitation phenomena due to the mechanical effect of the waves. The influence of US (when the frequency approaches 20 kHz) is based on the formation, growth, and collapse of small bubbles in a liquid; this mechanism is called 'cavitation'. The bubbles then grow larger by absorbing the gas or vapour present in the medium over several cycles. The bubbles just formed then collapsed in the second stage of the cavitation cycle to release the energy absorbed and to give rise to mechanical effects such as local heat and high pressure [4–6]. The resulting stresses and the repeated collapse of the bubbles bring about damage to the walls and surfaces of the particles [7]. Although cavitation is considered an event to be avoided in many fields, in other specific fields, such as the olive oil extraction process, it can be useful.

Recently, an extended frequency range of US, from 400 kHz to 2 MHz, has been tested in different applications, such as for the improvement of oil recovery and milk fat creaming acceleration [8] or for palm oil extraction in a semi-industrial plant [9].

In recent years, US technology has been applied to the olive oil extraction process due to its positive effects on the yield and quality of the olive oil. Virgin olive oil extraction consists of five main operations: (i) fruit cleaning; (ii) preparation and conditioning of the paste; (iii) separation of the solid and liquid phases; (iv) separation of the liquid phases; and (v) olive oil storage [10–15].

The effect of low-frequency US in an olive oil extraction process was reported in different papers such as Jimenez et al. [16], and Bejaoui et al. [17], where the authors found positive effects on the extraction process.

In research by Taticchi et al. [18], a high-power US device was introduced in an industrial plant. Research has shown that the sonication of olive pastes led to a significant increase in extraction yield and to the enhancement of phenol content, compared to the oil extracted using a traditional process at an early maturity index.

Relying on these perspectives, over the past few years, high frequency US (> 1 MHz) was used in the olive oil extraction process. The results showed that the chemical and physical effects related to cavitation were minimal, while the effect of acoustic flow was predominant; high frequency

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separation was based on the principle of displacing suspended particles or droplets exposed to an ultrasonic standing wave field [19–22].

As with all other more innovative processing technologies, US wave treatments (both with low and high frequencies) is not a standard technology and therefore must be studied and developed for each type of application before its commercial use. In the olive oil extraction process, the application of US improved the process, in particular helping the malaxation operation and exploiting the positive effects of this treatment (i.e. mechanical and/or chemical and physical effects on the olive paste matrix). The effects of this technology are considered interesting in the olive oil industry, mainly due to their implications to improve the qualitative characteristics and extractability of extra virgin olive oil. However, the interaction of the acoustic energy with a matrix (i.e. olive paste) mainly occurs though a liquid medium; if the implosion of bubbles creates an unusual substrate for chemical reactions implicate on the quality characteristics of olive oil, at the solid and liquid interface, even the water jet formed by transient cavitation might contribute to changes in the rheological properties with positive feedback on quantitative characteristics of the food process, i.e., the olive oil extraction yield.

Consequently, a parameter that could certainly have a strong impact on the effect of US in the olive oil extraction process is the pressure level generated in the US cell. No study has been carried out to examine this interaction.

To this end, a low-frequency ultrasonic device was inserted in an industrial plant for the extraction of oil between a fringe and a kneading machine. Two levels of pressure on olive paste in the US-cell were studied using four different olive varieties.

Finally, the extractability and the quality parameters of the resulting olive oil were evaluated.

2. Materials and methods

2.1. Low-frequency, high-power US machine

The US machine employed for the experimental tests was manufactured by Hielscher Gmbh (Teltow, Germany) and installed by Seneco Science (Seneco s.r.l., Milano, Italy). It was composed of a 4 kW power supply, an US generator working at 20 kHz, and an US probe (Cascatrode™, Hielscher Gmbh). All functions were controlled by a PLC equipped with a touch screen through which it was possible to set the amplitude value, between 0 and 100%, corresponding to 18–35 µm. The machine was connected between the crusher and malaxer through DN90 connections. The Cascatrode™ was placed in a vertical stainless-steel tube (cell). Olive paste flowed into the cell coming from the side top and exited at the side bottom (Fig. 1). A pneumatic valve was installed on the outlet of the US machine to set the pressure in the cell. A pressure probe was also installed on the output section of the US cell, to monitor the olive paste pressure every second. Data were recorded on a SD-card installed in the PLC. The pressure value determined the US effect on olive paste, in terms of energy transferred. In fact, the higher the pressure value, the more electric power was adsorbed by the machine, because the US frequency must be constant and to do this, the generator had to spend more energy in case the olive paste required more mechanical resistance due to the higher pressure inside the cell. All parameters (electric power, pressure, amplitude and pulsation frequency) were visible in real-time on the display of the PLC.

Considering the chosen frequency and amplitude parameters, the system was able to automatically modulate the electric power used as a function of the pressure detected in the US-cell.

2.2. Plant for olive oil extraction

Experimental tests were carried out in a commercial olive mill (Pietro Leone&Figli, s.n.c., Puglia, Italy). The mill was composed of a defoliator, a washing machine, a hammer crusher (mod. Hammer Mill Crusher; Alfa Laval Corporate AB, Lund, Sweden) with grid hole of

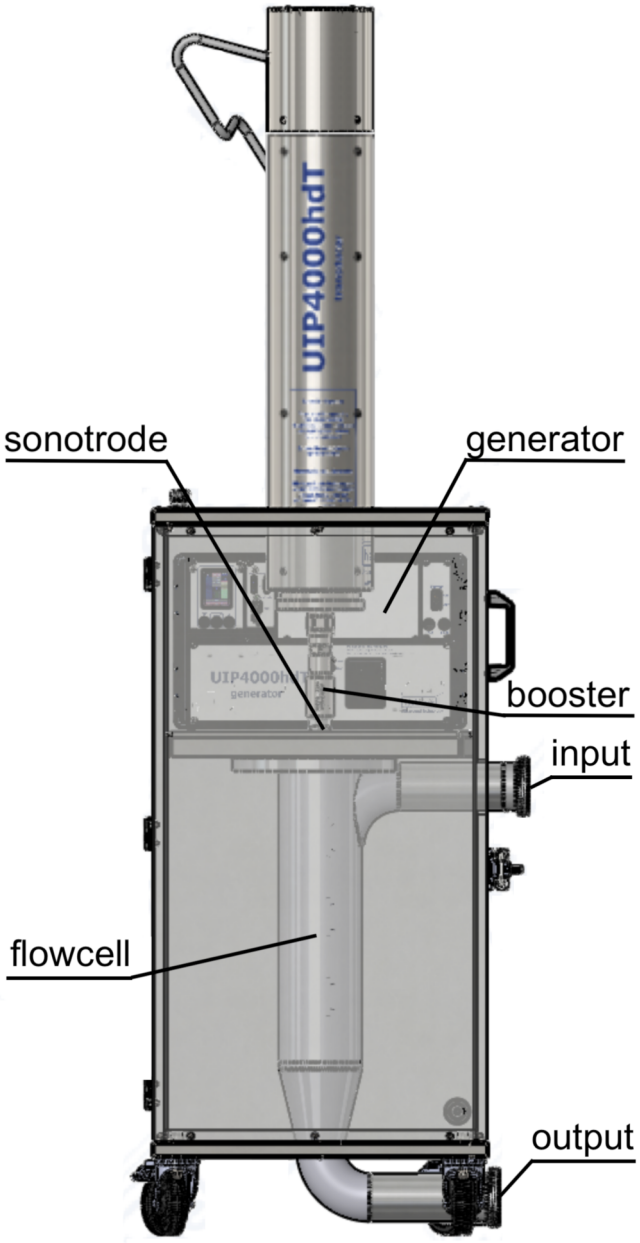

Fig. 1. Ultrasound machine used for experimental tests.

7 mm, a group of six malaxer machines arranged in parallel way, a decanter (mod. NX X32; Alfa Laval Corporate AB) and two vertical plate centrifuges (mod. UVPX 507; Alfa Laval Corporate AB). The decanter was set for a 3-phase way with 10% water added. The mass flow rate was set to 2.3 tonnes h^{-1} for all experiments. For all tests, the malaxation was performed for 30′ at 27 ± 0.5 °C.

The US machine was installed in the mill between the hammer crusher and the malaxer machine as reported in Fig. 2. By turning the US machine ON or OFF, it was possible to have two different extraction system configurations, a conventional extraction plant (US machine OFF) and an US extraction plant (US machine ON).

2.3. Experimental plan

Experimental tests were performed using olives of the Arbequina, Peranzana, Nocellara del Belice and Coratina cultivars. Olives were mechanically harvested in Foggia and processed within 6 h in order to carry out the tests. For each olive cultivar, five comparative tests

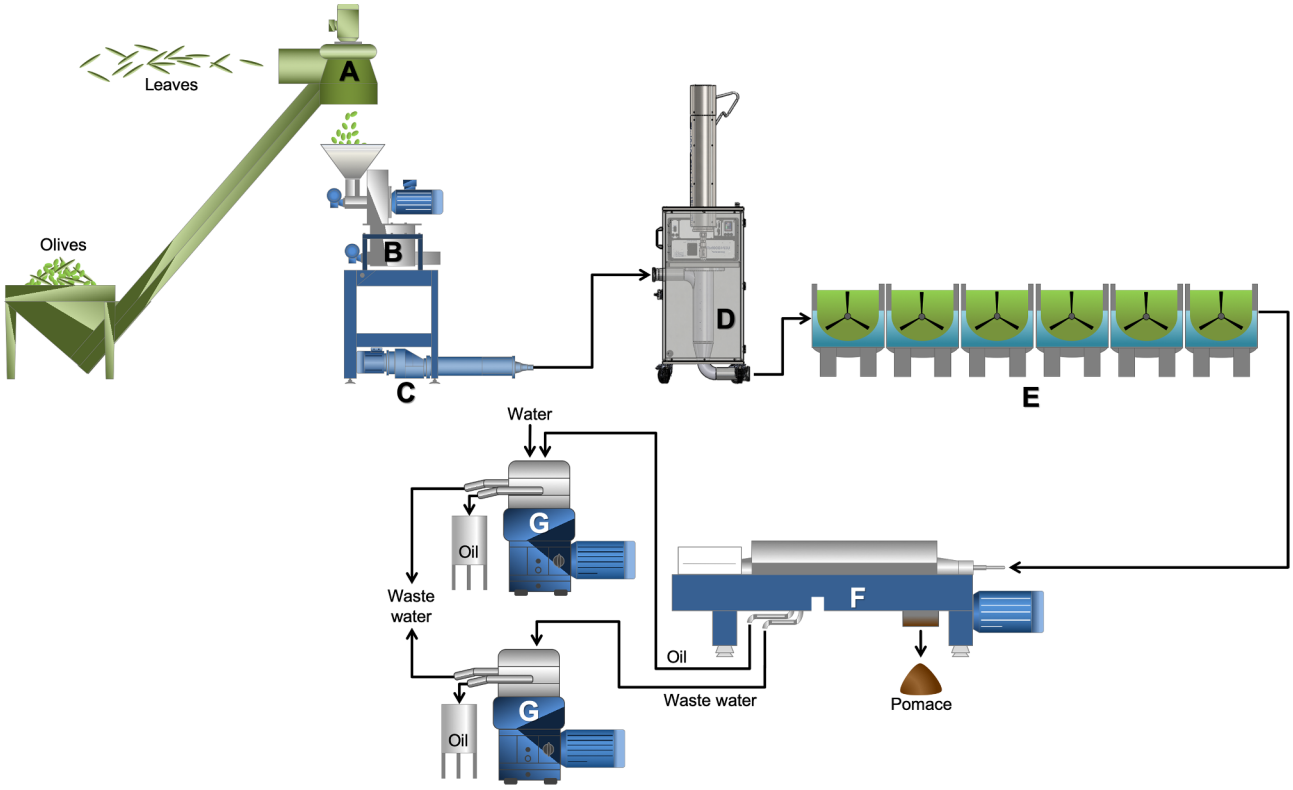

Fig. 2. Scheme of olive oil extraction line: A. cleaning section; B. crusher; C. cavity pump; D. US machine; E. 6-malaxer section; F. horizontal centrifuge; G. vertical centrifuges.

Table 1
Quantitative results and process parameters.

	Test conditions	Variety	Ultrasound power	Mean specific energy transferred	Pressure in US-cell	POMACE		Extractability
			(kW)	(kJ kg^{-1})	(Bar)	Moisture (%)	Oil (%. db)	(%)
1st trial	Control	Arbequina	–	–	–	56.0 ± 0.6 *a*	10.4 ± 0.8 *a*	83.5 ± 1.3 *a*
	US 1.7 bar	Arbequina	2.64 ± 0.26	4.13	1.7 ± 0.04	56.2 ± 0.6 *a*	10.7 ± 0.9 *a*	84.2 ± 0.3 *a*
2nd trial	Control	Peranzana	–	–	–	55.3 ± 0.8 *a*	5.6 ± 0.3 *a*	83.0 ± 0.6 *a*
	US 1.7 bar	Peranzana	2.61 ± 0.21	4.09	1.7 ± 0.02	55.6 ± 0.6 *a*	5.5 ± 0.7*b*	83.1 ± 0.8 *a*
3rd trial	Control	Nocellara	–	–	–	56.0 ± 0.5 *ab*	7.4 ± 0.7 *a*	82.7 ± 1.5*b*
	US 1.7 bar	Nocellara	2.63 ± 0.25*b*	4.12	1.7 ± 0.03*b*	55.5 ± 0.3*b*	7.4 ± 0.8 *a*	83.0 ± 1.1*b*
	US 3.5 bar	Nocellara	3.52 ± 0.38 *a*	5.51	3.5 ± 0.03 *a*	56.5 ± 0.3 *a*	5.6 ± 0.6*b*	87.1 ± 1.3 *a*
4th trial	Control	Coratina	–	–	–	56.3 ± 1.0 *a*	8.4 ± 0.5 *a*	82.3 ± 0.6*b*
	US 3.5 bar	Coratina	3.54 ± 0.41	5.54	3.5 ± 0.02	56.4 ± 1.2 *a*	6.2 ± 1.4*b*	86.9 ± 2.2 *a*

Different letters in columns denote significant statistical differences at $p < 0.05$ (Tuckey's test).

Table 2
Standard virgin olive oil parameters specified by the IOC (2017).

Test conditions		Variety	Free acidity (%)	Peroxide value (meq O_2 kg^{-1})	K232	K270	ΔK
Legal limits for EVOO			< 0.8	≤20	≤2.50	≤0.22	≤0.01
1st trial	Control	Arbequina	0.56 ± 0.06 *a*	7.57 ± 0.31 a	1.65 ± 0.05 a	0.11 ± 0.01 a	0.002 ± 0.002 a
	US 1.7 bar	Arbequina	0.54 ± 0.01 *a*	7.40 ± 0.10 a	1.63 ± 0.03 a	0.11 ± 0.01 a	0.003 ± 0.002 a
2nd trial	Control	Peranzana	0.44 ± 0.01 *a*	6.47 ± 0.21 a	2.05 ± 0.07 a	0.17 ± 0.00 a	0.006 ± 0.002 a
	US 1.7 bar	Peranzana	0.47 ± 0.02 *a*	6.63 ± 0.06 a	2.05 ± 0.03 a	0.17 ± 0.01 a	0.004 ± 0.003 a
3rd trial	Control	Nocellara	0.57 ± 0.01 *a*	7.53 ± 0.01 a	2.21 ± 0.07 a	0.15 ± 0.01 a	0.002 ± 0.010 a
	US 1.7 bar	Nocellara	0.57 ± 0.01 *a*	7.53 ± 0.25 a	2.16 ± 0.05 a	0.15 ± 0.02 a	0.003 ± 0.001 a
	US 3.5 bar	Nocellara	0.57 ± 0.02 *a*	7.27 ± 0.21 a	2.19 ± 0.07 a	0.15 ± 0.02 a	0.003 ± 0.001 a
4th trial	Control	Coratina	0.23 ± 0.01 *a*	6.67 ± 0.25 a	2.08 ± 0.03 a	0.17 ± 0.01 a	0.000 ± 0.001 a
	US 3.5 bar	Coratina	0.25 ± 0.01 *a*	6.70 ± 0.26 a	2.11 ± 0.09 a	0.16 ± 0.01 a	0.001 ± 0.001 a

Different letters in column, for each test conditions, denotes significative statistical differences among means, at *p*-level < 0.05 (Tuckey's test).

Table 3
Phenolic composition of EVOOs. Data expressed as mg kg^{-1}.

	Arbequina					Peranzana					Nocellara del Belice					Coratina				
	Control		US 1.7 bar			Control		US 1.7 bar			Control		US 1.7 bar		US 3.5 bar	Control		US 3.5 bar		
3,4-DHPEAa	0.4	±	0.1*a*	0.1	±	0.0*b*	0.7	±	0.1*a*	2.7	±	3.2*a*	2.3	±	0.7*ab*	1.3	±	0.4*b*	2.8	±	0.0*a*	1.2	±	0.0*a*	0.9	±	0.3*a*
p-HPEA	3.2	±	2.3*a*	1.9	±	0.7*a*	0.9	±	0.2*a*	2.4	±	2.3*a*	1.5	±	0.4*a*	0.9	±	0.1*b*	1.9	±	0.0*a*	2.2	±	0.2*a*	1.6	±	0.2*b*
Vanilic acid	0.2	±	0.1*a*	0.3	±	0.0*a*	0.1	±	0.0*a*	0.1	±	0.1*a*	0.1	±	0.1*a*	0.0	±	0.0*a*	0.0	±	0.0*a*	0.1	±	0.0*a*	0.1	±	0.0*a*
3,4-DHPEA-EDA	4.0	±	1.3*a*	3.9	±	2.9*a*	175.1	±	17.7*a*	173.2	±	23.1*a*	156.4	±	43.7*b*	163.6	±	13.7*b*	201.7	±	4.6*a*	415.4	±	7.7*a*	467.2	±	37.1*a*
p-HPEA-EDA	5.8	±	0.9*a*	5.5	±	1.7*a*	36.4	±	2.1*a*	36.2	±	1.7*a*	47.9	±	20.7*ab*	40.2	±	9.1*b*	66.5	±	1.1*a*	132.1	±	12.2*b*	156.8	±	5.8*a*
(+)-1-acetoxypinoresinol	25.6	±	5.3*a*	26.4	±	7.3*a*	6.5	±	1.5*a*	5.9	±	0.4*a*	3.7	±	0.3*a*	3.4	±	0.3*a*	3.4	±	0.0*a*	21.3	±	0.5*a*	20.1	±	0.1*b*
(+)-pinoresinol	4.7	±	0.5*a*	4.3	±	1.1*a*	8.4	±	0.8*a*	8.9	±	0.3*a*	7.1	±	0.8*a*	7.3	±	0.0*a*	7.4	±	0.1*a*	14.0	±	0.2*b*	14.6	±	1.1*a*
3,4-DHPEA-EA	0.0	±	0.0*a*	0.0	±	0.0*a*	14.5	±	2.9*a*	15.9	±	3.3*a*	60.5	±	10.8*b*	59.0	±	8.5*b*	71.7	±	1.0*a*	141.8	±	14.6*a*	137.3	±	15.4*a*
Ligstroside aglycone	1.6	±	0.2*a*	1.3	±	0.4*a*	4.0	±	0.1*a*	4.1	±	0.1*a*	4.1	±	2.3*ab*	3.9	±	0.4*b*	6.2	±	0.1*a*	21.2	±	3.5*a*	24.6	±	5.5*a*
Total phenols	45.4	±	6.0*a*	43.7	±	8.1*a*	246.6	±	18.1*a*	249.4	±	23.7*a*	283.4	±	49.6*b*	279.6	±	18.5*b*	361.6	±	5.0*a*	749.2	±	20.8*b*	823.1	±	40.9*a*

Different letters in rows denote significant statistical differences at $p < 0.05$ (Tuckey's test).

between the conventional and US plant were made. During the tests, the US frequency was 20 kHz and the amplitude chosen was 100%. In addition, the effect of olive paste pressure in the US cell was evaluated. The tests carried out with Arbequina and Peranzana olives were conducted by setting the pneumatic valve of the US machine at about 1.7 bar. For the Nocellara del Belice cultivar two levels of pressure, 1.7 and 3.5 bar, were used. For the Coratina Cultivar, only 3.5 bar was used. Every run was tracked by labelling each sample with the date and time when the US treatment began and ended.

2.4. Extractability and oil content in the olives, pomace and wastewater

The oil extractability parameters of the oil present in pomace and wastewater were used to evaluate the quantitative performance of the oil extraction plant.

The oil extractability (E) is the ratio between the percentage of oil extracted from the olives (P_{oe}) by the plant with respect to the percentage of the oil content in the olives (P_{oo}).

The E was calculated using the following equation:

$$E = \frac{Poe}{Poo} * 100 \quad (1)$$

Oil content in olives, pomace, and wastewater was evaluated according to Leone et al. [20].

2.5. Olive oil quality

2.5.1. Legal extra virgin olive oil quality parameters

The free acidity, peroxide value and UV absorption characteristics (K_{232}, K_{270} and ΔK) were determined according to the analysis methods described by Regulation (EU) 2015/1830 [30].

2.5.2. Phenolic compounds

The EVOO phenolic extracts were subjected to analysis after a liquid–liquid extraction using a methanol/water solution (80/20 v/v) as described by Selvaggini et al. [23]. The phenolic compounds were detected with an Agilent Technologies system Mod. 1100, composed of: vacuum degasser, quaternary pump, autosampler, thermostated column compartment and detectors [diode array detector (DAD) and fluorescence detector (FLD)]. A C18 column, Spherisorb ODS-1 (250 mm X 4.6 mm), with a particle size of 5 mm (Phase Separation Ltd., Deeside, UK) was used. The quantitative and qualitative evaluation of the hydrophilic phenols was carried out following the method described by Selvaggini et al. [24], the aglyconic derivatives of secoiridoid [the dialdehydic forms of decarboxymethyl elenolic acid linked to hydroxytyrosol (3,4-DHPEA-EDA or oleacein) and to tyrosol (p-HPEA-EDA or oleocanthal), the 3,4-(dihydrox-yphenyl)ethanol elenolic acid (3,4-DHPEA-EA or isomer of the oleuropein aglycon), the p-(hydroxyphenyl)ethanol elenolic acid (p-HPEA-EA or ligstroside aglycon)] and the phenolic alcohols [(3,4-(dihydroxyphenyl)ethanol (3,4-DHPEA or hydroxytyrosol) and p-(hydroxyphenyl)ethanol (p-HPEA or tyrosol)] were analyzed at 278 nm (DAD), whereas the lignans [(+)-1-acetoxypinoresinol and (+)-pinoresinol] at 280 nm ex. and 339 nm em., using the FLD. All the data were expressed as mg of phenols kg-1 of EVOO.

2.5.3. Volatile compounds

The main volatile compounds responsible of the positive sensory note of EVOO, belonging to the chemical classes of C5 and C6 saturated and unsaturated aldehydes and alcohols, and esters, were evaluated using a Varian 4000 GC–MS equipped with a 1079 split/splitless injector (Varian, Walnut Creek, CA) was used. A fused silica capillary column was employed (DB-Wax-ETR, 50 m, 0.32 mm ID, 1 lm film thickness; J&W Scientific, Folsom, CA). The headspace solid-phase microextraction, followed by gas chromatography–mass spectrometry (HS-SPME/GC–MS) was performed as described by Veneziani et al.

Table 4
Volatile compounds detected in EVOOs. Data expressed as μg kg^{-1}.

	Arbequina								Peranzana								Nocellara del Belice
	Control				US 1.7 bar				Control				US 1.7 bar				Control
Aldehydes																	
Pentanal	0.0	±	0.0	*a*	0.0	±	0.0	*a*	42.3	±	4.6	*b*	60.3	±	11.0	*a*	62.4
(*E*)-2-Pentenal	51.8	±	1.8	*a*	29.3	±	11.0	*b*	57.0	±	12.7	*a*	55.0	±	9.2	*a*	41.5
Hexanal	845.8	±	82.4	*b*	1173.0	±	156.3	*a*	716.5	±	117.4	*a*	1086.5	±	485.1	*a*	568.8
(*E*)-2-Hexenal	14487.5	±	1495.5	*a*	15109.0	±	2035.1	*a*	30015.0	±	721.2	*a*	29280.0	±	1888.0	*a*	7359.4
(*E,E*)-2,4-Hexadienal	139.3	±	4.6	*a*	111.3	±	16.6	*b*	311.0	±	14.8	*a*	242.0	±	46.0	*b*	198.9
2,4-Hexadienal (i)	329.0	±	104.7	*a*	190.8	±	13.1	*b*	613.3	±	36.4	*a*	498.0	±	94.0	*b*	406.9
Σ aldehydes	15853.3	±	1501.5	*a*	16613.3	±	2041.2	*a*	31755.0	±	731.9	*a*	31221.8	±	1952.2	*a*	8637.8
Alcohols																	
1-Pentanol	67.0	±	6.4	*a*	70.5	±	7.8	*a*	17.8	±	4.6	*a*	46.3	±	37.1	*a*	31.6
1-Penten-3-ol	186.3	±	3.2	*a*	183.3	±	3.9	*a*	260.8	±	69.7	*a*	323.5	±	9.9	*a*	233.0
(*E*)-2-Penten-1-ol	37.3	±	8.8	*a*	33.3	±	3.9	*a*	243.5	±	115.3	*a*	361.8	±	5.3	*a*	102.1
(*Z*)-2-Penten-1-ol	158.3	±	28.6	*a*	93.8	±	29.3	*b*	255.0	±	79.2	*a*	324.3	±	0.4	*a*	212.0
1-Hexanol	3158.0	±	84.1	*a*	2728.8	±	491.1	*a*	714.5	±	27.6	*a*	735.8	±	35.7	*a*	1354.6
(*E*)-2-Hexen-1-ol	2959.0	±	763.7	*a*	2503.5	±	244.7	*a*	635.0	±	190.9	*a*	680.5	±	91.9	*a*	1164.8
(*Z*)-2-Hexen-1-ol	92.3	±	1.8	*a*	70.0	±	7.1	*b*	0.0	±	0.0	*a*	0.0	±	0.0	*a*	0.0
(*E*)-3-Hexen-1-ol	31.0	±	1.4	*a*	26.8	±	6.0	*a*	18.8	±	1.1	*a*	29.8	±	11.7	*a*	36.3
(*Z*)-3-Hexen-1-ol	1527.8	±	283.2	*a*	1422.5	±	150.6	*a*	1361.3	±	80.3	*a*	965.8	±	76.7	*b*	2893.6
Σ alcohols	8216.8	±	819.4	*a*	7132.3	±	569.9	*a*	3506.5	±	260.9	*a*	3467.5	±	131.3	*a*	6028.0
Esters																	
Hexyl acetate	155.3	±	25.8	*a*	219.3	±	42.8	*a*	1601.3	±	58.3	*a*	1665.8	±	61.9	*a*	1131.4
(*Z*)-3-Hexenyl acetate	411.0	±	15.6	*b*	530.8	±	78.8	*a*	3275.5	±	97.6	*a*	3375.8	±	34.3	*a*	4156.5
(*E*)-2-Hexenyl acetate	24.3	±	1.8	*a*	25.8	±	8.1	*a*	32.5	±	18.4	*a*	41.8	±	6.7	*a*	74.6
Σ esters	590.5	±	30.2	*b*	775.8	±	90.1	*a*	4909.3	±	115.2	*a*	5083.3	±	71.1	*a*	5362.5

	Nocellara del Belice										Coratina									
	Control				US 1.7 bar				US 3.5 bar				Control				US 3.5 bar			
Aldehydes																				
Pentanal	±	12.1	*b*		70.0	±	0.7	*b*	271.0	±	96.2	*a*	50.0	±	19.8	*a*	31.0	±	43.8	*a*
(*E*)-2-Pentenal	±	7.4	*b*		32.3	±	0.4	*c*	21.5	±	2.1	*a*	71.5	±	2.8	*a*	48.0	±	24.7	*a*
Hexanal	±	187.1	*c*		726.5	±	10.6	*b*	1512.5	±	540.9	*a*	572.3	±	44.9	*a*	572.5	±	70.7	*a*
(*E*)-2-Hexenal	±	2104.0	*ab*		7645.5	±	152.7	*b*	8742.5	±	167.6	*a*	46360.0	±	629.3	*a*	46617.5	±	625.8	*a*
(*E,E*)-2,4-Hexadienal	±	111.0	*ab*		291.0	±	6.4	*a*	118.0	±	2.8	*b*	271.8	±	13.8	*a*	205.0	±	37.5	*b*
2,4-Hexadienal (i)	±	215.9	*ab*		591.0	±	12.0	*a*	252.5	±	6.4	*b*	476.0	±	25.5	*a*	359.0	±	67.9	*b*
Σ aldehydes	±	2126.2	*ab*		9356.3	±	153.7	*b*	10918.0	±	574.5	*a*	47801.5	±	631.9	*a*	47833.0	±	636.5	*a*
Alcohols																				
1-Pentanol	±	12.2	*b*		27.3	±	0.4	*b*	135.0	±	26.9	*a*	13.0	±	1.4	*a*	31.8	±	26.5	*a*
1-Penten-3-ol	±	43.8	*a*		257.0	±	17.0	*a*	262.0	±	18.4	*a*	522.5	±	0.7	*a*	367.8	±	133.3	*b*
(*E*)-2-Penten-1-ol	±	29.2	*b*		111.3	±	2.5	*b*	127.5	±	0.7	*a*	195.0	±	12.7	*a*	150.0	±	9.9	*b*
(*Z*)-2-Penten-1-ol	±	25.2	*a*		195.0	±	7.8	*a*	180.0	±	1.4	*b*	546.8	±	15.9	*a*	341.5	±	173.9	*b*
1-Hexanol	±	340.2	*a*		1210.5	±	25.5	*a*	901.0	±	2.8	*b*	390.3	±	18.0	*a*	394.3	±	8.1	*a*
(*E*)-2-Hexen-1-ol	±	233.7	*a*		832.5	±	0.7	*b*	903.0	±	80.4	*a*	1026.5	±	75.7	*a*	1073.0	±	237.6	*a*
(*Z*)-2-Hexen-1-ol	±	0.0	*a*		0.0	±	0.0	*a*	0.0	±	0.0	*a*	0.0	±	0.0	*a*	0.0	±	0.0	*a*
(*E*)-3-Hexen-1-ol	±	21.7	*a*		56.0	±	0.0	*a*	29.5	±	3.5	*a*	8.5	±	0.7	*a*	4.3	±	6.0	*a*
(*Z*)-3-Hexen-1-ol	±	1581.8	*a*		3846.8	±	78.8	*a*	811.0	±	110.3	*b*	720.5	±	15.6	*a*	581.5	±	188.8	*a*
Σ alcohols	±	1635.9	*a*		6536.3	±	85.0	*a*	3349.0	±	1103.7	*a*	3423.0	±	81.9	*a*	2944.0	±	375.5	*b*

(*continued on next page*)

Table 4 (*continued*)

	Nocellara del Belice								Coratina							
	Control			US 1.7 bar			US 3.5 bar			Control			US 3.5 bar			
Esters																
Hexyl acetate	±	73.3	*b*	1954.5	±	21.2	*a*	966.0	±	48.1	*c*	46.5	±	16.3	*a*	52.0	±	17.0	*a*
(*Z*)-3-Hexenyl acetate	±	565.2	*b*	5237.5	±	173.2	*a*	4899.5	±	361.3	*ab*	118.0	±	50.2	*a*	144.5	±	14.8	*a*
(*E*)-2-Hexenyl acetate	±	43.2	*ab*	61.5	±	0.7	*b*	114.5	±	0.7	*a*	0.0	±	0.0	*a*	0.0	±	0.0	*a*
Σ esters	±	571.5	*b*	7253.5	±	174.5	*a*	5980.0	±	364.5	*b*	164.5	±	52.8	*a*	196.5	±	22.5	*a*

Different letters in rows denotes significant statistical differences at $p < 0.05$ (Tuckey's test).

[25]. The values of the peak areas were determined on the basis of the relative calibration curve for each molecule and the data were expressed in μg of volatile compound kg^{-1} of EVOO.

2.6. Data processing

Pressure and energy data collected during the trials involving the US machine were processed by the signal processing toolbox of MATLAB (The Mathworks inc., Natick – MA, USA). The processing concerned the elimination of high-frequency noise using a median filter having a bandwidth of 10 s. Since the sampling rate was one read per second, the median filter excluded all signal noise falling in the bandwidth. For each trial involving different olive cultivars, the mean trends of pressure were obtained. The significance test was performed using ANOVA test and Tuckey test for the means separation, at $p < 0.05$.

3. Results and discussion

3.1. Effect of ultrasound treatment on extractability

Quantitative results are reported in Table 1. As shown, trials involving US treatment at low pressure (1.7 bar) led to the same extractability value as the control tests. On the contrary, trials where high pressure was used (3.5 bar), showed higher extractability values for US treatments compared to controls. US treatment on olive pastes did not have significant effects on extractability when the operating pressure was approximately 1.7 bar. As indicated in Table 1, the extractability value was not significantly different from the control test and the US test when Arbequina, Peranzana, and Nocellara del Belice were processed using 1.7 bar pressure. Because of these results, we decided to switch the pressure to 3.5 bar. In the latter condition, extractability was significantly higher than the control test. These data demonstrate the effect of US treatment on processing pressure. The increase in extractability was probably due to the major mechanical impact of the Cascatrode on the olive paste. This is also apparent when comparing the amount of energy power employed in the two different pressure conditions. A mean value of 2.7 and 3.4 kW was registered when 1.7 bar and 3.5 bar, respectively were used (see Table 1).

3.2. Olive oil quality

The evaluation of legal quality parameters (free acidity, peroxide values, K232, K270 and ΔK) (see Table 2) did not determine any significant variations as a consequence of the ultrasound treatment according to previous studies [17,18,26]. All the oils extracted from four different cultivar showed values into the limits established by the European Union Commission delegated regulation for the classification of an EVOO [30].

Table 3 showed a positive impact of the ultrasound treatment on the phenolic fraction of EVOO in function of the process pressure. In fact, the enhancement of phenolic compounds was detected only for the trials carried out with 3.5 bar of pressure in the US-cell during the oil extraction process. The two tests, on Nocellara del Belice and Coratina cvs, showed an increase of 24.4% and 9.8%, respectively. On the contrary, the analysis of phenolic concentration of the other trials, that have operated at lower pressure (1.7 bar), did not determined any significant differences compared with the related control test for Arbequina and Peranzana cvs, whereas, the Nocellara del Belice cvs. The data confirmed that at higher pressure (about 3–3.5 bar) the ultrasound system was able to determine a more efficient disruption activity on membranes and wall cells of olive tissues with a consequent release of intracellular matter into the water phase that allows to an improvement of the diffusion process of bioactive molecules into the oily phase, increasing the phenolic concentration [18,27].

The effect of ultrasound treatment on the single molecules belonging to the different classes of volatile compounds of EVOO seem to

be cultivar dependent, with probably different impact of the acoustic cavitation process on the different enzymes of the LOX pathways of different genetic origin and involved in the formation of aldehydes, alcohols and esters [28,29]. However, on the whole, the treatment at both pressure tested did not modify the sum of C_5 and C_6 saturated and unsaturated aldehydes and alcohols, and esters with the exception of a light variability of the sum of esters in Arbequina and Nocellara del Belice EVOOs and of the sum of alcohols and aldehydes in Coratina and Nocellara del Belice EVOOs, respectively (Table 4).

4. Conclusions

The experiments highlighted the effects of US mechanical waves on olive paste, as a way to improve the performance of the extraction process, increasing the extractability of the plant. US waves were applied to olive paste through a Cascatrode™, which was in contact with olive paste in a vertical tube. The efficiency of treatment was evaluated by considering the pressure on the olive paste in the ultrasound cell. As indicated, the pressure had an important impact on ultrasound treatment. In fact, increases of extractability were registered when olive paste was treated at 3.5 bar. These results have to be considered when taking into account the short treatment time, considering the olive paste only went through the US-cell once, at a mass flow rate of 2 tonnes h^{-1}.

The ultrasound technology did not determine any alterations to the main legal quality parameters and showed a positive impact to the phenolic composition of EVOO but only when the system operated at highest pressure of 3.5 bar, confirming the importance of the regulation of process pressure to improve the performance of the ultrasound treatment in the breakdown of olive cells and the release of intracellular content. The data of the sum of the main compounds responsible of EVOO sensory notes were not significantly modified by the ultrasound treatment compared to the respective control test, with some limited exceptions. The variability of the amount of single volatile compounds, did not show a common evolution trend, highlighting a high effect in relation to the different genetic origin of olives and to the adjustment of the operative parameters of ultrasonic device. Finally, studies on the investment and management costs of the ultrasound application in a mill could be important for an overall assessment.

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