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Combined industrial olive oil extraction plant using ultrasounds, microwave, and heat exchange: Impact on olive oil quality and yield

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ABSTRACT

In this study, an industrial combined plant (ICP) constructed from a low-frequency ultrasound device, microwave apparatus and heat exchanger is employed to investigate the real possibility of introducing these innovative technologies to the olive oil extraction process and evaluating their influence on olive oil quality and yield. The novelty of this study lies in the simultaneous use of these three technologies to condition the olive paste in a real olive oil extraction plant.

Different olive paste treatments were compared in order to define the effects on the olive oil quality and yield.

The use of a spiral heat exchanger in addition to the malaxer reduced the malaxation time to 20 min, and with the microwave apparatus it was possible to obtain an entirely continuous process, without interruptions, from the milling phase to the solid-liquid separation phase.

The internal spiral aids in moving the paste from the input to output section, resulting in limited operating pressure.

Using the ICP device led to an average increase in extractability ranging from 2.30 to 3.85% with respect to the control thesis, for the Arbosana and Arbequina varieties, respectively, but this difference was not statistically significant.

Regarding the virgin olive oil (VOO) quality, the use of the ICP did not affect the marketable parameters and total phenol content, while in terms of the process efficiency, the ICP obtained a higher value than the conventional process and improved the extraction yield.

1. Introduction

Ultrasound, microwave and heat exchangers are used for food processing in a variety of industrial and everyday applications. Increasing the thermal performance of virgin olive oil (VOO) plants, saving on process time and improving the olive oil efficiency and quality have become research goals in recent years. For this reason, researchers and private companies have conducted numerous studies to increase olive oil plant performance (Jiménez and Beltran, 2007; Juliano et al., 2013; Almeida et al., 2016; Bejaoui et al., 2015, 2016b, 2017; Chemat et al., 2017; Iqdiam et al., 2017; Leone et al., 2016, 2017, 2018; Juliano et al., 2017a,b).

The application of ultrasound treatment in olive oil extraction plants has been studied for the past 10 years, particularly in terms of aiding malaxation operations. Within this time, the application of low-frequency ultrasound (that is, 20–80 kHz) in olive oil extraction plants in order to condition the olive paste has been also investigated, using mainly laboratory-scale equipment and research conducted in full-scale plants (Jiménez and Beltran, 2007; Almeida et al., 2016). The impact on the olive oil quality and yield has been the main subject of investigation (Bejaoui et al., 2015, 2016a, 2017). The latest studies have been highlighted the positive impact of high-power ultrasounds on the oil extraction yield and extractability. These parameters resulted in higher than conventional malaxation (Bejaoui et al., 2016b) and did not affect the fatty acid and phenolic composition; however, the green sensorial attribute was increased significantly (Bejaoui et al., 2018).

In recent years, the impact of high-frequency ultrasound standing waves (megasonics) (that is, 300 to 400 or 800 kHz), applied before and/or after the malaxation process or by combining low- and high-frequency ultrasound, on olive oil extractability has also been studied

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(Leone et al., 2017; Juliano et al., 2017a,b). These studies demonstrate the enhancement of olive oil separation, thereby increasing the extraction yield. Several of these researches have also highlighted the instantaneous and homogeneous heating of olive paste, under continuous conditions, compare to traditional malaxation. Process time saving has been demonstrated by means of applying an industrial microwave prototype to olive oil extraction equipment, with significant potential to become a real alternative technique to continuous conditioning of the olive paste (Leong et al., 2015, 2017), also in combination with megasounds (Leone et al., 2018). Tamborrino et al. (2014) highlighted the higher concentration of volatile compounds, without compromising the high olive oil quality, when using a continuous microwave-assisted system for paste malaxation.

Regarding the application of heat exchange to the olive oil extraction process, the literature has reported one of the first researches conducted by Amirante in 2006, in which a positive influence on improving the malaxation efficiency was determined by introducing a heat exchanger between the crusher and malaxer. In recent years, the introduction of tubular heat exchangers has been studied experimentally during the olive oil extraction process, in order to evaluate its influence on the process rapidity of as well as the yield and olive oil quality (Esposto et al., 2013; Leong et al., 2015; Veneziani et al., 2015). In fact, the use of a heat exchanger plays an important role in transferring heat between the fluids circulating inside the pipes, thereby reducing the process time (Veneziani et al., 2015). The same study demonstrated a positive influence on the olive oil phenolic concentrations and volatile compounds. Thereafter, the introduction of a tubular heat exchanger to the mechanical olive oil extraction process was studied, evaluating the effect of olive paste cooling treatment on the yield and olive oil quality. The results demonstrated an insignificant difference regarding the yield and olive oil quality parameters, but a significant improvement in the phenolic compounds was observed (Veneziani et al., 2017).

In this research, a combination of innovative technologies including ultrasounds, microwaves and a horizontal spring heat exchanger was studied. The novelty of this study lies in the use of these three technologies assembled in a pilot plant. The industrial combined plant (ICP) used was a full-scale size, and was designed and assembled in order to gain increased knowledge concerning the use of innovative means of conditioning olive oil paste. The ICP was capable of operating by using all three technologies implemented simultaneously or individually. The study constituted the first experience of an ICP pilot plant using three implemented technologies proven in an operational environment, and evaluated the influence on olive oil quality and yield.

2. Materials and methods

2.1. Industrial olive oil extraction plant equipped with ICP

The experimental tests were performed in an industrial olive oil extraction plant located in Foggia (Italy). The mill included a leaf remover, washing machine (Special Automatic, Alfa Laval Corporate AB, Lund, Sweden), single-grid hammer crusher (75 hp Hammer Crusher, Alfa Laval Corporate AB, Lund, Sweden), six malaxer machines sealed on the top, each with a capacity of 700 L, three-phase solid/liquid horizontal centrifugal decanter (NX X32, Alfa Laval Corporate AB, Lund, Sweden), and a liquid/liquid vertical plate centrifuge (UVPX 507, Alfa Laval Corporate AB, Lund, Sweden).

The processing plant included the ICP for olive paste conditioning.

2.1.1. ICP system layout

The ICP was equipped with the following units: (i) continuous microwave machine (reverberant tunnel, generator head, power supply, water-cooled magnetron head and polypropylene pipe); (ii) ultrasonic pilot device (generator, transducer and vessel); and (iii) spiral-coil heat exchanger (with modular tubular units including helicoids). These three technologies were controlled by a programmable logic controller (PLC), capable of commanding simultaneous use or any possible combination among the components.

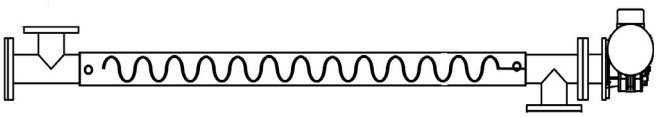

Fig. 1. Module of spiral-coil heat exchanger (SCHE).

In the following, the most relevant components of this plant are described in detail, with focus on the design specifications, constructive materials, suppliers and operational regimes.

The spiral-coil heat exchanger (SCHE) consisted of six units, each operating on tube-in tube, as illustrated in Fig. 1. The olive paste flowing through a straight tube was moved by a cavity pump placed in the output section of the grid hammer mill, while the hot fluid service flowed in counter-currently outside the tube in the heating jacket. Each module length was 2 m and heat exchange area of 0.56 m^2, and these were connected by flanges welded at their extremities and bolted. Inside the straight tube of each module, a spiral coil was connected to an electric motor equipped with a mechanical speed reducer. The SCHE was assembled by EMITECH s.r.l. (Corato, Italy).

The high-power ultrasound pilot device (UPD) was capable of producing electromagnetic waves at 20 kHz. It was equipped with an adjustable power supply, allowing the power output to range from 50 to 3000 W. A mean value of 2700 W was used during the experiments. The pilot equipment consisted of a stainless steel cylindrical tank, which was connected to the heat exchange and MW pilot device to ensure process continuity. An ultrasound transducer was placed inside the steel cylindrical tank.

The continuous microwave (CMW) system (Emitech s.r.l., Molfetta, BA, Italy), was constituted by a reverberant chamber constructed from AISI 304 stainless steel, equipped with a TM060 generator head (Alter s.r.l., Reggio Emilia, Italy) and coupled to a YJ1600C magnetron (Alter s.r.l., Reggio Emilia, Italy). The generator head was connected to a SM1180T power supply (Alter s.r.l., Reggio Emilia, Italy). This system had a maximum power of 6.0 kW at 2.45 GHz, and a mean value of 5.8 kW was used during the experiments. The magnetron was water-cooled. Inside the reverberant chamber there was a polypropylene tube with a diameter of 0.0654 m and length of 2 m.

A PLC included in the main electrical panel allowed for controlling the electrical power provided by the magnetrons and, consequently, the output temperature.

2.2. Experimental process description

The ICP was tested by selecting five different operational modes, schematized in Fig. 2, to investigate the optimal combinations for improving the oil extractability *(E)* and olive oil quality.

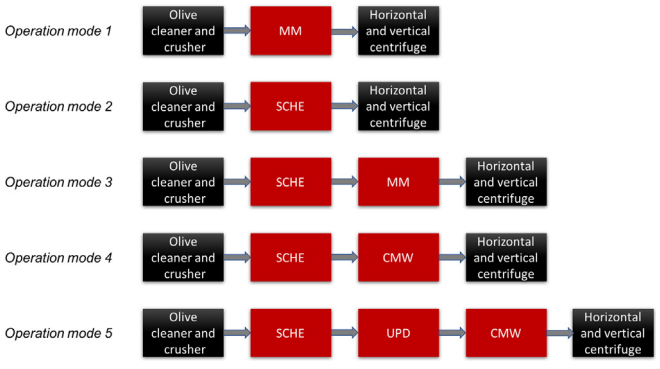

Fig. 2. Diagram flow of the five operation modes.

An experimental design was developed, using five replicates per treatment and the same olive batch.

Tests were carried out using olives of the Arbosana (*Olea europaea* L.) cultivar, with a maturity index of 2.6, and Arbequina (*Olea europaea* L.) cultivar, with a maturity index of 2.7. The fruit ripeness was determined according to the method proposed by the International Olive Council (IOOC, 2001). These values were chosen because represent the optimal status for olive harvesting (Proietti, 2014).

All tests were carried out at a mass flow rate of 1300 ± 10 kg h^{-1}, with 8.6% water added to the decanter.

The ICP operational modes were as follows:

2.2.1. Operation mode 1 (control test): malaxer machine (MM)

The ICP was bypassed during the process.

The olive paste that was just crushed was malaxed using a traditional MM at 25 ± 1 °C for 40 min and after being transferred to the decanter. These parameters were chosen to optimize the extractability and the reach a high-level olive oil quality.

2.2.2. Operation mode 2: SCHE

The ICP was operated by the SCHE device only, and the MM was bypassed.

The olive paste obtained was pumped by the main pump throughout the SCHE. The olive paste temperature was 25 ± 1 °C following SCHE processing, and the operating time was 3.61 min.

2.2.3. Operation mode 3: SCHE and MM (SCHE-MM)

The ICP was operated by the SCHE only, followed by malaxation treatment.

The olive paste that was just crushed was pumped throughout the SCHE. The olive paste output temperature was 23 ± 1 °C and the operating time was 3.61 min. Subsequently, the olive paste was transferred to the MM and processed for 20 min at 25 ± 1 °C.

2.2.4. Operation mode 4: SCHE and CMW machine (SCHE-CMW)

The ICP was operated by the SCHE, followed by the CMW machine, and the malaxer was bypassed.

The olive paste that was just crushed was pumped throughout the SCHE, reaching a temperature of 21 ± 1 °C, and then throughout the microwave device, reaching a temperature of 25 ± 1 °C.

The operating time was 3.61 min for the heat exchanger processing and 0.6 min for the microwave device processing.

2.2.5. Operation mode 5: SCHE, UPD and CMW machine (SCHE-UPD-CMW)

The ICP was operated by all three devices and the MM was bypassed.

The olive paste that was just crushed was pumped throughout the SCHE, reaching 21 ± 1 °C, and subsequently throughout the UPD followed by the CMW until reaching 25 ± 1 °C.

The operating time was 3.61 min for the heat exchange processing, 1.3 min for UPD processing and 0.6 min for the microwave device processing.

The specific energy transferred was 15.60 kJ kg^{-1} when the CMW machine was used and 8.31 kJ kg^{-1} when the UPD was used.

2.3. Sampling

The following samples were collected for each run:

- N. 1 sample of olives to determine oil and water content;
- N. 1 sample of pomace from the decanter. The sample was composed of a small fraction of pomace sampled from the decanter at regular time intervals of 1 min;
- N. 1 sample of wastewater from the decanter. The sample was composed of a small fraction of wastewater sampled from the decanter at regular time intervals of 1 min;
- N. 1 sample of olive oil. At the end of each run the oil was stored in a steel tank and one sample was taken.

All of the samples were stored at 4 °C until analysis.

2.4. Extractability (E)

The extractability (E) was calculated using the following equation:

$$E = \frac{W_{oil}}{W_{total\ \ oil}}.100, \quad (1)$$

where W_{oil} is the extracted oil mass (kg) and $W_{total\ oil}$ is the oil mass of the olives (kg).

2.5. Oil and water content in olives, pomace and wastewater

The total oil content was determined for a sample that was previously dehydrated until reaching a constant weight, and the moisture removed from the sample was recorded. The oil in the dried sample was extracted by hexane in an automatic extractor (Randall 148, Velp Scientifica, Milan, Italy), following the analytical technique described by Cherubini et al. (2009). The results were expressed as the percentage of oil in wet and dry matter.

2.6. VOO analysis

The VOO quality was assessed considering only the oils obtained from the Arbosana cultivar. The free fatty acids, peroxide number and spectrophotometric absorptions were determined and sensory analyses were carried out according to official methods (Regulation ECC 2568/91). Phenolic compounds were extracted by means of a methanol-water mixture (70:30, v/v) and the methanolic extracts were used to determine the total phenolic content (TPC) of the oil, as previously reported by (Caponio et al., 2018; Squeo et al., 2016). For the HPLC analysis, the extraction procedure was the same as that of TPC, with slight modifications. In particular, 5 g of oil and 2 mL of MeOH−H_2O mixture were used, and 250 μL standard solution of gallic acid (100 ppm in MeOH−H_2O) was added for quantification. All of the reagents used were HPLC grade. The chromatographic system and conditions are reported in (Tamborrino et al., 2017). The identification of single phenolics was carried out by comparing the retention times with those of the reference standards, or literature data where no standards were available. The tocopherols were analysed as previously reported in (Tamborrino et al., 2017), using the same chromatographic system. The carotenoids were spectrophotometrically determined by weighting approximately 0.25 g of a sample in a 10-mL volumetric flask and bringing it to volume with hexane. The absorbance was read at 449 nm against a blank of pure hexane. Quantification was performed by means of an external calibration curve of β-carotene (R^2 equal to 0.9987). The chlorophylls were determined in accordance with the IUPAC method (1995). All spectrophotometric analyses were performed on a Cary UV 60 spectrophotometer (Agilent Technologies, Inc., Santa Clara, CA, USA).

2.7. Statistical analysis

Each industrial treatment was performed five times, and all laboratory analyses were performed in triplicate. All experimental data were analysed using the ANOVA test and Tuckey's multiple range test ($\alpha = 0.05$), using the MATLAB® statistics toolbox (The Mathworks Inc., Natick, MA, USA). The data on the VOO quality were analysed by means of Minitab 17 software (Minitab Inc., State College, PA, USA). One-way ANOVA was applied, followed by Fisher's LSD post-hoc test for multiple comparisons, at a significance level of 5%. Principal component analysis (PCA) was carried out on the quality data using the same

Table 1
Quantitative results for the processing of the Arbosana olives.

	(MM)	(SCHE)	(SCHE - MM)	(SCHE-CMW)	(SCHE-UPD-CMW)
Dry matter in the waste water (%)	6.88 ± 0.17 b	17.32 ± 0.18 a	6.96 ± 0.14 b	6.82 ± 0.13 b	6.72 ± 0.19 b
Water content in the pomace (%)	55.30 ± 1.19 a	55.27 ± 0.94 a	55.66 ± 0.73 a	55.55 ± 0.65 a	55.27 ± 0.91 a
Oil content in the pomace (% d.m.)	13.54 ± 0.83 ab	24.38 ± 1.71 a	13.46 ± 1.12 ab	13.56 ± 1.72 ab	11.91 ± 1.23 b
E (%)	83.21 ± 1.13 ab	71.42 ± 1.69 b	83.20 ± 1.58 ab	83.50 ± 1.74 ab	85.51 ± 1.24 a
[a]Operating pressure (bar)	1.8	3.2	3.2	3.2	3.7

Data represent mean value ± standard deviation. Different letters in rows denote statistical significant differences ($p < 0.05$).
[a] The pressure value was measured after the cavity pump placed after the hammer crusher.

Table 2
Quantitative results for the processing of the Arbequina olives.

	(MM)	(SCHE)	(SCHE - MM)	(SCHE-CMW)	(SCHE-UPD-CMW)
Dry matter in the waste water (%)	7.24 ± 0.24 b	16.92 ± 0.20 a	6.74 ± 0.22 b	6.60 ± 0.07 b	6.72 ± 0.05 b
Water content in the pomace (%)	56.38 ± 0.71 a	55.26 ± 0.47 a	55.88 ± 0.76 a	55.26 ± 0.66 a	55.45 ± 0.68 a
Oil content in the pomace (% d.m.)	12.92 ± 1.44 ab	21.82 ± 1.73 a	13.28 ± 1.14 ab	12.78 ± 1.26 ab	10.67 ± 0.96 b
E (%)	80.00 ± 2.14 ab	67.56 ± 3.23 b	80.09 ± 1.40 ab	81.05 ± 1.31 ab	83.85 ± 1.12 a
[a]Operating pressure (bar)	1.8	3.2	3.2	3.2	3.7

Data represent mean value ± standard deviation. Different letters in rows denote statistical significant differences ($p < 0.05$).
[a] The pressure value was measured after the cavity pump placed after the hammer crusher.

software.

3. Results and discussion

3.1. Effects of ICP system on quantitative parameters

Tables 1 and 2 display the results of the dry matter in the wastewater, oil content in the pomace (dry matter), oil extractability (*E*) and operating pressure, respectively, for the Arbosana and Arbequina cultivars.

For both cultivars, the dry matter content in the wastewater was statistically higher when only SCHE was used for the olive paste conditioning. This means that the sedimentation of solids in the decanter took place with lower effectiveness than in other conditions, owing to the poor conditioning of pastes when only the SCHE was used. This aspect did not affect the oil lost in the wastewater. The value was less than 1% in all experimental tests (data not shown).

The water content in the pomace did not exhibit statistically significant differences among the tests for both varieties.

When only the SCHE was used, the oil content in the pomace was 24.38% (d.m.) and 21.82% (d.m.) for the Arbosana and Arbequina cultivars, respectively. This value is statistically higher than those obtained by other theses, which exhibited no significant differences.

Regarding the extractability, for both cultivars there were no significant differences between the conditions, except when only the SCHE was used, where the extractability value was statistically lower than that for the other four test conditions, with no significant differences between them. Based on the data analysis of the extractability and oil content in the pomace, it is possible to assert that the use of only the SCHE is not sufficient to provide high efficiency in olive paste conditioning.

The use of the SCHE in addition to the malaxer reduced the conditioning time, as also reported in previous papers such as those of Esposto et al. (2013) and Leone et al. (2016).

It should be emphasised that, by using the spiral heat exchanger, it was possible to work with pressures under 3.4 bar, which is significantly lower than the value of approximately 8 bar generally used in similar heat exchangers without the internal coil.

By comparing the MM, SCHE, SCHE-MM and SCHE-CMW, it is possible to analyse the individual effects of the microwave system on the plant performance. As reported in Tables 1 and 2, the use of the CMW machine in addition to the SCHE significantly increased the extractability and significantly decreased the oil content in the pomace. Moreover, the SCHE-MW condition exhibited no significant differences in extractability and oil content in the pomace compared to the control (MM) and SCHE-MM treatment. However, it was possible to achieve a significant reduction in the conditioning time, obtaining an entirely continuous process, without interruptions, from the milling phase to the solid-liquid separation phase. This confirmed previous conclusions drawn by Leone et al. (2014) and Tamborrino et al. (2014). The positive effect of CMW on *E* was demonstrated in this study, with a 4 °C thermal increase and a specific energy transfer of approximately 15.60 kJ kg^{-1}.

The impact of ultrasound on the plant quantitative performance can be assessed by comparing SCHE-UPD-CMW and SCHE-CMW. The use of the UPD (8.31 kJ kg^{-1} at 20 kHz) resulted in an increase of 2.01 and 2.80% for the Arbosana and Arbequina cultivars, respectively.

Comparing the use of the ICP system when operated with all three active devices, with respect to the control (MM), it can easily be observed that the increase in *E* was equal to 2.30 and 3.85% for the Arbosana and Arbequina cultivars, respectively. Although the extraction results are not statistically significant, it appears that the increasing *E* is inversely proportional to the data for the control thesis extraction. This means that a greater amount of oil contained in the pomace in the control test resulted in a greater effect of the combination of the three technologies on the decanter efficiency.

This result is in accordance with Bejaoui et al. (2016b), Iqdiam et al. (2017) and Jiménez and Beltran (2007).

3.2. Effect of ICP system on VOO quality

Considering the basic analytical parameters, all of the VOOs obtained from the experimental trials were below the maximum limits set for the extra virgin category, and no significant differences were highlighted owing to the paste conditioning process (data not shown). The fruity note median was higher than 0 in all cases, and no sensory defects were reported.

Table 3 displays the total phenolic, tocopherol and pigment contents of the samples, along with the statistical analysis results. The amount of phenolic compound in the Arbosana oils ranged from approximately 130 to 200 mg kg^{-1} oil, similar to the values previously reported for the same cultivar, which is recognised as belonging to the class of low-phenolic olive cultivars (Scarafia, 2013). Certain significant differences were highlighted as a result of the technological treatments. The superior performances in terms of the absolute phenolic amount were

Table 3
HPLC phenolic profile of Arbosana oil samples and results of the one-way ANOVA followed by Fisher's LSD test for multiple comparisons. Data are expressed as mean ± standard deviation (mg kg^{-1}; $n = 5$).

Identified compounds	SCHE-UPD-CMW	SCHE	MM	SCHE-MM	SCHE-CMW
Tyrosol	0.583 ± 0.140 c	0.076 ± 0.083 e	0.845 ± 0.194 b	1.025 ± 0.154 a	0.376 ± 0.024 d
Vanillic acid	0.175 ± 0.021 a	0.048 ± 0.046 c	0.209 ± 0.031 a	0.120 ± 0.023 b	0.120 ± 0.052 b
Syringic acid	0.150 ± 0.014 ab	0.091 ± 0.012 c	0.168 ± 0.017 a	0.092 ± 0.024 c	0.141 ± 0.006 b
p-cumaric acid	0.138 ± 0.023 ab	0.000 ± 0.000 d	0.144 ± 0.022 a	0.092 ± 0.020 c	0.118 ± 0.015 b
t-ferulic acid	0.151 ± 0.049 a	0.029 ± 0.040 b	0.099 ± 0.092 a	0.162 ± 0.030 a	0.010 ± 0.022 b
3,4-DHPEA-EDA	0.283 ± 0.024 b	0.205 ± 0.022 c	0.219 ± 0.015 c	0.265 ± 0.012 b	0.340 ± 0.017 a
3,4-DHPEA-EA-dialdehyde form	0.243 ± 0.020 a	0.119 ± 0.012 d	0.218 ± 0.011 b	0.160 ± 0.008 c	0.205 ± 0.008 b
p-HPEA-EDA	3.054 ± 0.198 a	1.800 ± 0.046 d	2.822 ± 0.283 b	1.928 ± 0.037 d	2.337 ± 0.014 c
Pinoresinol	2.101 ± 0.122 a	1.376 ± 0.066 c	2.056 ± 0.148 a	1.437 ± 0.035 c	1.660 ± 0.087 b
3,4-DHPEA-EA	7.535 ± 0.562 a	5.857 ± 0.165 c	7.369 ± 0.388 a	6.641 ± 0.338 b	7.309 ± 0.254 a
Luteolin	2.752 ± 0.338 a	0.511 ± 0.209 d	2.471 ± 0.340 a	2.128 ± 0.134 b	1.033 ± 0.218 c
p-HPEA-EA	1.821 ± 0.087 a	1.388 ± 0.051 d	1.786 ± 0.076 ab	1.578 ± 0.055 c	1.707 ± 0.022 b
Apigenin	3.367 ± 0.135 a	0.818 ± 0.355 c	3.217 ± 0.085 a	3.173 ± 0.181 a	1.553 ± 0.277 b
Total	22.354 ± 0.842 a	12.318 ± 0.582 d	21.623 ± 0.724 a	18.801 ± 0.481 b	16.909 ± 0.495 c

3,4-DHPEA-EDA, dialdehydic form of elenolic acid linked to hydroxytyrosol; *p*-HPEA-EDA, dialdehydic form of elenolic acid linked to tyrosol; 3,4-DHPEA-EA, oleuropein aglycon; *p*-HPEA-EA, ligstroside aglycon.
Different letters on the same row indicate significant differences ($p \leq 0.05$).

attributed to the combined SCHE-UPD-CMW treatment, which was not statistically different from the malaxation trial (MM). In the other cases, the oils were progressively poorer in phenolics, with the SCHE trial being most inferior. When the heat exchanger was coupled with malaxation or microwaves, the results were intermediate to the two conditions described above and not significantly different. In 2006, the first paper reporting on the use of a heat exchanger during the olive extraction process, by Amirante et al., stated that the oil phenolic content was positively influenced by the heat exchanger. However, in that case it was only used as a rapid tool for paste conditioning, and the malaxation conditions was similar to that of other tests (30 min at 27 °C). Subsequently, Leone et al. (2016) reported that a heat exchanger allowed for a reduced malaxation time from 40 to 10 min, without affecting the phenolic content, using olives of Peranzana cultivar. These results are not confirmed by this study, in which by using olives of Arbosana and Arbequina cultivars the phenolic content was lower even when coupling the heat exchanger with the traditional malaxation step for 20 min. In contrast, Esposto et al. (2013) found that similar behaviour was exhibited by a phenolic reduction when a heat exchanger was used. Similarly, studies on the application of microwaves have demonstrated that this treatment alone could not extract a comparable amount of hydrophilic antioxidant to that of the common process, even if the effect of such technology is clear and correlable with the reduction of the olive paste's conditioning time (Leone et al., 2018; Tamborrino et al., 2014). To the best of the authors' knowledge, no previous studies have taken into account the impact of new conditioning technology on lipophilic antioxidants and pigments. Considering such compounds, once again the SCHE-MM and SCHE-CMW trials provided very similar results, with the sole significant exception being the carotenoids content, which was higher when the heat exchanger was coupled with microwaves. Among the other trials, significant differences were only exhibited in the contents of the β-γ-tocopherols and chlorophylls, with MM increasing the content of the former and decreasing that of the latter. Overall, this is noteworthy as the different conditioning treatments did not negatively affect the lipophilic antioxidant contents compared to those observed for the hydrophilic ones. However, traditional malaxation was proven to be the superior treatment in terms of tocopherol extraction.

Table 4 reports the oil HPLC phenolic profile of the identified compounds. Overall, the data were in agreement with the TPC results and, considering the total amount, the same pattern could be concluded. Indeed, the highest values were determined in the SCHE-UPD-CMW and MM oils, with the lowest amount again detected in the SCHE oils. Considering the phenolic classes, the most abundant were the secoiridoids, arising from oleuropein and ligstroside (Bendini et al., 2007), while remarkable amounts of flavones and lignans were also detected. Tyrosol was the only phenyl ethyl alcohol found in the samples, with the highest significant value in the SCHE-MM trial. The SCHE-UPD-CMW and MM trials exhibited very similar phenolic profiles, with few significant differences, owing to the tyrosol and dialdehydic forms of secoiridoid contents. In particular, the SCHE-UPD-CMW oils were poorer in tyrosol but significantly richer in 3,4-DHPEA-EDA, 3,4-DHPEA-EA-dialdehyde form and *p*-HPEA-EDA. The compound *p*-HPEA-EDA, also known as oleocanthal, was indicated as the main factor responsible for the pungent notes of the oil (Bendini et al., 2007). The effect of the heat exchanger differed in the coupled treatment function. Indeed, when associated with microwaves (SCHE-CMW), higher amounts of secoiridoid derivatives and lignans were found (3,4-DHPEA-EDA, 3,4-DHPEA-EA-dialdehyde form, *p*-HPEA-EDA, pinoresinol and 3,4-DHPEA-EA), while, on the contrary, the association with traditional malaxation (SCHE-MM) provided significantly higher amounts of flavones. The treatment using only the heat exchanger exhibited the worst results, and almost all of the phenolic compounds were significant lower than those of the other trials. Considering the traditional extraction process, three main factors affected the olive paste preparation following crushing: malaxation time, temperature and air contact. The only processing factor that was significant different among the treatments was the paste conditioning time. Indeed, the processing temperatures in the different trials were almost the same and the observed difference could not be a result of this parameter. The same statement could be made regarding the air with which the olive paste was in contact, which was not modified, although modification in processing duration also caused changes in air/paste contact time. It is well known that, during malaxation, several enzymatic reactions take place, and wall-degrading enzymes (pectinases, cellulases and hemicellulases) along with endogenous glycosidases (β-glucosidase) and oxidoreductases (polyphenol oxidase and peroxidase) can affect the release of phenolic compounds as well as their profiles and contents (Cirilli et al., 2017; García-Rodríguez et al., 2011; Montedoro et al., 2002). In light of these considerations, as suggested by Esposto et al. (2013), the observed differences could be owing to the different times available for the enzymes to work. A shorter time resulted in less enzyme action and thus phenolic reduction. In fact, in the SCHE-MM, SCHE-CMW and SCHE trials, the paste conditioning was faster and the phenolic compounds were lower than in the common malaxation (MM). However, these considerations are not fully supported by the experimental results, in which the most superior were determined in the SCHE-UPD-CMW trial, where the paste conditioning time was also very short and lasted for approximately 6 min. In this case, the additional effect of the ultrasound treatment appeared to overcome the issue owing to the short

Table 4
Total phenolic, tocopherols and pigments content of Arbosana oil samples and results of the one-way ANOVA followed by Fisher's LSD test for multiple comparisons. Data are expressed as mean ± standard deviation (mg kg^{-1}; $n = 5$).

Compounds	SCHE-UPD-CMW	SCHE	MM	SCHE-MM	SCHE-CMW
TPC	195 ± 15 a	129 ± 15 c	194 ± 16 a	169 ± 13 b	158 ± 14 b
β + γ-tocopherols	4.73 ± 0.14 b	4.35 ± 0.09 b	5.83 ± 0.13 a	4.33 ± 0.08 b	4.56 ± 0.68 b
α-tocopherol	260.73 ± 1.36 ab	262.46 ± 3.15 ab	273.64 ± 3.64 a	255.23 ± 2.74 b	255.33 ± 30.14 b
Total tocopherols	265.45 ± 1.38 ab	266.81 ± 3.16 ab	279.48 ± 3.72 a	259.56 ± 2.72 b	259.89 ± 30.71 b
Carotenoids	8.07 ± 1.79 ab	8.25 ± 0.99 ab	8.55 ± 1.64 ab	6.45 ± 1.51 b	8.98 ± 2.77 a
Chlorophylls	5.18 ± 0.06 a	4.23 ± 0.03 b	4.12 ± 0.02 c	3.80 ± 0.05 d	3.79 ± 0.03 d

TPC, total phenolic content.
Different letters on the same row indicate significant differences ($p \leq 0.05$).

paste conditioning time. Indeed, acoustic cavitation owing to ultrasound may disrupt biological cell walls, facilitating the release of minor compounds (Chemat et al., 2017). Regarding the air/paste contact time, experimental evidences suggested that the benefits (in terms of phenolic content) of a shorter contact, likely due to a reduced paste oxidation, are outclassed by those arising from a longer one. As proved by Parenti et al. (2007), this could be explained considering that during malaxation only a small quantity of air is dissolved in the olive paste. Hence, despite the great differences in air/paste contact, it was not responsible for the differences in the final oil phenolic content. In summary, from a quality point of view, the heat exchanger and microwave have been proven as useful technologies for rapid olive paste conditioning, but they need to be associated with a common malaxation step or ultrasound treatment, which can respectively exert the mechanical effect required for enhancing phenolic release.

Moreover, paste malaxation was found to be the principal step in which volatile compounds were formed, through the activity of other fundamental enzymes, those of the so-called lipoxygenase (LOX) pathway. It is possible that modifications in the paste conditioning duration will alter the activity extent of such enzymes and, consequently, change the oil volatile profile. Further studies are necessary to investigate this aspect.

Fig. 3 illustrates the score plot (A) and loading plot (B) of the PCA performed on the oil quality data. Overall, the first two principal components accounted for approximately 70% of the total variance. The treatments were effectively separated within the space of the principal components, with the only exception being the MM and SCHE-UPD-CMW trials, which were overlapped. This is in accordance with previous discussions, from which it emerged that the oils from these trials exhibited very similar characteristics. The PCA clearly depicted that when only the heat exchanger was used (SCHE), the oils were poorer in both lipophilic and hydrophilic antioxidants. The SCHE-CMW and SCHE-MM trials lay in different areas on PC2, mainly owing

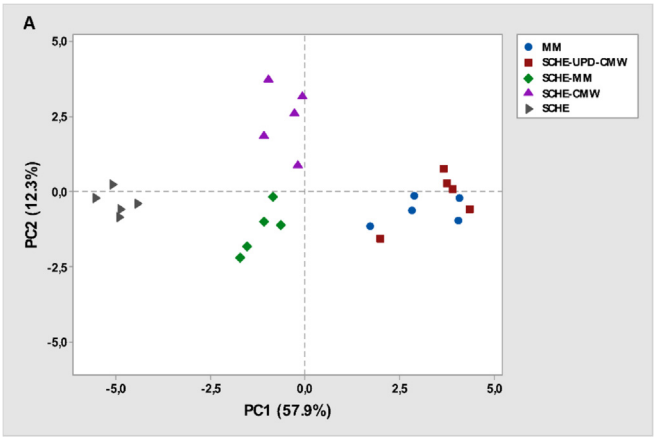

Fig. 3. Score plot (A) and loading plot (B) of the principal component analysis performed on the oil's quality data.

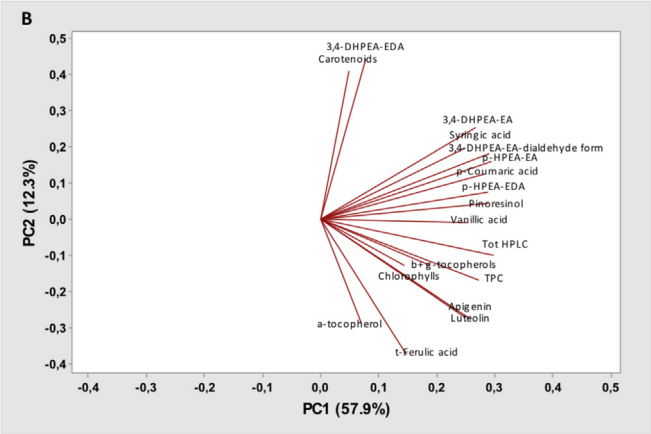

Fig. 3. (*continued*)

to the differences in the carotenoids and the 3,4-DHPEA-EDA content.

4. Conclusions

The experimentation highlighted the importance of using the spiral heat exchanger, which, in addition to the malaxer, reduces the conditioning time by half. Furthermore, the internal spiral aids in moving the paste from the input to output section, resulting in limited operating pressures. Therefore, the heat exchanger is a useful device for rapid temperature adjustment of the olive paste following crushing, thereby improving the malaxation effect, making its implementation within the oil extraction process necessary.

The research emphasises that, by using a microwave apparatus in addition to a heat exchanger, it is possible to obtain a continuous conditioning process of the olive paste with only several minutes of treatment. Finally, although the use of a low-frequency ultrasonic apparatus for conditioning the olive paste led to an average extractability increase of 2.30% and 3.85% with respect to the control thesis for the Arbosana and Arbequina varieties, respectively, this difference was not statistically significant. Although the use of ultrasound did not result in significant extractability increases, in light of the average differences obtained, it is probable that this difference could be significant when increasing the number of comparative tests.

Regarding the oil quality, the use of alternative conditioning technologies, individually or in combination, may save the lipophilic antioxidant furniture, while resulting in a hydrophilic antioxidant reduction. The ultrasound cavitation effect is capable of overcoming this drawback.

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