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Chapter 3

Ultrasound in Olive Oil Extraction

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http://dx.doi.org/10.5772/64765

Abstract

Each olive oil extraction system should combine the best product quality and the highest efficiency. At the same time, the innovative technologies can develop only if they provide sustainable processes. To reach these goals, academic and industrial researchers need to understand the key elements that allow to modulate the events that occur during oil extraction. In the past years, many emerging technologies, that is techniques perceived as capable of changing the present situation, have been developed. Among these, ultrasounds applications seem to be the most promising for their mechanical and slightly thermal effects, without affecting sustainability. In order to explain the maturity of this emerging technology, the main effects of the ultrasounds application in the olive oil extraction process are discussed, the developed plants are presented, and the patents are reported.

Keywords: emerging technologies, ultrasound-assisted extraction, pressure, viscosity, hydrophobic effect, emulsion breaking, olive oil quality, ultrasound patents

1. Introduction

Emerging technologies are techniques perceived as capable of changing the *status quo*. Food emerging technologies include a variety of technologies, such as high pressure, high intensity pulsed light, radio frequency electric fields, ohmic heating, microwave, ultrasound (US), and many others. Significant advances have been made in the research, development, and application of these technologies in food processing [1]. In the olive oil sector, ultrasound technology seems to be the most promising [2–6] due to its mechanical and slightly thermal effects. From a technological point of view, the entire virgin olive oil process has changed very little over the past 20 years. Crushing and malaxation are the most important critical points of the oil mechanical extraction process [7]. Crushing is the first step in the olive oil extraction.

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The fruits are ground up into a paste that will have different characteristics depending on the mill type, the olive variety and ripening stage and the type of the crusher employed. The next step is malaxation, a continuous slow stirring of the olive paste that increases the amount of free oil, more easily separable by centrifugation, by helping the oil droplets to merge into large drops and breaking the oil/water emulsion. The efficiency of malaxation depends on the rheological characteristics of olive paste and on technological parameters, such as time and temperature of the operation. However, the malaxer, actually is a machine working in batch, located between two continuous apparatuses, the crusher and the centrifugal decanter; consequently, it represents the bottleneck of the continuous extraction process.

The ultrasound waves applied to olive paste, before or during malaxation, could reduce the process time through two different mechanisms: a thermal effect and a mechanical effect. The thermal effect occurs when kinetic energy of the ultrasound waves is absorbed by a medium, and it is converted into the thermal energy. The mechanical effect is due to the cavitation phenomena. The cavitation is the formation, growth, and implosion of gas bubbles at high negative pressure. This process promotes the release of soluble compounds from the plant tissue by disrupting cell walls and improves mass transfer also in the olive tissues [3].

High-power ultrasound represents an efficient tool for large-scale food processing [8]. For each industrial application, this technology has to be developed and scaled up. When ultrasound is applied on a continuum fluid, it causes an acoustic pressure in addition to the hydrostatic pressure already acting on the medium. This acoustic pressure is a periodic wave dependent on time, frequency and the maximum pressure amplitude. There is a proportional relationship between the maximum pressure amplitude of the wave and the power input of the ultrasonic transducer.

When the intensity (amplitude) is low, the pressure wave causes an acoustic streaming within the fluid, consisting of a mixing action. When the intensity is higher, tiny gas bubbles grow within the fluid because the local pressure in the expansion phase of the cycle falls below the vapour pressure of the liquid. If the intensity increases, it can generate a negative transient pressure into the fluid that causes the bubble growth and produces new cavities due to the tensioning effect on the fluid [9]. If the bubble growth reaches a critical size, it implodes causing the phenomenon of cavitation, the most important effect in high-power ultrasound. Cavitation produces very high shear energy waves and turbulence into the medium, coupled with a localized increment of pressure and temperature. The combination of these factors causes the rupture of the biological cells accelerating the mass transfer.

Regarding the ultrasound frequency, the number of vibrations per second affects the bubble size in an inversely proportional manner. Consequently, low frequency ultrasound (i.e. power ultrasound 16–100 kHz) can generate great cavitation bubbles, which cause an increment of temperatures and pressures in the cavitation zones.

The industrial application of ultrasound requires two main conditions: a liquid medium (at least 5% of the overall substrate) and a source of high-energy vibrations (the ultrasonic device). The ultrasonic source is named "transducer." There are two main types of transducers: piezoelectric and magnetostrictive. Piezoelectric transducers utilize the piezoelectric property of a material to convert electrical energy directly into mechanical energy. Magnetostrictive transducers utilize the magnetostrictive property of a material to convert the energy in a magnetic field into mechanical energy.

Many parameters can be useful to describe an ultrasonic process, such as amplitude, pressure, temperature, viscosity, and concentration of solids. However, the main effects are function of the specific energy per kilogram (J/kg), that is the total amount of vibrational energy being delivered per mass unit, that are independent of scale. This is the reason of the scalability of ultrasonic technology.

If the pressure increases, the cavitation threshold and thus the number of cavitation bubbles is reduced [9]. The increment of the back pressure represents an effective tool to intensify the process maintaining low the amplitude.

Vapour pressure, surface tension, and viscosity of the liquid medium are parameters able to influence the cavitation phenomena and are temperature dependent. Increasing temperature causes an increase in the number of cavitation bubbles but their collapse results "amortized" due to the higher vapour pressure. On the contrary, decreasing temperature is followed by the viscosity decrease, allowing a more violent collapse. Thus, there is an optimum temperature, which enables to obtain both a low viscosity value, enough to create a violent cavitation collapse, and to avoid the dampening effect caused by a high vapour pressure.

2. Useful ultrasound effects in the virgin olive oil extraction process

2.1. Cell disruption

Oil is contained inside the olives in small cellular bags, the elaioplasts, specialized leucoplasts protected by a cellular membrane, responsible for the storage of lipids. Crushing is not effective in terms of oil separation, because it breaks olives but only few cellular membranes. Ultrasound disrupts the tissue structures, including membranes freeing the trapped oily phase.

2.2. Hydrophobic effect

The coalescence phenomenon of oily drops inside the olive paste is due to the hydrophobic interactions. During the malaxation, the oil droplets in the olive paste combine to form larger drops [10, 11]. This phenomenon, called cohesion, is related to the presence of water and its extensive hydrogen bonding. The water molecules tend to stick to each other in a regular pattern. Non-polar oil molecules are neither repelled nor attracted to each other. Water tends to squeeze non-polar molecules together, so oil droplets tend to form larger drops. In reference [12], Jordan observed that ultrasound positively affects collision and attachment of droplets and the coalescence phenomena. In reference [13], Filippov et al. confirmed that hydrophobic particles that are characterised by a low speed can be accelerated and activated by ultrasounds which promote the separation of the slow fraction. Ultrasound cavitation can increase the hydrophobic character of the water and modify the surface of hydrophobic particles by the formation of micro- or nanobubbles, facilitate the bubble-particle attachment and increase kinetics. Moreover, the ultrasound can enhance the probability of particles collision leading to an increase of oil recovery.

2.3. Emulsion breaking

Sometimes olive fruits can give, after milling, a kind of olive paste defined "difficult" or "very difficult" due to the formation of a strong oil/water emulsion that is hard to break even after malaxation. In these cases, the separation of the oil phase during centrifugation is more laborious. This kind of olive paste cannot give acceptable oil yields because of the fruit characteristics: very high moisture content (50–60%), high oil content (about 30%) and very low level of dry extract (10–15%).

If ultrasound is applied at high frequencies (<30 kHz), it can split the emulsion into its component, aqueous and oily phases [14].

3. Olive oil quality

Many authors have studied the effect of ultrasound applications during the extraction of oil from olives on oil quality parameters, nutritional and sensory characteristics [2]. Each of them, using different experimental conditions and olive varieties, reached similar conclusions. In 2007, Jiménez, Beltrán and Uceda applied, for the first time, high-power ultrasound on olive paste at laboratory scale during the EVOO extraction process. They observed no change in quality parameters (free acidity, peroxide value, K_{270} and K_{232}) in the resulting oils. The ultrasounds showed significant effects on the levels of bitterness and minor compounds. Oils from sonicated pastes showed lower bitterness and higher content of tocopherols (vitamin E), chlorophylls and carotenoids. Sensory evaluation by panel test showed higher intensity of positive attributes in sonicated oils.

Clodoveo et al., in 2013, tested at pilot scale the ultrasound treatment both on olive fruits submerged in a water bath (before crushing) and on olive paste before the malaxation (immediately after crushing). The aim of the experimental plan was to investigate the possibility of reducing the malaxing time. The ultrasound technology allowed a reduction in the duration of malaxing phase improving oil yields and its minor compounds content. Better results were obtained by sonicating the olives in a water bath than sonicating the olive paste.

A new experiment conducted by Bejaoui et al. (2015) confirmed the results obtained by Clodoveo et al. in 2014. They studied the employment of a continuous high-power ultrasound treatment on the olive paste before malaxation at pilot scale with the aim to study its effects on oil yield and quality. They observed that the ultrasound treatment caused an improvement of the oil yield of about 1% and the oil extractability equal to approximately 5.7%.

Recently, Bejaoui et al. (2016), testing high-power ultrasound on olive paste (before the malaxation), developed a response surface model to predict the olive paste temperature based on olive characteristics, olive paste flow rate and high-power ultrasound intensity. Moreover, they confirmed that ultrasound treatment did not cause changes in the oil quality indexes (free acidity, peroxide value, K_{232}, K_{270}) and in the volatile compounds, assuming that the fatty acid autoxidation was not accelerated by this treatment. The oils obtained from olive pastes treated with high-power ultrasound showed higher content of tocopherols, chlorophylls and carotenoids, while a reduction in phenolic content and bitterness intensity was observed. The constructive characteristics of the ultrasonic device, which caused a large exposition of the olive paste to the atmospheric oxygen, could explain these results. Furthermore, the heating of olive paste induced by high-power ultrasound was faster than that induced by the traditional warming system based on the conductive and convective systems. Tests conducted comparing the time required to warm the olive paste up to 27°C, a temperature useful to promote the coalescence phenomena, with the ultrasonic device and with the traditional malaxer, showed that ultrasound allows to reduce the pre-heating time by 66%. The reduction of the malaxation time represents a first step towards the conversion of malaxation in a continuous phase [6]. In reference [3], Clodoveo et al. used an ultrasound (US) treatment of olive paste before the malaxation step on *Coratina* and *Peranzana* cultivars. Different duration of US treatments was tested (0, 2, 4, 6, 8, 10 min), and free acidity, peroxide value, K_{232}, K_{270}, sensory analysis, tocopherols, total carotenoids, chlorophylls and total polyphenols were determined. Results showed that the US treatment caused a quick heating of the olive paste as a function of the US treatment time, allowing the reduction in the malaxation phase from 60 min to about 40 min. The ultrasound technique increased the antioxidants content in both oils, except for phenols that decreased due to the exposition to the atmospheric oxygen. This negative effect could be reduced or eliminated by designing a ultrasound devices equipped with a system able to modulate the atmosphere composition in contact with the olive paste.

The high-power ultrasounds do not affect oil quality parameters, while nutritional and sensory characteristics can be significantly influenced. The biophenols content, as well as tocopherols (vitamin E), chlorophylls and carotenoids content can be improved. Sensory evaluation by panel test showed higher intensity of positive attributes and lower of negative characteristics in oils from sonicated pastes compared to those untreated. Off-flavours were not detected in oils from sonication treatments

3.1. Aromatization

Clodoveo et al. [15] in 2016 studied a new aromatization technique based on the ultrasound treatment of olive paste mixed with aromatic plants (thyme and oregano). The aim of the work was to develop a system applicable in the industrial sector to obtain aromatized oils in a shorter time and with an antioxidant content higher than those obtained by traditional methods (infusion of herbs in olive oil or crushing the herbs together with the olive fruits). They found that ultrasound can inhibit the olive polyphenol oxidase, the endogenous enzyme responsible for olive oil phenol oxidation. Moreover, the ultrasound treatment of olive paste with herbs was the most suitable method to obtain aromatized olive oil, due to the best efficiency, reduced time consumption and labour and the enhancement of the product quality.

4. US prototypes

The US employment, in industrial scale, for VOO extraction process is only in prototype versions. One of them is the full-scale plant realized and tested in a full scale in Italy on October 2014, when Amirante and Clodoveo et al. placed a so-called sono-exchanger immediately after the crusher, in order to transform the discontinuous malaxing step in a continuous phase. The main goals of the research project were to improve the working capacity of the industrial plants and to increase the extraction of the main virgin olive oil minor compounds. The industrial plant was designed on the basis of previous experiences and tests performed by means of a fluid dynamic analysis and reported in [1, 4, 9] by the same authors. A continuous plant was modified by adding a "sono-exchanger" between the crusher and the malaxer, as shown in **Figure 1**.

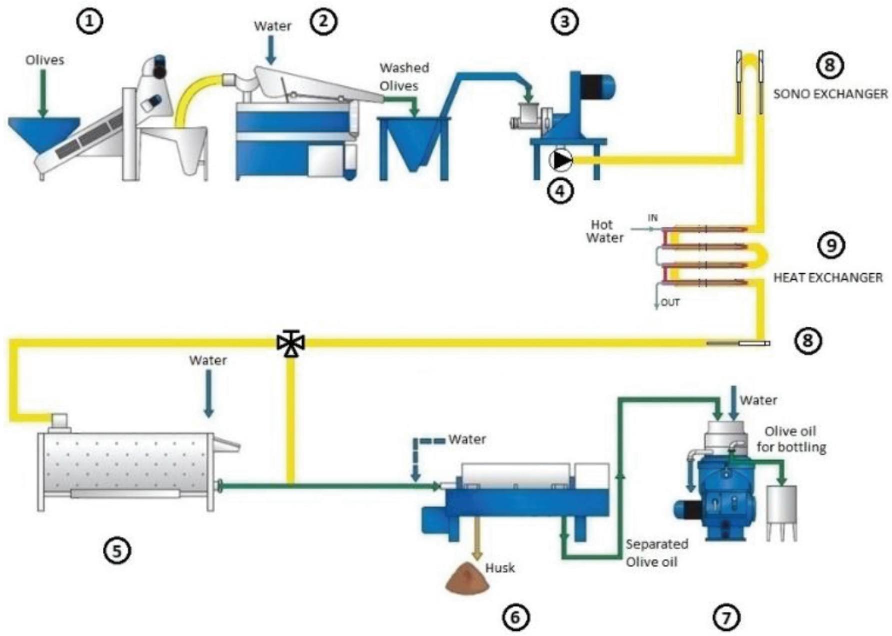

Figure 1. US prototype layout: 1. reception stage; 2. washing stage; 3. crushing stage; 4. pump; 5. malaxing stage; 6. separation stage; 7. clarification stage; 8. ultrasonic probes; 9. heat exchanger.

The sono-exchanger was made of two straight pipes connected by an elbow (cfr. **Figures 1** and **2**). Two ultrasonic rod-style transducers (Sonopush Mono® 30–1500 W–30 kHz) were plugged into the straight pipes through the bend. At last, a third ultrasound transducer (30–1400 W output power) was placed downstream the heat exchanger.

The ultrasonic probes inside the pipe provided a vibrational energy transfer to the olive paste flowing through the sono-exchanger. A specific energy of 12÷15 kJ/kg provided the best results [4]. A series of two annular heat exchangers, suitable for non-Newtonian products with high viscosity and for products that contain particulates, was used to fine-tune the temperature of the olive paste before it reached the malaxer. The heat exchanger consists of four concentric tubes.

The product medium flows in between two service channels and is heated from the inside and outside at the same time. The media fluid was water and flowed in opposite direction to the olive paste.

Figure 2. The sono-exchanger system and the three drivers, which provide the periodic signals (Colletorto, Italy, Aloia farm).

The plant is very easy to set-up for each technical staff, and it gives the opportunity of selecting the process parameters to extract virgin olive oil in relation to the heterogeneity of the fruit characteristics.

5. Ultrasound patents the edible oil industrial sector

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The manufacturing industry of olive oil extraction plants and the research institutes have a positive view of the ultrasound technology, patenting its uses.

Jimenez et al.'s researchers of the Andalusian Institute of Agricultural Research and Training, Fisheries, Food and Organic Production registered a patent in 2007 entitled "Apparatus and method for the continuous heating and uniform ultrasonic treatment of olive paste" [16]. The equipment is composed of the following elements:

- a temperature sensor for the inflow of the olive paste;
- an ultrasonic piezoelectric transducer (power comprised between 120 and 1200 W);
- a temperature sensor for the output of the olive paste;

- a system for the control of applied ultrasound and inflow ground olive mass based on the information provided by the temperature sensors.

Pieralisi Srl, one of the main manufacturers of olive oil extraction plants in Italy, registered a patent in 2011 named “Installation for extraction of oil from olive paste” [17]. The purpose of the invention is to eliminate the drawbacks of the prior art by disclosing an installation for extraction of olive oil that considerably reduces kneading time, while improving the quality of the oil without impairing the extraction yield. The installation comprises:

- a crushing station to crush olives in such a way to obtain a paste composed of pulp and olive pit;
- a kneading station (malaxer) comprising at least one basically cylindrical tank with rotating blades supported by a shaft disposed in axial position in the tank to knead the paste;
- a centrifuge for the separation of the oil from olive paste;
- a heater-conveyor disposed between the crushing station and the kneading station, comprising a cylindrical tubular structure with air space with circulation of hot water and a worm conveyor axially disposed inside said cylindrical tubular structure in such a way to generate an auger conveyor with product inlet and outlet.

In order to additionally accelerate the oil extraction process, ultrasounds can be applied in direct contact with the olive paste. The synergic effect of the ultrasound treatment that causes the breakage of the membranes and the release of oil and the heater-conveyor allows a considerable reduction of the kneading time, ensuring high yield without impairing the quality of the oil.

The ultrasound treatment device can be installed upstream the heater-conveyor. In such a case, the application of ultrasounds to the olive paste favours the breakage of the pulp cells, allowing the release of oil from the vacuoles. The above makes the paste more oily and slicker, thus reducing friction on the internal walls of the heater-conveyor. Therefore, the synergetic effect of the ultrasound treatment and the piston pump favours the passage of the olive paste to the heater-conveyor, avoiding possible deposits of paste on the internal walls that may overheat and damage the quality of the extracted oil.

Femenia et al. [18] registered a patent in 2012 with a method for preventing total or partial crystallization of olive oil during storage at low temperature. It includes the application of ultrasonic energy to the olive oil allowing to retain the physical/chemical and sensory properties of the product even if subjected to low temperature during storage.

Besides these patents, specifically related to the virgin olive oil industry, other patents for the industrial application of ultrasound have been registered. The declared effects of these patents could be exploited also to improve the virgin olive oil extraction process.

Bates and Bagnall [19] registered in 2009 a patent entitled “Viscosity reduction”. It is a method for reducing a product viscosity by applying highly propagating ultrasonic energy. The method requires the contact of at least a portion of the product with an assembly that propagates highly emission of ultrasonic energy

http://dx.doi.org/10.5772/64765

Bates and Bagnall [20] registered in 2012 the patent "Methods for isolating oil from plant material and for improving separation efficiency". This invention is based in part on the finding that high velocity microliquid streaming, created when cavitation bubbles collapse within a liquid, causes oils to be separated from cellular materials more easily, efficiently and quickly, with higher extraction yield. In this patent description, the inventors employed standing waves at high ultrasonic frequencies, >400 kHz to facilitate the separation of oil from solids. At frequencies >400 kHz, it is practical to produce large area standing waves at low amplitudes. Plate transducers are employed to create standing waves, because they operate at specific amplitudes, very much lower than those accomplished by horn transducers. Acoustic separation by standing waves is in principle quite rapid, separating particles breaking down to submicron size in seconds. Sonication can also reduce the pressure head required to pump liquid and minimize clogging and consequent maintenance costs.

It offers a means of further segregating particles on the basis of their density and compressibility. Moreover, ultrasonic waves have the ability to alter the interaction between fat globules through acoustic pressure. Under the appropriate conditions, they can cause aggregation of fat globules/fine particles, which induce the separation on the basis of the relative specific gravities of the phases and recovery of these particles.

Adnan et al. [21] registered the patent entitled "Improving oil recovery and reducing the oxygen demand of palm oil mill effluent". This method allows to increase the oil recovery and to reduce the biochemical oxygen demand (BOD) and chemical oxygen demand (COD) of palm oil mill effluent reducing the amount of palm oil in the effluent.

6. Conclusion

The application of new emerging technologies, such as ultrasounds, in the virgin olive oil production process could offer an interesting number of advantages due to their mechanical and thermal effects. However, many of them are not sustainable because of their "energy vampire" attitude or their very high price. The most promising emerging technology appears to be the ultrasound (US) application. In the recent years, the use of the ultrasound conditioning in adsorptive bubble separation process has been expanding due to the evidence that this technology increases the efficiency of the hydrophobic particles separation. Furthermore, the US technology is able to induce the rupture of cell walls and facilitate the recovery of the oil and minor compounds trapped in the uncrushed olive tissue; this increases the work capacity of the extraction plant and reduces the process time. In recent years, many papers and patents have been presented with concordant results on the use of ultrasound, applied in different ways, by different universities and research institutions. Analysing each activity, it is possible to conclude that one of the most important future challenges is to design and build ultrasound machines to improve the working capacity of the industrial plants and to perform a real continuous process able to increase quality as well as extraction yield. At the same time, it is necessary to reduce production and investment costs and optimise the plant working capacity.

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