processes

MDPI

Article

Adjustment of Olive Fruit Temperature before Grinding for Olive Oil Extraction. Experimental Study and Pilot Plant Trials

Eddy Plasquy [1,*], José María García Martos [1,*], María del Carmen Florido Fernández [2], Rafael Rubén Sola-Guirado [3] and Juan Francisco García Martín [4]

[1] Department of Biochemistry and Molecular Biology of Plant Products (CSIC), Instituto de la Grasa, 41013 Seville, Spain
[2] Department of Crystallography, Mineralogy and Agricultural Chemistry, ETSIA, University of Seville, 41013 Seville, Spain; florido@us.es
[3] Department of Mechanics, University of Cordoba, 14014 Cordoba, Spain; ir2sogur@uco.es
[4] Departamento de Ingeniería Química, Universidad de Sevilla, 41012 Seville, Spain; jfgarmar@us.es
* Correspondence: eddy.plasquy@telenet.be (E.P.); jmgarcia@cica.es (J.M.G.M.)

Abstract: Harvesting at high temperatures and bulk transport can negatively influence the quality of olives and lead to undesirable alterations in the extracted oil. Cooling the fruit in the field would be the most logical solution, but it means that the olives arrive too cold at the mill for immediate processing. In this work, the use of warm water in the washing tub to warm up the fruit before grinding instead of flash heat treatment on the paste was assessed in two experiments. In the first one, at the laboratory level, the temperature after milling was determined in three olive cultivars, previously stored at 5 or 10 °C, and then submerged at different water temperatures (25, 30, and 35 °C) for 15, 30, 45, and 60 s. In the second one, two batches of olives were cooled in the field at 5 °C and then conditioned with washing water to obtain a paste at the entrance of the pilot plant malaxer at 27 °C. The temperature of the olives was measured at five points from the discharging up to their entering, as paste, into the malaxer. The results demonstrated the feasibility of the method as the temperature of the ground olives was kept at the desired temperature (28 ± 1 °C). The trials highlight the potential for automating an even more precise adjustment of the temperature of the olives before milling once the washing tub is equipped with a safe heating system.

Keywords: malaxation; olive fruit; olive oil; postharvest; thermal treatment; washing

check for updates

Citation: Plasquy, E.; García Martos, J.M.; Florido Fernández, M.d.C.; Sola-Guirado, R.R.; García Martín, J.F. Adjustment of Olive Fruit Temperature before Grinding for Olive Oil Extraction. Experimental Study and Pilot Plant Trials. *Processes* **2021**, *9*, 586. https://doi.org/10.3390/pr9040586

Academic Editor: Carl Schaschke

Received: 3 March 2021
Accepted: 24 March 2021
Published: 27 March 2021

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https://creativecommons.org/licenses/by/4.0/).

1. Introduction

The quality of virgin olive oil and its organoleptic characteristics are directly related to the integrity of the harvested fruit as well as the conditions under which the extraction is carried out. In this respect, multiple studies have underlined the importance of the used harvesting method and the postharvest conditions of the fruit before its processing [1,2]. Available methods to harvest olives [3] influence the quality parameters, and the detrimental effects of a mechanized harvest can be aggravated during their transport and storage [4,5].

When storage is needed and mechanical harvesting is employed, a low temperature is recommended [6,7]. Climatological changes are provoking a shift in the phenology of the olive tree and are bringing forward the harvest time by several weeks to warmer seasons [8,9]. The high temperatures of harvested fruit along with a transport to the olive oil mill of a couple of hours in trailers may create conditions of asphyxia and induce anaerobic respiration [10]. Consequently, fermentation processes produce undesirable by-products that lead to detectable organoleptic defects and high levels of alcoholic esters in the extracted oil and ultimately to the rejection of the oil as extra virgin olive oil [11].

The conditions under which the malaxing phase takes place (time and temperature) are known to play a crucial role in the olive oil yield, the phenolic content, and the formation of volatile components [12–14]. During the slow and continuous kneading of the ground olives (olive paste), the temperature influences the physicochemical quality parameters and the enzymatic activity. Temperatures above 35 °C increase the oil yield but induce lipolytic, hydrolytic, and oxidative degradation, affecting both phenols and volatile compounds [15]. Malaxing temperatures below 14 °C negatively affect the oil extraction performance and induce compositional changes in the resulting olive oil [12–16]. Extending the malaxation time up to 60 min increases the oil yield but leads to a decrease in its phenolic content and drastically reduces the shelf life of the olive oil [17]. Shortening of the kneading reduces the enzymatic activity necessary for the development of sensorily interesting volatile components. Nowadays, it is widely accepted that the ideal malaxing temperature ranges between 25 and 30 °C for a malaxing time between 30 and 45 min [12–14].

Grinding the olives raises the temperature by at least 5 °C [18]. Consequently, obtaining the ideal malaxation temperature becomes exceedingly difficult with olive fruits that arrive at the olive oil mill at a temperature above 25 °C. Cooling the olive fruit with cold air or hydro-cooling, or passing the paste through a heat exchanger, have already been suggested as potential alternatives [16,19,20]. However, cooling the fruit right after the harvest seems more logical, as it does have the additional advantages of reducing the fruit metabolism and avoiding the action of enzymatic agents involved in deterioration processes [21,22]. Although cooling the olives in the field still has to be developed on an industrial scale, a small-scale work demonstrated the feasibility of an on-farm cooling room [23].

The aforementioned small-scale work at the farm level also revealed important issues that are easily overlooked when theorizing the possibility of cold storage of olives [24]. Supplying olives to olive oil mills at 5 °C does not imply that the olives can be easily processed. Even after being warmed up during its transport to the mill, the cleaning, the washing, and finally the grinding made the temperature of the olive paste remain far below the desired temperature in the malaxer and could not be warmed up to the desired temperature by the inbuilt heating system. Therefore, a heating phase is required before entering the malaxation machine.

During the last decade, flash heat treatment has been suggested to overcome the limitations of the traditional malaxation process. It is argued that malaxers are inefficient not only in terms of working continuity but also for the great amount of thermal energy that is required to warm the olive paste after crushing because malaxers are poor heat exchangers [13]. Ultrasound, microwave, heat exchangers, and combinations of them have been proposed as alternatives for malaxers [25–31]. Currently, several models of heat exchangers (Pieralisi Protoreattore, Pieralisi, Jesi, Italy; Alfa Laval EVO-line, Alfa Laval, Lund, Sweden) are available on the market, while the combination of a heat exchanger and ultrasound (sono-heat exchanger) is in its final stage before industrial production [32]. However, these innovative technologies imply serious investments, modifications of the production lines, and higher energy consumption [33].

Using warm washing water to bring the olive fruit to the ideal temperature before grinding has not been taken into consideration to date. Currently, the washing tubs are filled with cold water from the tap (10–12 °C). The water then further warms or cools during the day, depending on the air temperature. The washing phase is solely regarded as a step to remove impurities and pesticide residues [34]. This work aimed to assess whether the use of warm washing water could solve the specific problem that arises when olives arrive too cold at the mill, as in the on-the-farm cooling case, and as such can be considered as a potential alternative to optimize the malaxation process.

2. Materials and Methods

Two experiments were carried out at the Instituto de la Grasa (Spanish National Research Council) in Seville (Spain) between September and December 2019. At laboratory scale, the heating of different varieties of olives with water at different temperatures and times was simulated to determine the temperature of the paste after milling. At the available pilot plant of the Instituto de la Grasa, the temperature of two batches of olives, previously stored at 5 °C at the farm, was controlled by keeping the washing water at a chosen temperature using heaters. The used olive fruits were harvested in the Del Cetino farm in Bollullos par del Condado (Huelva, Spain).

2.1. Laboratory-Scale Experiment

Fifty kilograms of three different cultivars, 'Arbequina', 'Cobrançosa', and 'Gordal' (*Olea europaea* L.), were hand-picked, immediately brought to the research center, and stored at 5 °C or 10 °C in perforated plastic boxes inside two cooling rooms. The varieties were chosen for their distinct mean weight. All the fruits were healthy, free of diseases, and without signs of deterioration. The maturity index was determined according to the Jaen method, and their weight measured (Table 1) [35].

Table 1. Maturity index (M.I.), weight, and proportionality constant (k) empirically calculated for different varieties, at two different initial temperatures (T_i) and after being submerged in water at different temperatures.

Variety	M.I.	Weight (g)	T_i (°C)	Value of k T_w = 25 °C	T_w = 30 °C	T_w = 35 °C
Arbequina	2.1 ± 0.2	0.84 ± 0.17	7.1	−0.056 (0.019; 1.43 °C) *	−0.043 (0.017; 2.87 °C)	−0.032 (0.012; 1.79 °C)
			12.2	−0.045 (0.015; 1.96 °C)	−0.043 (0.017; 1.29 °C)	−0.031 (0.010; 1.85 °C)
Cobrançosa	1.9 ± 0.3	2.75 ± 0.17	7.1	−0.055 (0.019; 2.29 °C)	−0.037 (0.017; 3.45 °C)	−0.028 (0.011; 1.88 °C)
			12.2	−0.043 (0.020; 2.67 °C)	−0.042 (0.014; 1.09 °C)	−0.031 (0.010; 1.94 °C)
Gordal	1.7 ± 0.4	8.72 ± 1.73	7.1	−0.021 (0.010; 2.34 °C)	−0.017 (0.007; 2.79 °C)	−0.015 (0.003; 0.95 °C)
			12.2	−0.020 (0.010; 2.85 °C)	−0.015 (0.005; 1.04 °C)	−0.012 (0.004; 2.46 °C)

* mean values of k (calculated at 15, 30, 45, and 60 s); values within brackets (SD; RMSE).

Throughout the experiment, a cooled pot was used to transport the olives to the laboratory (10 m distance), while an isolating plate was placed on top of it to reduce the warming of the fruit during the transfer. The experiment consisted of immersing roughly 0.5 kg of each batch in a stirring water bath at three different temperatures: 25 (T25), 30 (T30), and 35 (T35) °C, for 4 different time intervals: 15 (t15), 30 (t30), 45 (t45), and 60 (t60) s.

A Unitronic 320 OR stirring water bath (P-Selecta, Barcelona, Spain) was filled with approximately 20 dm^3 of water. A plasticized iron wire basket equipped with a lid (25 × 15 × 7 cm) was placed on the moving plate to keep the olives together and allow the water to flow. The recipient could contain 0.5 kg. Keeping the olives moving was crucial to simulate the passing of the olives in the washing tub and to attain a maximum heat transfer during the immersion time. When the set temperature was attained, a pot of olives was taken out of the cooling room, brought to the laboratory, and poured into the basket as fast as possible. The lid was closed, the moving system activated, and a stopwatch pressed. At the set time, the basket was taken out of the water, quickly stirred to remove as much water as possible, and transferred to the close-by grinder. The olives were ground in the MM-100 hammer mill which forms part of the Abencor (MC2 Ingeniería y Sistemas S.L., Seville, Spain) laboratory sets for olive analysis, present in the laboratory and used for the extraction of small samples of olive oil. The paste was collected in a flat plastic bowl and quickly shaped into a rectangular block with the aid of a spatula once the grinding was finished. Immediately afterward, the internal temperature was measured with a digital probe thermometer. The control trial consisted of fruit that was ground and measured without being immersed. Each trial was performed in triplicate. During the whole process, either an IR thermometer or a digital probe thermometer was used to properly control the temperature.

The grinding of the olive fruit in the hammer mill increased the temperature of the resulting paste. This temperature increase (ΔT_g) was determined experimentally for each variety and each storage temperature. The temperature of the intact fruit on arrival in the lab (T_i) and that of the paste obtained from this fruit (T_{ig}) allowed for calculating ΔT_g. The values of ΔT_g were used to calculate the fruit temperature during the immersion:

$$T_{ft}(t) = T_{pt}(t) - \Delta T_g \tag{1}$$

(for t = 15, 30, 45, and 60 s)

T_{ft} = Temperature of the fruit, after being immersed in water for a given time t.

T_{pt} = Temperature of the paste, obtained from fruit immersed in water for a given time t.

ΔT_g = Temperature increase as a consequence of the grinding.

Based on Newton's cooling law, the temperature difference between the fruit and its surroundings (water) allows for calculating the rate of heat loss as a function of time when assuming a low Biot number and a specific heat capacity (k) independent of temperature. The solution to that equation describes an exponential decrease in the temperature difference over time:

$$T(t) = T_a + (T_i - T_a)e^{kt} \tag{2}$$

where k can be expressed as:

$$k = \frac{\ln\left(\frac{T(t) - T_a}{T_i - T_a}\right)}{t} \tag{3}$$

Knowing the initial temperature of the fruit (T_i), the calculated values of T_{ft} for the immersion time (t) and the given water temperature (T_a) allowed for calculating the value of k for each batch of olives at different times, thus obtaining the average k value for each variety. When the proportionality constant is known, the temperature at a set time can be calculated, given the temperature of the object and the temperature of the surroundings.

2.2. Pilot Plant Experiment

The experiment was performed with two batches of olives ('Arbequina' and 'Picual'). Both were harvested, stored, and transported similarly to those of the laboratory-scale experiment. The harvested fruit was cleaned to remove leaves and twigs, weighed, temporarily stored in a hopper, and finally washed before being ground as the first step of the extraction process (Figure 1). The experimental mill extracted only one batch a day and the production line was cleaned between tests.

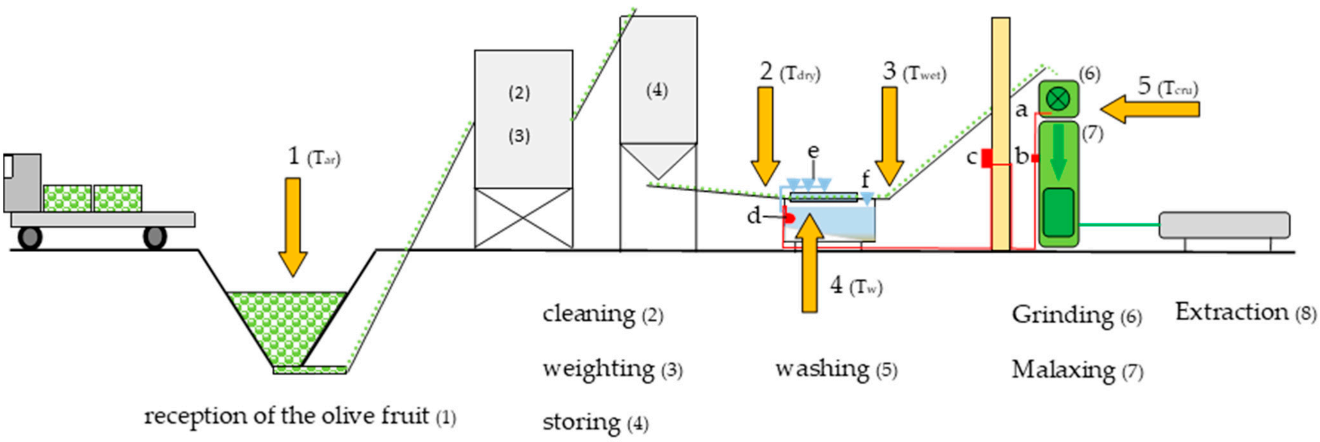

Figure 1. Processing steps of the olive fruit from their discharge up to malaxation of the paste. The different measurement points are indicated: At the reception of the olive fruit (**1**); before entering the washing tub (**2**); right after leaving the washing tub (**3**); and after being crushed (**5**). The water temperature is also continuously measured (**4**). The automized control system (red line) consisted of a temperature gauge at the exit of the grinder (**a**), a thermostat (**b**), a relay (**c**), and 4 heaters placed in the water tub (**d**). The water circulated in the washing tub from the lower to the upper part (**e**) and back (**f**).

The Lav Air/R-5000 washing tub (JAR, Mancha Real, Spain) has a capacity of 1 m^3 and is divided into two parts. In the upper part, water flows through an open stainless box of 50 × 100 cm, in which injected air provokes fierce turbulence of the water. The olives in the water are pushed forward by the ones entering and end up on a vibrating perforated plate and finally an inclined plate from where they fall on a moving belt that transports them to the indoor part of the milling facility. The lower part of the tub is an open reservoir for the circulating water and is designed to facilitate sedimentation. A pump continuously moves water from the lower to the upper part where it falls back into the reservoir through the perforated plate. It was estimated that between 15 to 20 kg were washed at a time. The average immersion time was estimated as the relation between the fruit quantity (Cw, kg) in the washing tub and the washing capacity (kg/h), being 18 s (Cw = 15 kg) and 24 s (Cw = 20 kg) when processing 3000 kg/h and between 11 s (Cw = 15 kg) and 15 s (Cw = 20 kg) when processing 5000 kg/h.

The olives were directed toward an FP HP 40 hammer mill (Pieralisi España, S.L.U., Mengíbar, Spain). The grinder was mounted on top of the malaxer so that the crushed olives (or paste) fell straight into it. A malaxer model 1250 2C E (Pieralisi España, S.L.U., Mengíbar, Spain) was used, which has a capacity of 6 m^3 and is composed of two connected chambers, one on top of the other. Within each chamber, two slow-turning propellers move the paste continuously. The chambers can be heated by hot water, circulating between the double walls, to warm the olive paste if necessary. The whole malaxing process was fully automized and continuously monitored through sensors that registered the fill level and the temperature of the paste as well as the circulating water and the malaxing time. After 2700 s at 28 °C in the malaxer, the paste was injected into the decanter, the next step being to extract the oil by gravitational forces. Once the extraction started, the process continued until the malaxer was empty.

Based on the results of the laboratory-scale experiment, it was decided to bring the temperature of the washing water to 40 °C. During the discharge of the olives, an operator filled the tub with hot water while the temperature was controlled with a digital thermometer of which the probe was fixed in the water. At the same time, four electric heaters were hung in the water at the edge of the bath to keep the water temperature at 40 °C during the whole process.

The temperature of the olives was measured at 4 different points from their discharge at the reception yard up to their entering in the malaxer, while the temperature of the water in the washing tub was continuously measured (Figure 1). The first measurement was done on the discharged pile of olives (arrival temperature, T_{ar}). The second took place after the olives had been cleaned, weighed, and temporarily stored in a hopper. The temperature of the olive fruit was measured during the transport from the hopper to the washing tub (T_{dry}). The third measurement took place right after the washing, on a sample of roughly 500 g olive fruit, collected in a plastic jar before falling on the transporting belt (T_{wet}). All these measurements were performed with an IR thermometer. Finally, the last measurement was realized on the crushed olives before entering the malaxer (T_{cru}). Measurement of the temperature of the ground olives before they entered the malaxer was hindered by the safety prescriptions that made it impossible to directly access the grinder. Therefore, a 15 cm stainless steel shaft was mounted in the front lid of the grinder and placed right below the exit. A PTC S6-S temperature probe (Osaka Solutions, S.L., Barcelona, Spain) was placed in the shaft together with thermal silicon grease to facilitate the heat transfer. The digital display/thermostat of the Mundocontrol FN-42 probe (Salvador Escoda S.A., Barcelona, Spain, 0.1 °C precision) was fixed at one side of the malaxer and made it possible to register the temperature of the olive paste. The temperature of the washing water (T_w) was measured by a probe that was fixed in the lower water compartment of the washing tub. The digital thermometer was fixed on the side to allow easy reading. Three operators recorded the temperatures T_{dry}, T_{wet}, T_w, and T_{cru} every 3 to 4 min until all olives were completely ground. The air temperature (T_{air}) was measured at the start of the experiment.

For the second trial, an electric circuit (Figure 1a–d) was mounted to facilitate the automized activation of the heaters in the washing tub. The digital display was replaced by a thermostat that was set at 27 °C (±1 °C). The thermostat was connected to a relay that in turn switched the heaters on and off.

2.3. Statistical Analysis

The data of the experiments were organized in Microsoft Excel 2010 (version 2101) (Microsoft, Redmond, WA, USA) sheets and further analyzed and visualized with IBM SPSS Statistics (version 24) for Windows (IBM, Armonk, NY, USA). ANOVA was applied for assessing, firstly, the effect of the variety, initial fruit temperature, water temperature, and time of immersion on the fruit temperature at time t (four-way), and for each variety separately (three-way), secondly, the effect of the variety on the value of the k (one-way) within each variety, and, finally, the effect of the initial temperature and the water temperature (two-way). When a significant effect was detected, a Tukey test was applied for differentiating mean values. The root mean square error (RMSE) was calculated to evaluate the average deviation of the predicted values, obtained from a theoretical exponential curve, from the actual values.

3. Results

3.1. Laboratory-Scale Trials

The measurement of the temperature during the transfer of the olive pots revealed that there were no significant differences between the three cultivars at any of the two storage temperatures. The olives cooled at 5° C attained 7.1 ± 0.2 °C (Ti7) and the ones stored at 10.0 °C reached 12.2 ± 0.2 °C (Ti12). These values were taken as the initial temperatures at time 0, i.e., at the moment of immersion.

The average temperature of the paste from olives at 7.1 °C was 12.0 ± 0.2 °C (Tg7), while the olives at 12.2 °C reached 17.6 ± 0.4 °C (Tg12). No significant differences were observed among varieties. The mean values of Tg7 and Tg12 were considered in further calculations. The mean values of the temperature of the fruit at the distinct immersion times are represented in Figure 2.

The temperature of 'Arbequina' at T_i = 7.1 °C increased similarly in the three assayed water temperatures. A steep increase in the temperature was noticeable from the start, after which a flattening of the curves set in. At t15, fruit immersed in water at 35 °C was already 5 °C warmer than that immersed in water at 25 °C, increasing almost 7 °C at t60. A similar profile was observed at T_i = 12.2 °C, although with smaller differences between T25 and T35 at t15 and t60. The 'Cobrançosa' variety showed a similar temperature increase to that of 'Arbequina', characterized by a steep increase during the first 15 s, after which the temperature of the fruit flattened. However, the gap between the obtained fruit temperatures at T25 and T35 was less pronounced when compared to 'Arbequina' for both initial temperatures. The profiles of the 'Gordal' samples did not present a similar steep increase at the start, and, especially, the fruit with T_i = 12.2 °C. The spread between T25 and T35 reached only a few degrees at t15 while at t60 it widened to almost 5 °C.

The levels of significance (three-way ANOVA) of the different factors on the fruit temperature showed similar results for the three varieties (Table 2).

The initial fruit temperature (T_f), the temperature of the water (T_w), and the time of immersion (t) presented a highly significant effect on the fruit temperature at t. For the interaction of $T_f \times T_w$, no significant effect was observed for the 'Arbequina' and the 'Gordal' varieties. In the first case, the temperature increase during the first 15 s of immersion led to almost similar values at t15 for the trials at different T_f. From then on, and up to 60 s, the effect of T_f was hardly noticeable. In the second case, this correction is less pronounced at t15, and also attained a lower temperature when compared to the other varieties. During t15 and t60, the fruit temperature of the 'Gordal' variety evolved similarly although with a greater range between the different Tw than with the 'Arbequina' variety. Concerning the interactions between T_f and t, and $T_w \times$ t, a highly significant effect was observed, indicating that the effect of the initial fruit temperature, as well as the water temperature, on the fruit temperature at each t, varied for each t. Finally, when the interaction between the three factors was taken into account, no significant effect was observed in the case of 'Arbequina', due to highly similar progression at both T_f from t15 on.

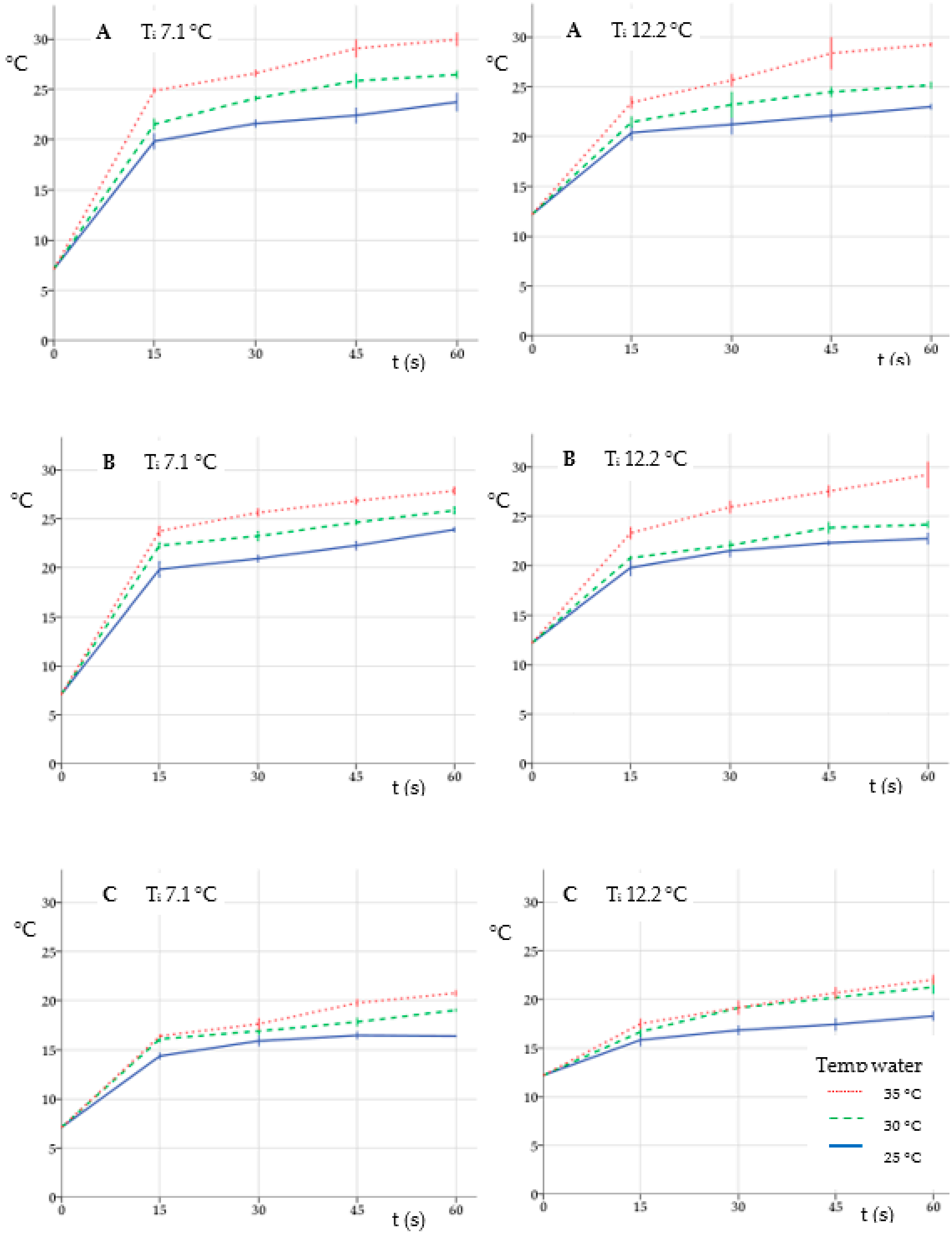

Figure 2. Evolution of the fruit temperature of 3 olive varieties (A: Arbequina; B: Cobrançosa; C: Gordal) at two initial temperatures (T_i) and immersed in moving water at different temperatures. Vertical bars express mean ± SD.

Table 2. Level of significance of the effect of the factors initial fruit temperature (T_f), temperature of the water (T_w), the time of immersion (t), and their interactions, on the fruit temperature for the three varieties ('Arbequina', 'Cobrançosa', and 'Gordal').

Variety	T_f	T_w	t	$T_f \times T_w$	$T_f \times t$	$T_w \times t$	$T_f \times T_w \times t$
Arbequina	0.001	0.000	0.000	0.254	0.000	0.000	0.405
Cobrançosa	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Gordal	0.000	0.000	0.000	0.059	0.000	0.000	0.000
All varieties	0.000	0.000	0.000	0.073	0.000	0.000	0.158

These observations were confirmed by the calculated mean of the different k values, for each variety and temperature (Table 1).

A one-way ANOVA revealed significant differences ($p < 0.001$) among varieties, while the post hoc Tukey test ($p < 0.05$) detected a significant difference between 'Arbequina' and 'Cobrançosa' varieties on the one hand, and the 'Gordal' variety on the other. A two-way ANOVA within each variety showed no significant effect of the initial temperature, the water temperature, or their interaction on the value of k, for none of the varieties. The RMSE ranged between 0.95 °C ('Gordal'; T_i = 7.1; T_w = 35 °C) and 2.85 °C ('Gordal'; T_i = 12.2 °C; T_w = 25 °C). The results of the different trials varied within each variety and initial fruit temperature (Figure 2). The deviations were largely due to the difference between the calculated and the measured values at t15. The higher values of the latter at t15 (data not shown) were a consequence of the applied measurement technique which implied a correction of the temperature of the paste, subtracting the temperature generated by the grinding (Equation (1)). During the time between taking out the fruit and the actual grinding, a certain amount of time went by, during which the temperature of the fruit could further evolve, leading unavoidably to a variation in the exact time. This time was relatively much higher at t15.

The results made clear that to raise the temperature of olives from 7 and 12 °C to 20–25 °C, respectively, it sufficed to submerge them into water at a temperature between 30 and 35 °C, respectively, for 10–20 s. However, this estimation did not take into account the specific industrial processing conditions. For example, the time needed to transport the olives from the washing tub to the grinder, during which they cool down. To take this unavoidable cooling into account, it was estimated that raising the water temperature by an additional 5–10 °C would be enough. At the same time, the results showed a possible solution, knowing that the average immersion time was calculated to be in the range of 15–20 s, equal to the calculated average washing time in an industrial washing tub. Finally, these water temperatures neither damaged the fruit tissues nor jeopardized the quality of the olives. Therefore, the water temperature in the washing tub was set to 40 °C for the industrial-scale trials.

3.2. Industrial-Scale Trials

The measurements during the two trials allowed us to monitor the fruit temperature during its processing, from the discharge at the reception yard of the olive oil mill up to entering the malaxer (Figure 1). The first trial was carried out with a batch of 2134 kg 'Arbequina' olives. On arrival, the fruit temperature was 12.8 ± 0.3 °C (T_{ar}). The outdoor temperature was 13.2 °C at 9:00 a.m. The processing of the fruit started at 9:45 a.m. and took 63 min. For the second trial, a batch of 4721 kg 'Picual' olives was used. On arrival, their temperature was 11.1 ± 0.2 °C (T_{ar}). The outdoor temperature at 9 a.m. was 11.3 °C. The cleaning process started at 9:30 a.m. All the fruit was processed and ground within 118 min. The processing times were slightly below the calculated capacity of the washing tub. The rate at which the olives left the hopper was regulated by lifting manually a small slider and controlled by an operator of the olive oil mill. The mean immersion time was adjusted accordingly to 26 ± 5 s.

During the first trial, the fruit temperature at the exit of the hopper (T_{dry}) increased from 13.2 °C at the first measurement at t3 to 16.8 at t55 (Figure 3). From t9 to t41, the temperature remained constant around 15 °C, from then on it rose consistently to attain its maximum at t55. The increase was a logical consequence of the natural warming up during the day.

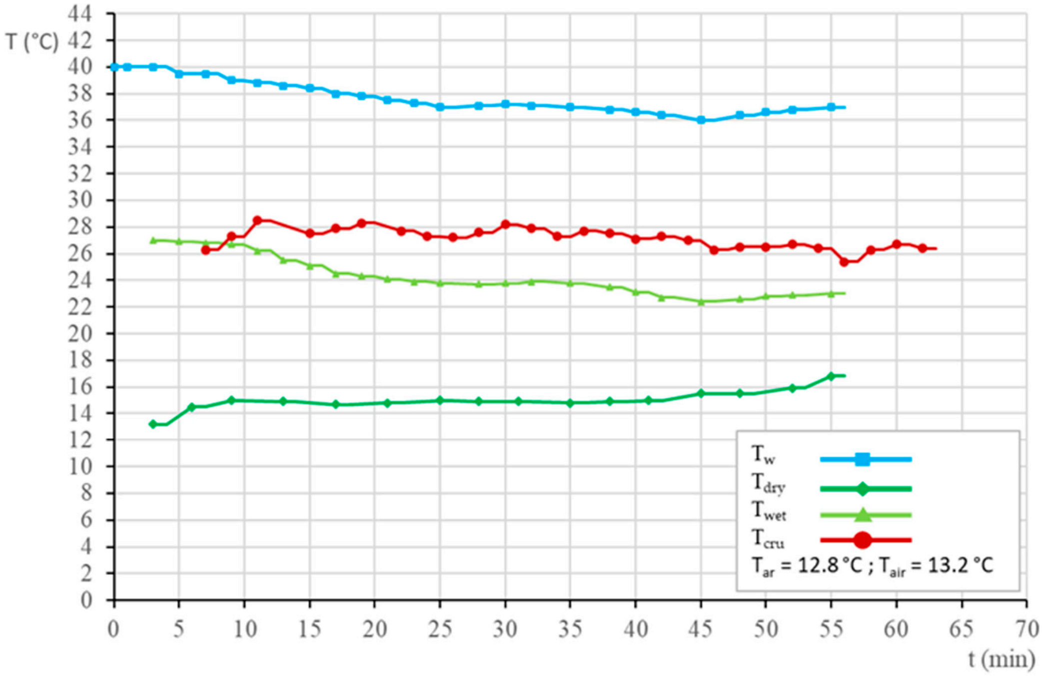

Figure 3. Evolution of the temperature during the processing of a batch of olives ('Arbequina' cv., 2134 kg). Each point represents a registration on one of the 4 different sites: the fruit temperature at the exit of the hopper (T_{dry}), the water temperature in the washing tub (T_w), the temperature exiting the washing tub (T_{wet}), and the temperature of the crushed olives (paste) before entering the malaxer (T_{cru}). On arrival, the fruit temperature was 12.8 ± 0.3 °C.

The washing tub was filled with warm water at 40 °C at the start of the processing. The different heaters were activated from the start to compensate for the cooling of the water. Notwithstanding, after a few minutes, the water started to cool down steadily to 37 °C at t25 and further to 36 °C at t45. At that moment it was decided to inject warm water into the tub, resulting in the temperature rising to 37 °C at t55. It was clear that the five heaters were not enough to keep the water at the desired temperature. In addition, it became clear that the water strains, produced within the tub, made it more difficult to warm the water from above.

The temperature of the olives measured after the washing (T_{wet}) followed a similar pattern of T_w. Starting at 27 °C at t3, the temperature declined from t9 and attained values just below 24 °C from t25 up to t35. From then on, a new pronounced decline set in, with the lowest value found at t45 (22.4 °C). Subsequently, the temperature rose again to 23 °C at t55. The fluctuations in the water temperature did have an immediate effect on the temperature of the olives and confirmed the efficacy of the used method. The data were also in agreement with the results of the laboratory experiments as they fell within the expected values.

Finally, the temperature of the olive paste just before entering the malaxer (T_{cru}) showed an equally consistent profile. The first olives were ground at t7, thus the measured temperature of 26.3 °C rose quickly to 28.5 °C at t11. From then on, the temperature maintained values between 28.3 and 27 °C up to t44, after which it declined to 25.4 °C at t56. In the final phase, the temperature rapidly attained values above 26 °C. The olives cooled down during their transport to the grinder since the ambient temperature was at least 10 °C lower than the fruit temperature. Meanwhile, the grinding generated heat, hence the olive paste attained the desired temperature for the malaxing (27 °C). Therefore, it sufficed to keep this temperature constant during the process without the need to heat-up the circulating water around the malaxer. The lower temperatures at the start of the process can be attributed to the heating of the metal grinder which absorbed the first generated heat produced by the grinding. Once stabilized, more heat was transferred to the paste. It was also observed that the major temperature shifts occurred with a delay of several minutes, being the time needed to transport the wet olives to the grinder. Consequently, it is shown that there exists a stable relationship between the temperature of the paste and the wet olives and, given the evenly strict relation between the fruit and the water temperature, also with the temperature of the latter.

During the second trial, the temperature of the fruit exiting the hopper (T_{dry}) remained constant at around 11 °C throughout the processing (Figure 4). The day temperature hardly rose during the forenoon and this meant that the fruit was not warmed up. Considering that the day temperature, as well as the temperature of the fruit on arrival, was several degrees lower than in the first trial, the water temperature was set to 45 °C instead of 40 °C. The heaters were controlled by a thermostat, connected to the thermometer probe in the grinder, but were activated from t0 to compensate for the cooling of the water. The supply of warm water was secured to correct this if necessary. The water temperature remained constant throughout the processing, except at t88, when it decreased to 44 °C, and t112, when temperature further declined to 43 °C. The temperature of the wet olives (T_{wet}) fell to 23 and 22 °C from t0 to t88. From then on, the temperature declined to 20 °C at t112. Similar to what was pointed out in the first trial, a strict relation between the T_w and T_{wet} was observed. The temperature of the paste during the whole grinding process reached around 28 °C, except for the first 5 min, where it was a few degrees lower due to the warming of the grinder. From t100, a minor decrease was observed, although without being below 27.2 °C at t110. This change was produced shortly after the temperature of the wet olives presented a small decline.

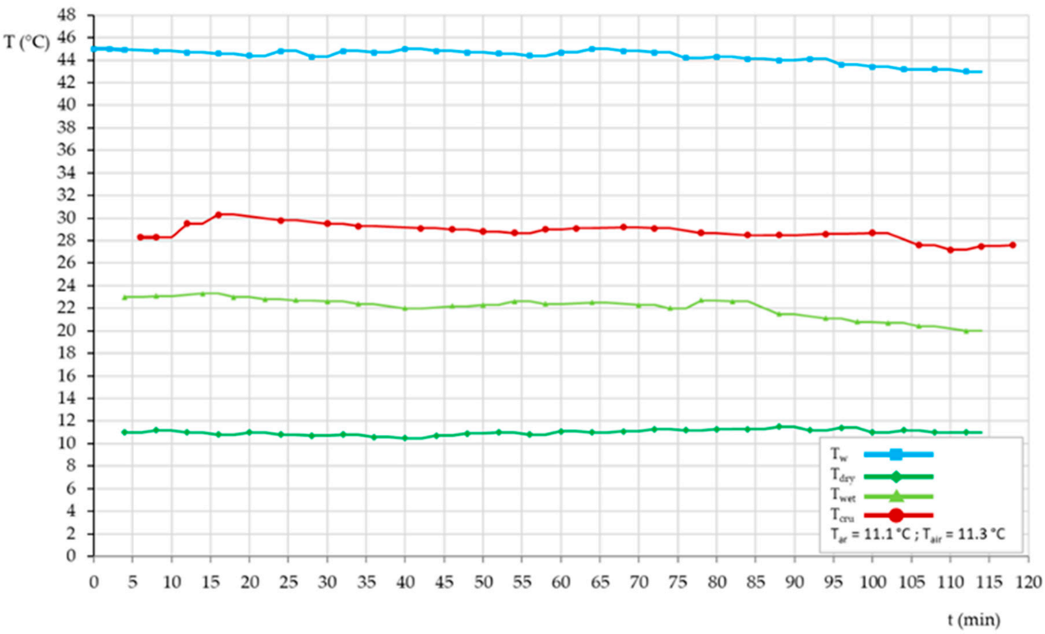

Figure 4. Evolution of the temperature during the processing of a batch of 'Picual' olives. Each point represents a registration on one of the 4 different temperature measurement points: fruit temperature at the exit of the hopper (T_{dry}), water temperature in the washing tub (T_w), temperature of the fruit exiting the washing tub (T_{wet}), and temperature of the crushed olives (paste) before entering the malaxer (T_{cru}). On arrival, the fruit temperature was 12.8 ± 0.3 °C. The outdoor temperature was 11.3 °C at 9 a.m.

4. Discussion

This work intended to investigate whether it was feasible to warm up olives at 10 °C or less during their immersion in the washing tub to obtain ground fruit at a temperature close to 27 °C before entering the malaxer. The laboratory experiment showed that for various varieties and water temperatures, this goal can be reached with an immersion time around 15 to 20 s and temperatures in a range of 35–40 °C, depending on fruit characteristics. The trials at the pilot plant did confirm this assumption on a pilot scale.

The temperature rise during the first 15 s stood out as the most significant observation in the first experiment, as well as the consistent separation of the curves at the different water temperatures. The spread between T25 and T35 varied according to the varieties. This indicated that the effect of the water temperature varied across the cultivars as well as across the initial fruit temperatures. The smaller the fruit and the greater the temperature difference, the greater the effect of the water temperature on the measured fruit temperature at t15. It was also observed that the warming up of the different fruit batches of each variety followed an exponential curve that flattened when the initial temperature of the fruit was higher or the water temperature lower. This shift was the most pronounced in 'Arbequina' as compared to 'Gordal'. 'Cobrançosa', on the contrary, showed only slight differences with the former. The ANOVA confirmed the highly significant effect of all the factors studied (variety, fruit temperature, water temperature, and time), and their interactions. The fact that no effect in the interaction between T_f and T_w was detected for 'Arbequina' can be explained by the more pronounced flattening of the curve from t15 (Figure 2). In the case of the 'Gordal' variety, this lack of interaction effect can be attributed to the greater variability that is observed within the samples at t60 (Figure 2).

The ANOVA applied to the values of the proportionality constant did confirm that they vary among the cultivars, given their different weights. However, they are not expected to present deviant values within each cultivar, as the constant k is specific for a given body and its surroundings, which was the case. The fact that there were no significant differences between 'Arbequina' and 'Cobrançosa', despite their difference in weight, can be explained using the results of an empirical study that determined the cooling rate of six different cultivars using thermal imaging [20].

It was shown that the relation between the cooling rate of olive fruit and the specific surface area can be characterized by a sigmoid (Boltzmann) function and suggested that the flattening of the curve can be attributed to the higher Biot number of the heaviest cultivar, 'Gordal', on one side, while the stone/flesh relation of the lightest fruit stabilized the cooling rate on the other side. The curve also revealed that other varieties with a heavier weight, such as 'Verdial', which had a mean weight of 2.95 ± 0.42 g, did present values that were close to the one observed for 'Koroneiki'. The values for k obtained for 'Arbequina' and 'Cobrançosa' varieties (mean weight of 0.84 ± 0.17 g and 2.75 ± 0.17 g, respectively) are thus comparable with the one obtained in the cited study, although in this case, the fruit was warmed up instead of cooled down. As thermodynamics works in the same way in both directions, these results do provide a reasonable explanation. However, the obtained results deviate strongly from the one published by other authors [36], who predicted a cooling time of 296 s to bring 'Gordal' olives from 17.7 °C to 4 °C in water at 0.46 °C. The mathematical calculations made by these authors, based on the biometrical and thermal characteristics of one olive, did not coincide with the empirically measured values obtained in the present two experiments.

The second experiment made clear that a large number of continuously changing variables influenced the heat exchange between the fruit and the different surroundings (air and water). As a result, accurate estimations of the required water temperature were not feasible. However, when the temperature in the grinder is constantly monitored with a thermostat, the temperature of the water can be perfectly adjusted to provide the necessary heat to the fruit. Overall, the data of the second trial do confirm the results of the first one. The lower temperature of the olive fruit was correctly predicted and corrected using a higher water temperature. The experience of the first trial meant that the water temperature could be better kept under control, even when dealing with olives at a lower initial temperature. The thermostat worked correctly but the heaters were not able to keep the temperature at the set level. On several accounts, warm water was added to the washing tub.

The used temperatures and the immersion time remained far below the conditions (3 min in 60 °C) that other authors [37] applied to study the blanching effect on the biosynthesis of olive oil aroma, as well as experiments in which heat treatment was applied to olive fruits to lower their bitterness, using water at 50 and 72 °C for 3 to 5 min [38–40]. As the fruit temperature measured right after the immersion (T_{wet}) was always lower than 25 °C after an average immersion time of 25 s, it can be assumed that modifications of the aroma profile, as well as the decrease in oxidative stability and polyphenol content, as pointed out in these studies, were not present.

The results of the flash heat treatment experiments demonstrated that bringing the temperature to the ideal malaxation temperature with a heat exchanger leads to a reduction in the malaxing time without significant modifications of the organoleptic characteristics [26,28,31]. Providing paste at the desired temperature further guarantees a constant temperature from the moment the malaxation starts, which is equivalent to extending the malaxation time by an interval equal to the time that the mixer employs to heat all the olive paste mass [25]. Whether this is also the case when the fruit is warmed before the crushing needs to be experimentally confirmed. Nevertheless, given the similar conditions of the paste entering the malaxer when compared with a heat exchanger, a strong argument can be made that this will be the case. Similarly, a significant impact on the plant's working capacity, oil yield, and oil quality can be expected to be the same as those achieved with a heat exchanger [26]. Bringing the olives to an adequate temperature significantly reduces the need to warm up the wall of the malaxer, and thus implies a lower energy cost during the malaxation process. At the same time, it avoids the risk of overheating the fraction that is in direct contact with the heated wall which, once attaining 45 °C, produces irreversible and detrimental changes to the chemical composition of the oil [41].

The energetic cost to keep a washing tub at a temperature between 35 and 45 °C will largely depend on its capacity, isolation, and ambient temperature. The potential sources and equipment for this necessary heat need further study but direct heating with electric resistances or a heating system based on an external heat exchanger may be a feasible, economic solution that can even be introduced to existing washing tubs. The latter solution can easily be combined with a filtering and UV system to keep the circulating water clean and free of bacterial contamination.

5. Conclusions

To optimize the malaxing process, flash heat treatments are proposed to control the temperature of the paste before entering the malaxer. While the commercially available and experimental systems are intended to control the temperature of the ground olives or paste, the present work studied the possibility to adjust the temperature of the intact fruit before grinding. At the laboratory level, the possibilities to warm up olive fruit were explored under different conditions. Based on these results, it was concluded that it is feasible to bring the fruit to the ideal temperature for malaxing without applying excessive heat and within a time frame that is adjusted to the characteristics of an industrial washing tub. Two trials at a pilot plant showed a satisfactory result and confirmed the estimated values. Meanwhile, the trials made clear that the washing tub needed to be equipped with a warming system that is safe and efficient to guarantee a constant water temperature during the processing. Once the equipment is installed, a quick and precise adjustment of the paste can be attained by controlling the temperature of the washing water through a thermostat that monitors the entering paste. The presented solution has the potential to be easily integrated into an existing production line with lower investment costs than those of the commercially available and experimental flash heat treatment systems.

Author Contributions: Conceptualization, E.P.; methodology, E.P.; software, E.P.; validation, J.M.G.M., M.d.C.F.F. and R.R.S.-G.; formal analysis, E.P. and R.R.S.-G.; investigation, E.P.; resources, J.M.G.M.; data curation, E.P.; writing—original draft preparation, E.P. and J.F.G.M.; writing—review and editing, J.M.G.M., M.d.C.F.F., R.R.S.-G. and J.F.G.M.; visualization, E.P.; supervision, J.M.G.M., M.d.C.F.F. and R.R.S.-G.; project administration, E.P. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Acknowledgments: We thank the Del Cetino Olive Farm and their personnel for their collaboration, as well as Fernando Martínez, Paco Hidalgo, and Cristobal Hidalgo and the staff of the experimental plant of the Instituto de la Grasa for the technical assistance.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Rallo, L.; Díez, C.M.; Morales-Silero, A.; Miho, H.; Priego-Capote, F.; Rallo, P. Quality of olives: A focus on agricultural preharvest factors. *Sci. Hortic.* **2018**, *233*, 491–509. [CrossRef]
2. Famiani, F.; Arinelli, D.; Urbani, S.; Al Hariri, R.; Paoletti, A.; Rosati, A.; Esposto, S.; Selvaggini, R.; Taticchi, A.; Servilli, M. Harvesting system and fruit storage affect basic quality parameters and phenolic and volatile compounds of oils from intensive and super-intensive olive orchards. *Sci. Hortic.* **2020**, *263*, 109045. [CrossRef]
3. Sola-Guirado, R.R.; Castro-García, S.; Blanco-Roldán, G.L.; Jiménez-Jiménez, F.; Castillo-Ruiz, F.J.; Gil-Ribes, J.A. Traditional olive tree response to oil olive harvesting technologies. *Biosyst. Eng.* **2014**, *118*, 186–193. [CrossRef]
4. D'Imperio, M.; Gobbino, M.; Picanza, A.; Costanzo, S.; Della Corte, A.; Mannina, L. Influence of harvest and period on olive oil composition: An NMR and statistical study. *J. Agric. Food Chem.* **2010**, *58*, 11043–11051. [CrossRef]
5. Morales-Silero, A.; García, J.M. Impact assessment of mechanical harvest on fruit physiology and consequences on oil physicochemical and sensory quality from 'Manzanilla de Sevilla' and 'Manzanilla Cacereña' super-high-density hedgerows. A preliminary study. *J. Sci. Food Agric.* **2015**, *95*, 2445–2453. [CrossRef]
6. García, J.M.; Yousfi, K. The postharvest of mill olives. *Grasas Aceites* **2006**, *57*, 16–24. [CrossRef]
7. Brkić, K.; Lukić, M.; Novoselić, A.; Krapac, M.; Lukić, I. Olive Fruit Refrigeration during Prolonged Storage Preserves the Quality of Virgin Olive Oil Extracted Therefrom. *Foods* **2020**, *9*, 1445. [CrossRef] [PubMed]
8. Lorite, I.J.; Gabaldón-Leal, C.; Ruiz-Ramos, M.; Belaj, A.; de la Rosa, R.; León, L.; Santos, C. Evaluation of olive response and adaptation strategies to climate change under semi-arid conditions. *Agric. Water Manag.* **2018**, *204*, 247–261. [CrossRef]
9. Benlloch-González, M.; Sánchez-Lucas, R.; Bejaoui, M.A.; Benlloch, M.; Fernández-Escobar, R. Global warming effects on yield and fruit maturation of olive trees growing under field conditions. *Sci. Hortic.* **2019**, *249*, 162–167. [CrossRef]
10. Fernández-Bolaños, J.; Heredía, A.; Vioque, B.; Castellano, J.M.; Guillén, R. Changes in cell-wall-degrading ezyme activities in stored olives in relation to respiration and ethylene production. *Z Lebesm. Unters. Forsch. A.* **1997**, *204*, 293–299.
11. Di Serio, M.G.; Giansante, L.; Di Loreto, G.; Faberi, A.; Ricchetti, L.; Di Giacinto, L. Ethyl esters versus fermentative organoleptic defects in virgin olive oil. *Food Chem.* **2017**, *219*, 33–39. [CrossRef]
12. Inarejos-García, A.M.; Gómez-Rico, A.; Salvador, M.D.; Fregapane, G. Influence of malaxation condition on virgin olive oil yield, overall quality and composition. *Eur. Food Res. Technol.* **2009**, *228*, 671–677. [CrossRef]
13. Clodoveo, M.L. Malaxation: Influence on virgin olive oil quality. Past, present and future. An overview. *Trends Food Sci. Technol.* **2012**, *25*, 13–23. [CrossRef]
14. Peri, C. Quality Excellence in Extra Virgin Olive Oils. In *Olive Oil Sensory Science*; Monteleone, E., Langstaff, S., Eds.; John Wiley: Chichester, UK, 2014; pp. 3–32.
15. Selvaggini, R.; Esposto, S.; Taticchi, A.; Urbani, S.; Veneziani, G.; Di Maio, I.; Sordini, B.; Servili, M. Optimization of the temperature and oxygen concentration conditions in the malaxation during the oil mechanical extraction process of four Italian olive cultivars. *J. Agric. Food Chem.* **2014**, *62*, 3813–3822. [CrossRef] [PubMed]
16. Dourou, A.M.; Brizzolara, S.; Meoni, G.; Tenori, L.; Famiani, F.; Luchinat, C.; Tonutti, P. The inner temperature of the olives (cv Leccino) before processing affects the volatile profile and the composition of the oil. *Food Res. Int.* **2020**, *129*, 108861. [CrossRef] [PubMed]
17. Amirante, P.; Cini, E.; Montel, E.; Pasqualone, A. Influence of mixing and extraction parameters on virgin olive oil quality. *Grasas Aceites* **2001**, *52*, 198–201. [CrossRef]
18. Caponio, F.; Gomes, T.; Summo, C.; Pasqualone, A. Influence of the type of olive-crusher used on the quality of extra virgin olive oils. *Eur. J. Lipid Sci. Technol.* **2003**, *105*, 201–206. [CrossRef]

19. Veneziani, G.; Esposto, S.; Taticchi, A.; Urbani, S.; Selvaggini, R.; Di Maio, I.; Sordini, B.; Servili, M. Cooling treatment of olive paste during the oil processing: Impact on the yield and extra virgin olive oil quality. *Food Chem.* **2017**, *221*, 107–113. [CrossRef] [PubMed]
20. Plasquy, E.; García, J.M.; Florido, M.C.; Sola-Guirado, R.R. Cooling rate estimation of six olive cultivars using thermal imaging. *Agriculture* **2021**, *11*, 164. [CrossRef]
21. Brosnan, T.; Sun, D. Precooling techniques and applications for horticultural products. A Review. *Int. J. Refrig.* **2001**, *24*, 154–170. [CrossRef]
22. Elansari, A.M. Design aspects in the precooling process of fresh produce. *Fresh Prod.* **2009**, *3*, 49–57.
23. Plasquy, E.; Blanco-Roldán, G.; Florido, M.C.; García, J.M. Effects of an integrated harvest system for small producers on the quality of the recollected olive fruit. *Grasas Aceites* **2021**, *72*, e436. [CrossRef]
24. García, J.M.; Gutiérrez, F.; Barrera, M.; Albi, M. Storage of mill olives on an industrial ccale. *J. Agric. Food Chem.* **1996**, *44*, 590–593. [CrossRef]
25. Amirante, P.; Clodoveo, M.L.; Dugo, G.; Leone, A.; Tamborrino, A. Advance technology in vigin olive oil production from traditional and de-stoned pastes: Influence of the introduction of a heat exchanger on oil quality. *Food Chem.* **2006**, *98*, 797–805. [CrossRef]
26. Esposto, S.; Veneziani, G.; Taticchi, A.; Selvaggini, R.; Urbani, S.; Di Maio, I.; Sordini, B.; Minnocci, A.; Sebastiani, L.; Servili, M. Flash Thermal Conditiong of Olive Pastes during the Olive Oil Mechanical Extraction Process: Impact on the structural modifications of pastes and oil quality. *J. Agric. Food Chem.* **2013**, *61*, 4953–4960. [CrossRef]
27. Fiori, F.; Di Lecce, G.; Boselli, E.; Pieralisi, G.; Frega, N.G. Effects of olive paste fast preheating on the quality of extra virgin olive oil during storage. *LWT Food Sci. Technol.* **2014**, *58*, 511–518. [CrossRef]
28. Leone, A.; Esposto, S.; Tamborrino, A.; Romaniello, R.; Taticchi, A.; Urbani, S.; Servilli, M. Using a tubular heat exchanger to improve the conditioning process of the olive paste: Evaluation of yield and olive oil quality. *Eur. J. Lipid Technol.* **2016**, *118*, 308–317. [CrossRef]
29. Bejaoui, M.A.; Sánchez-Ortiz, A.; Sánchez, S.; Jiménez, A.; Beltrán, G. The high-power ultrasound frequency: Effect on the virgin olive oil yield and quality. *J. Food Eng.* **2017**, *207*, 10–17. [CrossRef]
30. Cecchi, L.; Bellumori, M.; Corbo, F.; Milani, G.; Clodoveo, M.L.; Mulinacci, N. Implementation of the sono-heat-exchanger in the extra virgen olive oil extraction process: End-user validation and analytical evaluation. *Molecules* **2019**, *24*, 2379. [CrossRef]
31. Tamborrino, A.; Romaniello, R.; Caponio, F.; Squeo, G.; Leone, A. Combined industrial olive oil extraction plant using ultrasounds, microwave, and heat exchange: Impact on olive oil quality and yield. *J. Food Eng.* **2019**, *245*, 124–130. [CrossRef]
32. Clodoveo, M.L. Industrial ultrasound applications in the extra-virgin olive extraction proces: History, approaches, and key questions. *Foods* **2019**, *8*, 121. [CrossRef]
33. Stillitano, T.; Falcone, G.; De Luca, A.I.; Piga, A.; Conte, P.; Strano, A.; Gulisano, G. A life cycle perspective to assess the environmental and economic impacts of innovative technologies in extra virgin olive oil extraction. *Foods* **2019**, *8*, 209. [CrossRef] [PubMed]
34. Kalogianni, E.P.; Georgiou, D.; Hasanov, J.H. Olive oil processing: Current knowledge, literature gaps, and future perspectives. *J. Am. Oil Chem. Soc.* **2019**, *96*, 481–507. [CrossRef]
35. Uceda, M.; Frías, L. Harvest dates: Evolution of the fruit oil content, oil composition and oil quality. In Proceedings of the 2nd International Olive Oil Seminary, Cordoba, Spain, 6–17 October 1975; pp. 125–130.
36. Cuesta, F.J.; Alvarez, M.D. Mathematical modelling for heat conduction in stone fruits. *Int. J. Refrig.* **2017**, *80*, 120–129. [CrossRef]
37. Luaces, P.; Pérez, A.G.; Sanz, C. Effect of the blanching process and olive fruit temperature at milling on the biosynthesis of olive oil aroma. *Eur. Food Res. Technol.* **2006**, *224*, 11–17. [CrossRef]
38. García, J.M.; Yousfi, K.; Oliva, J.; García-Diaz, M.T.; Pérez-Camino, M.C. Hot water dipping of olives (*Olea europaea*) for virgen oil debittering. *J. Agric. Food Chem.* **2005**, *53*, 8248–8252. [CrossRef] [PubMed]
39. Cruz, S.; Yousfi, K.; Oliva, J.; García, J.M. Heat Treatment Improves Olive Oil Extraction. *J. Am. Oil Soc.* **2007**, *84*, 1063–1068. [CrossRef]
40. Yousfi, K.; Moyano, M.J.; Martinez, F.; Cayuela, J.A.; García, J.M. Postharvest heat treatment for olive oil debittering at the industrial scale. *J. Am. Oil Soc.* **2010**, *87*, 1053–1061. [CrossRef]
41. Boselli, E.; Di Lecce, G.; Strabbioli, R.; Pieralisi, G.; Frega, N. Are virgin olive oils obtained below 27 C better than those produced at higher temperatures? *LWT Food Sci. Technol.* **2009**, *42*, 748–757. [CrossRef]