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Heat Treatment Improves Olive Oil Extraction

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Abstract To determine the effect of hot water pre-treatment on olive oil extraction, six cultivars of olive fruit (*Olea europaea* L. cvs. "Arbequina", "Hojiblanca", "Lechín", "Manzanilla", "Picual", and "Verdial") were heated at 50, 55, and 60 °C prior to laboratory scale oil extraction. Heat treatment resulted in higher oil extraction than unheated control samples. Quality parameters of the oils were not significantly ($P < 0.05$) affected by these treatments; however, oil stability and bitterness intensity were reduced and pigment content was increased through pre-heat treatment. This process may be incorporated economically into olive oil processing.

Keywords *Olea europaea* L. · Quality · Yield · Stability · Olive · Extraction

Introduction

Olive oil can only be classified as virgin olive oil if it has been extracted exclusively by physical methods such as pressing or centrifugation. Before using these operations, the olive fruits must be ground in a mill and the resulting paste kneaded in a malaxer to facilitate the coalescence of microscopic oil drops released from the olive cells. During these essential stages of oil extraction, if the fruit presents certain physical characteristics, such as high firmness and moisture content or damage due to chilling or parasitic infections, the micro oil droplets may become trapped by the remnants of cell walls and plasmatic membranes of the milled olive cells, constituting stable emulsions. Under these circumstances, oil cannot be extracted from the olive paste by the normal physical methods used by the industry and a significant amount of oil is lost as industrial waste [1]. The olive fruits at the green-mature stage of ripening are especially susceptible to forming these pastes that pose difficulties in oil extraction (difficult pastes). "Hojiblanca" and "Picual" fruits, widely used in Spain for oil production, may present this problem at any stage of ripening [2].

To alleviate this situation, micronized talc (hydrated magnesium silicate, particle size <40 μm) is commonly used by the Spanish olive oil industry as a coadjuvant to treat "difficult pastes" during kneading to achieve improved oil yield. This treatment blocks the emulsifying action of the cell wall derivatives, more oil with less water and suspended solids content can be recovered [3]. The action of talc appears to be purely mechanical and exits the process as solid waste. The improved oil yield achieved by using this system is economically important since the highest increases obtained are around 3% [2]. The time or temperature of kneading can be increased in order to break the emulsion and to improve the oil extraction, but such actions may result in significant deterioration in the oil quality [4].

Olive tissues contain enzymes that are capable of catalyzing the oxidation of lipids with molecular oxygen [5]. Lipoxygenase is responsible for the initial stages of oil oxidation with the formation of fatty acid hydroperoxides leading to an increased peroxide value, and hydroperoxide lyases continue the oxidative process, cutting these compounds, and producing short-chain aldehydes and ketones, which increases the absorbance in the ultraviolet region of the oils and are related to the development of rancidity [6, 7]. These enzymes are released during fruit milling and can

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be activated during paste kneading. Increased time and temperature of kneading accelerate these activities, causing oil deterioration. To avoid oxidation, the use of controlled atmospheres during oil extraction is being studied, but maintaining hermetically closed industrial systems is very difficult and the use of the N_2 necessary to maintain an inert atmosphere is expensive, because the feed of olive fruits containing O_2 is continuous in the industrial processing lines.

Immersion of olive fruits in hot water has been proposed as a means of controlling the level of bitterness of the virgin olive oil extracted from unripe olives [8]. This treatment is based on inactivating the enzymes involved in developing bitterness during oil extraction to enable early harvest. In spite of the high temperatures used (≥60 °C), this treatment did not significantly compromise the parameters legally established to measure oil quality, such as acidity, UV absorption, peroxide value or sensory panel tests. These results suggested that the enzymes responsible for oil deterioration were also inactivated with this heat treatment prior to olive milling. Furthermore, significantly higher oil yield (14.2%) was obtained from unripe “Manzanilla” fruits compared to non-treated fruit (10%). This approach allows higher processing temperatures without affecting the quality of the virgin oil extracted. In the present study, dipping the fruit in water at temperatures ≤60 °C was evaluated as a treatment to improve to the extraction of olive oil using the most cultivated olive varieties in Andalusia (southern Spain) at the ripening stages normally used for oil extraction.

Experimental Procedures

Plant Material

Olive fruits (*Olea europaea*) cvs. “Arbequina”, “Hojiblanca”, “Lechín” “Manzanilla”, “Picual”, and “Verdial” were harvested at the Ripening Indices (RI) of 2.8, 3.8, 4.5, 1.1, 3.4, and 2.5, respectively, during the 2004–2005 season in three different orchards of Andalusia (Spain), and transported the same day to the Instituto de la Grasa. About 32 kg of healthy fruits were selected from each variety. The olives were randomly distributed into four treatment groups, each containing 8 kg of fruits.

Evaluation of the Maturity Level

From each variety four replicates of 100 fruits were randomly taken from each group to evaluate maturity. The traditional system to evaluate the level of olive maturation (RI) was calculated by the same evaluator for each variety by using subjective evaluation of the color of the olive skin and flesh [9].

Heat Treatment

Three of the four groups were immersed into a 400-L thermostatic water bath at 50, 55, and 60 °C for a period of 3 min each prior to oil extraction. Each treatment was carried out in triplicate. The fourth group received no pretreatment.

Oil Yield, Total Oil Content and Physical Extractability

After heat treatment, each 8-kg olives group of each replicate and treatment was milled and distributed into eight batches of 1 kg each. A sample of 700 g of paste was taken from each batch and weighed in a metallic pitcher. The paste of each pitcher was independently homogenized by using a spatula, 1% micronized talc was added, and the oil was extracted by using an “Abencor” analyzer (Comercial Abengoa S.A., Seville, Spain). This unit, consisting of three basic elements: a mill, a thermobeater (30 °C, 30 min) and a pulp centrifuge (1,500*g*, 1 min), simulated the industrial process of virgin olive oil production [10]. After centrifuging, the oil was decanted into a graduated cylinder to measure the volume (mL) obtained and to calculate oil yield, which was expressed as the percentage of the fresh weight considering the value 0.915 g mL^{-1} as the mean olive oil density. Subsequently, the eight extracted oils of each replicate and treatment were mixed, filtered and stored at –20 °C under N_2 atmosphere until analysis.

From each replicate of each treatment, eight 50-g samples of surplus paste were separately weighed in previously weighed capsules and dried at 105 °C to constant weight. The oil of the dried paste was solvent extracted with hexane by using the Soxhlet method to determine the total oil content of the paste as a percentage of the paste fresh weight. The extractability obtained by the different treatments tested was calculated based on the percentage of oil physically extracted from the total oil content of each variety tested.

Oil Analysis

The titratable acidity, peroxide value, and the extinction coefficients at 232 and 270 nm (K_{232} and K_{270}) were determined on the extracted oils according to the European Union standard methods [11]. The overall sensory quality of each oil sample was evaluated by a panel of eight trained

(≥5 years experience) tasters according to a nine-points scale, “1” being the poorest quality possible and “9” for the best. Oxidative stability was measured by using the Rancimat method, which evaluates the time (h) of resistance to oxidation of 3-g oil samples exposed to streams of dry air at heated to 100 °C [12]. The pigment contents of the oils was evaluated by determining their absorbances at 470 and 670 nm for carotenoids and chlorophyll, respectively, and the results were expressed as mg/Kg [13]. Intensity of oil bitterness (IB) was determined by the absorbance at 225 nm [14].

Statistical Analysis

One-way analysis-of-variance (ANOVA) was carried out on all data for each variable studied, independent for each variety to test the effects each treatment. If a significant ($P < 0.05$) effect was identified by ANOVA, separation of the means was carried out using Tukey’s test ($P < 0.05$).

Results and Discussion

Physical Extractability of the Oil

In each one of the varieties tested, at least two of the pre-treatment temperatures gave significantly higher oil extractabilities than when no heat was applied (Table 1). With the exception of “Manzanilla” fruit, where the maximum of extractability occurred at 55 °C, the values of extractability increased with temperature of the heat treatment used. Increasing the processing temperature, due to the previous immersion at increasing temperatures, improved oil extractability.

Pre-treated olives commence milling at >40 °C and cool to 30 °C in the temperature-controlled malaxer, whereas untreated olives commence milling at 18 °C (ambient temperature) and increase to 30 °C during kneading. As a consequence, the increased oil extractability produced was probably due to the use of higher kneading temperature. Nevertheless, without prior fruit heating and enzymatic inactivation, the simple increase of kneading temperature would significantly deteriorate the quality of oil subsequently extracted [4].

“Manzanilla” olives are the most cultivated table olives in Andalusia, and are always harvested in the green-mature ripening stage. The surplus, which cannot be used by the table olive industry, is destined for oil production. For this reason, the use of fruits of this variety with higher maturity level for oil extraction is not habitual. Unripe fruits are highly consistent with high contents of cellulose and pectins in the cell walls of their mesocarp cells. Furthermore, the ratios between oil content and the contents of polar lipids and membrane-bound proteins are lower in these fruits than in riper fruits. These circumstances make these fruits especially susceptible to formation of oil emulsions during physical extraction. Prior heat treatment should favor oil extraction of these fruit, but excess temperature could denature proteins, which may induce oil emulsion formation. Probably, emulsion formation is the reason for different effects observed for the heat treating “Manzanilla” olives. Similarly, this reason may account for the results previously published [8], using heat treatment at ≥60 °C to debitter oil in immature (RI = 1.0) “Manzanilla”, “Picual” and “Verdial” olives observed that increasing immersion temperature progressively reduced oil yield.

Oil Quality

The parameters established to measure the quality of virgin olive oil were not compromised by the heat treatment (Table 2). Free acidity, absorbance at 270 nm, and sensory panel tests were not significantly affected by heat treatment. In previous work [8], a reduction of free acidity in oils extracted from “Manzanilla” olives that were pre-treated at 60 °C was observed, but this difference, although significant, was very slight. A similar reduction of free acidity in the oil extracted was found after exposing

Table 1 Oil contents of pre-heat treated and non-treated olives by chemical and physical extraction (Mean ± SD, $N = 3$)

Variety[a]	Oil content (%)	Physical extractability (100 × oil yield/oil content)			
		Control	50 °C	55 °C	60 °C
Arbequina	28.5 ± 1.0	83.9 ± 1.2 c	82.6 ± 1.2 c	85.1 ± 1.4 b	88.3 ± 1.3 a
Hojiblanca	14.5 ± 1.2	43.7 ± 1.3 c	46.5 ± 1.1 b	49.3 ± 1.3 a	51.4 ± 0.9 a
Lechín	19.8 ± 1.3	83.1 ± 0.8 c	89.0 ± 0.9 b	91.6 ± 0.7 a	91.6 ± 0.8 a
Manzanilla	16.6 ± 1.4	71.7 ± 0.6 b	72.3 ± 1.0 b	80.8 ± 0.7 a	79.6 ± 0.9 a
Picual	21.8 ± 1.5	78.7 ± 0.9 c	81.4 ± 0.9 b	81.0 ± 1.0 b	83.4 ± 1.1 a
Verdial	20.0 ± 1.5	86.1 ± 1.2 b	89.0 ± 1.3 a	88.9 ± 1.2 a	89.5 ± 1.6 a

[a] For each variety, two values followed by different small letter are significantly different ($P < 0.05$) according to the Tukey’s test

"Picual" and "Shemlalli" olive fruit to microwaves [15]. It was probable that the internal olive lipase only acted in the non-treated pastes of these two varieties during oil extraction, whereas in the other ones, this enzyme did not play any part.

Although heat treatment did not significantly affect sensory traits of the oils for any of the varieties tested, increased temperature used clearly coincided with a slight, but systematic, reduction in sensory values. This observation could result from heat-induced reduction of aroma compounds of the olive oils. In previous work, a decrease of the volatile compounds content in the olive oils was found as consequence of the prior immersion of unripe "Manzanilla", "Picual" and "Verdial" olives in water at ≥60 °C [16].

The absence of any effect due to heat treatment on the absorbance at 270 nm suggests that hydroperoxide lyases were not active during oil extraction of all fruits tested. In general, the peroxide and K_{232} values behaved similarly in response to heat treatment. Although these values were significantly decreased in the oils extracted from treated "Hojiblanca" and "Lechín" olives, peroxide and K_{232} values of the other oils were not affected by heat treatment. In a previous work, in contrast, a decrease of both parameters was found after heating "Manzanilla" olives at 60 °C [8]. These parameters are related to the first step of oil oxidation, since peroxide value measures fatty acid hydroperoxides contents and K_{232} evaluates the oil content of conjugated fatty acid, the step prior to the incorporation of atmospheric oxygen into these molecules. Because the enzyme lipoxygenase is responsible for hydroperoxide formation, the response of these parameters to heat treatment should be similar to and consistent with inhibition of this enzymatic activity. The inhibition of lipoxygenase activity during olive processing induced by microwave pretreatment of ripe "Picual" and "Shemlalli" fruits has been previously described [15].

The oxidative stabilities of extracted oils were only significantly affected by heat treatment in the case of "Manzanilla" olives (Table 3). Although oxidative stabilities of oils obtained from "Verdial" fruits were slightly diminished as dipping temperature increased, no effects on oil stability were observed in the other experiments.

Table 2 Quality of olive oil extracted from fruit following pre-treatment at different temperatures (Mean ± SD, $N = 3$)

Variety[a]	Treatment	Acidity (% oleic)	K_{232}	K_{270}	Peroxide value (mg O_2 kg^{-1})	Sensory panel test
Arbequina	Control	0.50 ± 0.13	1.42 ± 0.23	0.09 ± 0.04	5.2 ± 1.2	7.3 ± 0.4
	50 °C	0.45 ± 0.12	1.35 ± 0.24	0.09 ± 0.05	4.4 ± 0.9	7.2 ± 0.5
	55 °C	0.42 ± 0.12	1.48 ± 0.20	0.08 ± 0.05	4.4 ± 1.1	6.9 ± 0.5
	60 °C	0.39 ± 0.13	1.34 ± 0.19	0.08 ± 0.05	4.8 ± 0.9	6.9 ± 0.6
Hojiblanca	Control	0.09 ± 0.03	1.35 ± 0.10 a	0.09 ± 0.05	5.9 ± 0.9 a	7.2 ± 0.4
	50 °C	0.11 ± 0.03	1.10 ± 0.12 b	0.09 ± 0.04	2.9 ± 0.7 b	7.0 ± 0.5
	55 °C	0.10 ± 0.03	1.08 ± 0.10 b	0.09 ± 0.05	3.2 ± 0.9 b	7.0 ± 0.4
	60 °C	0.10 ± 0.03	1.08 ± 0.11 b	0.08 ± 0.05	3.3 ± 0.8 b	6.8 ± 0.5
Lechín	Control	0.23 ± 0.04	1.68 ± 0.18 a	0.10 ± 0.05	12.3 ± 1.4 a	7.1 ± 0.4
	50 °C	0.21 ± 0.05	1.56 ± 0.16 a	0.09 ± 0.05	9.4 ± 1.2 b	6.9 ± 0.4
	55 °C	0.22 ± 0.06	1.20 ± 0.14 b	0.08 ± 0.06	6.9 ± 1.4 c	7.0 ± 0.4
	60 °C	0.22 ± 0.04	1.19 ± 0.15 b	0.08 ± 0.04	6.9 ± 1.6 c	6.8 ± 0.5
Manzanilla	Control	0.58 ± 0.06	1.48 ± 0.20	0.11 ± 0.04	4.4 ± 0.7	6.7 ± 0.3
	50 °C	0.52 ± 0.07	1.55 ± 0.15	0.10 ± 0.04	5.2 ± 0.5	6.8 ± 0.4
	55 °C	0.53 ± 0.08	1.59 ± 0.17	0.09 ± 0.05	5.4 ± 0.4	6.5 ± 0.5
	60 °C	0.53 ± 0.07	1.51 ± 0.18	0.10 ± 0.04	4.8 ± 0.8	6.5 ± 0.3
Picual	Control	0.54 ± 0.07	1.34 ± 0.15	0.10 ± 0.04	6.3 ± 0.8	7.2 ± 0.4
	50 °C	0.59 ± 0.08	1.38 ± 0.18	0.10 ± 0.03	6.2 ± 0.8	7.0 ± 0.4
	55 °C	0.58 ± 0.08	1.34 ± 0.16	0.10 ± 0.04	6.2 ± 0.6	6.7 ± 0.5
	60 °C	0.59 ± 0.09	1.38 ± 0.17	0.10 ± 0.03	6.2 ± 0.6	6.8 ± 0.5
Verdial	Control	0.29 ± 0.07	1.70 ± 0.18	0.15 ± 0.05	9.5 ± 0.9	7.3 ± 0.4
	50 °C	0.26 ± 0.08	1.72 ± 0.20	0.14 ± 0.05	8.7 ± 1.0	7.0 ± 0.5
	55 °C	0.31 ± 0.08	1.70 ± 0.20	0.14 ± 0.05	8.8 ± 0.9	7.1 ± 0.5
	60 °C	0.28 ± 0.07	1.62 ± 0.19	0.14 ± 0.05	8.3 ± 0.8	7.0 ± 0.5

[a] For each variety, two values followed by different letters are significantly different ($P < 0.05$) according to the Tukey's test. Absence of a letter following means indicate no significant effect ($P < 0.05$) by ANOVA

In general, the stabilities of oils extracted from treated "Manzanilla" olives were still considerable, clearly higher than stabilities of oils extracted from non-treated fruits of other olive varieties. Reduced oxidative stabilities of "Manzanilla", "Picual" and "Verdial" oils have been previously observed after heat pre-treatment at ≥60, ≥64 and ≥68 °C, respectively [8].

Hot water pre-treatment significantly increased pigment (chlorophyll and carotene) contents of the oils extracted from all olive varieties tested. These results confirmed the previous observations when using air heating at ≥40 °C [17], or using immersion at ≥60 °C [18]. It is likely that heat treatment reduced enzyme activity that is responsible for pigment degradation. In spite of the increase in the carotene content induced by the heat pre-treatment, this oil showed a more intense green color than that obtained from control fruit, since the "greening" induced by the increase in chlorophylls had a larger effect on the final color. This olive oil "greening" obtained by a relatively inexpensive physical method may be important to improve the oil appearance, because consumers prefer green to yellow olive oils. Besides, the increase of carotene content supposes an improvement of the nutritional value of the oil, because carotenes are natural antioxidants with vitamin activity.

As was also observed with oxidative stabilities, the IB of the oils were only significantly reduced as a consequence of heat treatment in those extracted from "Manzanilla" olives, whereas no significant effect on IB values due to heat treatment was observed in the oils obtained from other varieties. Both parameters, stability and IB, are related to phenol content [19, 20]. Oxidative stability is a good estimation of the content of antioxidant phenols, and IB values give an approximation of the presence of phenols responsible for oil bitterness. Both parameters give more information about the level of active phenols in the oils than the total phenol content, which includes active as well as non-active phenols.

Table 3 Effects of different heat pre-treatments on oxidative stability, pigment contents, and the intensity of bitterness of olive oils physically extracted from different olive varieties (Mean ± SD, $N = 3$)

Variety[a]	Treatment	Stability (h)	Chlorophyll (mg kg $^{-1}$ oil)	Carotene (mg kg $^{-1}$ oil)	Intensity of bitterness
Arbequina	Control	34.8 ± 2.7	10.12 ± 1.22 b	8.40 ± 0.80 b	2.0 ± 0.2
	50 °C	35.0 ± 2.4	22.95 ± 2.13 a	17.23 ± 1.64 a	1.8 ± 0.3
	55 °C	34.8 ± 2.6	23.56 ± 2.22 a	17.97 ± 1.72 a	1.7 ± 0.3
	60 °C	35.1 ± 2.5	24.90 ± 2.31 a	18.85 ± 1.67 a	1.7 ± 0.3
Hojiblanca	Control	27.9 ± 2.8	11.12 ± 0.38 b	9.43 ± 0.52 b	2.2 ± 0.2
	50 °C	28.0 ± 2.6	41.18 ± 2.50 a	21.06 ± 1.55 a	2.2 ± 0.3
	55 °C	27.6 ± 2.4	42.36 ± 2.22 a	22.62 ± 1.75 a	2.1 ± 0.2
	60 °C	29.3 ± 2.8	40.64 ± 2.65 a	20.07 ± 1.68 a	2.0 ± 0.2
Lechín	Control	24.2 ± 2.6	4.50 ± 0.33 b	4.10 ± 0.24 b	2.3 ± 0.2
	50 °C	23.9 ± 2.4	7.90 ± 0.85 a	8.42 ± 0.56 a	2.2 ± 0.2
	55 °C	24.1 ± 2.7	8.59 ± 0.76 a	9.02 ± 0.61 a	2.0 ± 0.2
	60 °C	24.0 ± 2.8	8.92 ± 0.82 a	8.88 ± 0.58 a	2.0 ± 0.2
Manzanilla	Control	95.4 ± 6.4 a	22.51 ± 2.45 b	13.24 ± 1.74 b	7.9 ± 0.4 a
	50 °C	80.4 ± 6.1 b	62.65 ± 5.12 a	24.91 ± 2.64 a	6.5 ± 0.5 b
	55 °C	79.6 ± 6.2 b	64.47 ± 5.56 a	25.49 ± 2.89 a	5.4 ± 0.5 c
	60 °C	77.6 ± 7.5 b	65.72 ± 5.29 a	26.42 ± 2.85 a	4.8 ± 0.4 d
Picual	Control	86.6 ± 6.8	13.20 ± 1.69 b	10.84 ± 1.56 b	3.3 ± 0.5
	50 °C	87.5 ± 7.3	45.62 ± 3.82 a	20.46 ± 2.18 a	3.2 ± 0.6
	55 °C	88.0 ± 8.1	46.89 ± 3.46 a	21.21 ± 2.25 a	3.1 ± 0.6
	60 °C	87.0 ± 7.5	48.23 ± 3.37 a	21.83 ± 2.28 a	2.8 ± 0.6
Verdial	Control	58.4 ± 6.3	33.89 ± 2.25 b	20.23 ± 1.05 b	4.4 ± 0.5
	50 °C	57.9 ± 6.1	104.35 ± 2.21 a	40.23 ± 2.24 a	4.2 ± 0.5
	55 °C	56.3 ± 5.8	105.42 ± 2.19 a	41.64 ± 2.62 a	4.1 ± 0.5
	60 °C	55.9 ± 6.5	106.79 ± 2.18 a	42.47 ± 2.35 a	4.1 ± 0.5

[a] For each variety, two values followed by different letters are significantly different

($P < 0.05$) according to the Tukey's test. Absence of small letter following mean values indicate no significant effect ($P < 0.05$) among treatments detected by ANOVA

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