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Research Article

Optimization Olive Oil Extraction by Pre-heat Treatment Olive Fruits

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Abstract: The present investigation was conducted on the aim to determine the optimum pre-heat treatment (temperature and time) for olive fruits, at early stages of maturity, in order to enhance oil extraction. The heat treatment was performed of two Jordanian cultivars "NabaliBaladi" (NB) and "NabaliMuhassan" (NM) by dipping the olive fruits in heated water at 50, 55, 60 and 65°C, for 2, 3 and 4 minutes at each heating temperature. Olives are then coarsely ground in fruit mill, the olive paste pressed in hydraulic press at 120 k N/cm^2 for 45 min and finally the oil-water mixture was centrifuged to separate oil. On the basis of results obtained, the yield of oil was significantly ($p \leq 0.05$) increased, the increase for NB ranged 0.2-4.0% and the optimum heat treatments were 55°C/ 3-4 min and 60°C/ 2-3 min, while the increase for NM ranged 0.6-3.8% and the optimum heat treatment was 60°C/ 3-4 min. The acidity of oils obtained with pre-heat treatment had slightly improved, while there were some adverse effect regarding peroxide values, UV absorption, oxidative stability and sensory evaluation. Phenolic compounds and α-tocopherols contents significantly decreased, but their values were within the legal limits. The chlorophyll pigment intensity increased significantly in the extracted oil through raising pre-heat treatment of olives, the increase was 3.6-fold and 2.8-fold of NB and NM, respectively, compared to the olive oil control, where the rise of carotene was 2.5-fold in both varieties. These results conclude that pre-heat treatment processing is beneficial for quantity and quality of olive oil produced.

Keywords: α-tocopherols, extractability, oxidative stability, oil yield; oil quality parameters, phenolic compounds

INTRODUCTION

In recent years, there has been an increasing demand for olive oil throughout the world. This increase is ascribed to the awareness of its high nutritional value and its potential health benefits. Olive oil has a main role in the human diet wherever olive trees have grown. It is well-known that long life and good health of the Mediterranean basin public is associated with their traditional diet of olive oil which contains a wide range of valuable bioactive compounds. Those compounds include: linoleic acid and oleic acid. Additionally, high levels of natural antioxidative (phenols, tocopherols and carotenoids), sterols, volatile compounds as well as its pleasant sensory (Visioli and Galli, 1998; Covas, 2007; Najafian *et al*., 2009). According to these facts, it is important to improve olive oil recovery without sacrificing other quality attributes. Specific enhancement aides discussed will help to address the growing global need for this commodity.

Oil accumulation in olive fruit, occurs rapidly during the first stages of fruit maturation whereas a slower increase occurs later in the season (Mailer *et al*., 2007; Gucci *et al*., 2004). About 76% of oil located in the vacuoles is in a free state but the remaining quantity (~24%) is trapped in the mesocarp cells. The oil in the mesocarp cell takes the form of minute droplets that constitute an emulsion state with the colloids. Thus, a large amount of the oil is not accessible by the traditional extraction procedures and is therefore lost in the byproducts (Sharma and Sharma, 2007).

To maximize olive oil output from the enclosed olive fruit tissues, farmers traditionally wait until advanced fruit maturity. At this point, the fruit cell walls will be softer and facilitate more oil from the olive fruit cells during extraction. Despite the benefit, advanced fruit maturity it also brings some consequences. During the maturation time, some of the beneficial oil quality properties such as: oil acidity, peroxide value, phenolic compound contents, volatile constituents, tocopherols and pigments have significantly degraded.

Several methods have been proposed to improve oil extraction from olive fruit, at early harvesting season in order improve both quantity and quality of the

olive oil. Among these methods is the use of specific enzymes, which breakdown the cell walls structure and improve the release of oil from mesocarp cells ((Najafian *et al*., 2009; Mailer *et al*., 2007; Sharma and Sharma, 2007). Others suggested coadjutant agents, such as micronized talc (Valdivia *et al*., 2008) and calcium carbonate (Espínola *et al*., 2009). Some researchers preheated the fruit prior to the start of the oil extraction process, which will soften the cell walls and thereby facilitate the release of oil from the olive cells (Cruz *et al*., 2007).

Pre-heating of olive fruit has numerous purposes. Besides the improvement of extraction output, pre-heating can control bitterness intensity of olive oil extracted from unripe olives (García *et al*., 2005; Yousfi *et al*., 2010; García *et al*., 2001). Additionally, pre-heating can modify olive oil aroma (Luaces *et al*., 2006; Pérez *et al*., 2003) and darken the oil pigments (Cruz *et al*., 2007; Yousfi *et al*., 2010; Luaces *et al*., 2005).

The aim of this study was to optimize the pre-heating olive fruits process and evaluate the effects of these processes on the quality of olive oil extracted for the two most predominant varieties olive fruit in Jordan. These were the NabaliBaladi (*NB*) and NabaliMuhassan (*NM*) vareties picked at early mature stages. Chosen quality indices included free fatty acid content, peroxide value and spectrophotometric characteristics in the ultraviolet region, tocopherols content, total phenolic compounds, oil pigments and sensory evaluation.

MATERIALS AND METHODS

Seventy two kilo gram of healthy fruits from each two abundant varieties in Jordan (Nabali Baladi, Nabali Muhassan), were handpicked at the green mature stage of ripening (Ripening Index were 2.9, 2.7 for *NB*, *NM*, respectively) during crop season 2012/2013. The trees are rain-fed and located in Bani Kananeh district (north of Jordan). The 72 kg of olive fruits were divided into 3 lots (each 26 kg). The first lot of olive fruits were randomly distributed into 13 batches of 2 kg each, one of these batches served as control, while the remaining 12 batches were underwent heat treatment. The heat treatment consisted of dipping them in a 30 L thermostatic water bath as followed: 1st at 50°C/2 min, 2nd at 50 °C/3 min, 3rd at 50°C/4 min, 4th at 55°C/2 min, 5th 55°C/3 min, 6th at 55°C /4 min, 7th at 60°C/2 min, 8th at 60°C/3 min, 9th at 60°C/4 min, 10th at 65/°C/2 min, 11th at 65°C/3 min and 12th 65°C/4 min.

The heated fruits were cooled at ambient temperature (about 18°C). They were then coarsely ground in a Hobart mincer (Hobart, London) that was provided with a 5.0 mm hole diameter sieve. Next they were kneaded at about 15 rpm/45 min in order allow the oil drops to joint together to form the largest droplets of oil to release maximum amount of oil. Olive paste (about 2 kg) was filled in 15 cloth mats designed as disks. The disks were stacked on top of each other into a stainless steel hydraulic piston, forming a pile (the mat disks diameter were little less of piston diameter). Hydraulic pressure was applied on the disks at 120 k N/cm^2 for 45 min. The percolating oily was obtained (oil and vegetation water) and centrifuged at 5000 rpm for 20 min to separate the oil. A control batch was processed with no pre- heat treatments.

The percentage of the oil obtained from the two varieties of olives, were calculated as dry weight. Note that the olive fruits moisture was 48.3, 51.6% in NB and NM, respectively. This was determined by the oven method of keeping the ground samples at 105°C until a constant weight. The remaining two lots were treated as the first one.

Each extracted oil lot had one replicate, which means that we had three replicates. The three values we obtained as a result of quantity and quality evaluation underwent statistical analysis.

Total oil content and extractability: The total oil content (% w/w) was estimated in a prior crushed and dried olive fruit sample, with a Soxhlet fat extraction apparatus using petroleum ether (bp 40-60°C) as solvent. The oil in the preheated samples was extracted by a hydraulic pressing method. The extractability for all treatments was calculated by the following formula:

$$\text{Extractability} = \frac{\text{Weight of oil extracted by hydraulic method}}{\text{Weight of oil content in the control extracted by Soxhlet method}} \times 100$$

Quality indices determinations:

Sensory characteristics: These were evaluated by taste panel composed of seven judges according to the methods described by the International Olive Oil Council (IOOC) (1997). Each oil sample was evaluated by a seven-member trained panel, using a descriptive nine-point hedonic scale were 9 was "likes extremely" and 1 was "dislikes extremely".

Titratable acidity: These were determined according the official methods of the European Union Commission (1991) and expressed as a percent free fatty acids on the basis of oleic acid.

Peroxide value: This was carried out following the official methods of the European Union Commission (1991) and expressed as a value in milliequivalents of free oxygen per kilogram of oil (mEq O_2/kg).

K_{232} and K_{270} extension coefficients: These were estimated according the official methods of the European Union Commission (1991) and were

calculated from the absorption of 232 nm and 270 nm, respectively. A spectrophotometer (Spectro UV-VIS Double beam PC, UVD-2950. Labomed, INC. USA) was used with a 1% solution of oil in cyclohexane.

Tocopherols: These were quantified by the HPLC following the method of the American Oil Chem. Soc. (AOCS) (1989). This method consisted of a Knauer pump and a Knauer and Smartline 2500 UV Detector (Advanced Scientific Instrument, Berlin, Germany). Olive oil samples were dissolved in *n*-hexane 0.36% (w/v), 20 µL of the solution was injected into a silica column (thermoQuest, 10 µ particle size, 4.0 mm ID×30 cm). The mobile phase was hexane-isopropanol (99:1). The flow rate was 1.3 mL/min. The wavelength was programmed at 295 nm.

Total phenolic content: This was determined using the Folin-Ciocalteu method (Gutfinger, 1981). Briefly explained, 10 g of olive oil was dissolved in 50 ml hexane. Twenty milliliters of aqueous methanol (60%) were added and vigorously mixed for 2 min. The methanolic phase was removed and placed in a beaker each time after the two phases were separated. The combined extracts were layed out to dry in a vacuum rotary evaporator at 70ºC. The residue was dissolved in 1 ml methanol. One-tenth of a milliliter of methanolic extract was placed into a 10 mL volumetric flask. Five milliliters of distilled water and 0.25 mL FolinCiocalteau (2N) were added and mixed well for 3 min. One milliliter of sodium bicarbonate (Na_2CO_3 35%) was added and the flask was filled with distilled water up to the mark. The specific absorbance of the blue color formed was measured, after 1 h, at 725 nm (Spectro UV-VIS Double beam PC, UVD-2950. Labomed, INC. USA). A reference curve was prepared using gallic acid as the most representative of phenolic standards and the data expressed as mg gallic acid/kg of oil.

Chlorophyll pigment: This was evaluated according the method of the American Oil Chem. Soc. (AOCS) (1989). Briefly, the spectrophotometer (Spectro UV-VIS Double beam PC, UVD-2950. Labomed, INC. USA) cell was filled with oil heated to 30°C and the absorbance was read at 630, 670 and 710 nm, using carbon tetrachloride as a blank. The results were calculated by the following equation:

$$\text{Chlorophyll (mg/kg)} = \frac{A_{670} - (A_{630} + A_{710})}{0.101L}$$

where,
A : Absorbance
L : Thickness of cuvette, (1 cm)

Carotene pigment: This was measured from the absorbance of oil diluted in cyclohexane, measured at the wavelength 445 nm. The proportion of carotenoids was expressed by β-carotene content calculated using the following equation:

$$\beta\text{-carotene (mg/kg)} = 383E/PC$$

where,
E : The difference in measured absorbance values for oil sample and cyclohexane
P : Optical path length (cm)
C : Concentration of the sample (g/100 mL)

Oxidative stability: This was measured by using the Rancimat method, which evaluates the time (h) of resistance to oxidation of the oil sample (5g) exposed to a stream of dry air passing through the samples (22 L/h) which were heated to 115°C (892 Professional Rancimat, Swiss made).

Statistical analysis: All experiments were carried out in triplicate. The data were statistically analyzed by using the statistical package for Social Science (SPSS), version 19.0, 2010, Chicago. IL. One way analysis of variance (ANOVA) test was applied to test difference between the treatments followed by mean separation using Duncan's Analysis. Finding with a p-value of ≤ 0.05 were considered to be statically significant.

RESULTS AND DISCUSSION

The olive oil content in both NB and NM was 51.9 and 44.3%, respectively, expressed on dry matter basis. This was in agreement with the results published by Al-Rousan (2004) and Al-Maaitah *et al*. (2009). Data in Table 1 revealed the improvement of oil extractability from olives pre-treated at a rising treatment temperature. The maximum extractability in NB occurred at 55°C for 3 or 4 min and at 60°C for 2 or 3 min, with no significant differences between the four conditions ($p<0.05$). The extractability reached 79.9, 79.7 and 79.5, 79.8%, respectively, in contrast to 75.9 % in the untreated samples. In the NM variety, the highest extractability was 70.7, 70.9% occurred at a heat treatment of 60°C for 3, 4 min, respectively, while it was 67.1% in the control samples.

The increasing extraction efficacy obtained was believed to be a result of softening the olive tissue cells via pre-heat treatment. This process caused the breakdown of the pockets of emulsion and denatured the protein portion of colloid drops, which spread through the cytoplasm, consequently, releasing a greater quantity of oil. The percentage increase of oil recovery was 4, 3.8% for NB and NM, respectively. Cruz *et al*. (2007) reported that the increasing extractability ranged from 0.5% to 8.5%, when they pre-heated 6 varieties of olives at 60°C for 3 min, in contrast to the control. While Najafian *et al*. (2009) and Sharma and Sharma (2007) founded that the increase

Table 1: Extractability of oil for heat treated samples and unheated by pressing extraction

Temperature °C/time min	Extractability = Oil yield/ oil content×00		Sensory evaluation	
	NB	NM	NB	NM
Control (unheated)	75.9±0.92^{c}	67.1±1.24^{c}	8.2±0.4^{a}	7.9±0.2^{a}
50/2	75.7±0.90^{c}	67.7±1.42^{c}	8.3±0.4^{a}	7.9±0.2^{a}
50/3	76.2±0.95^{b}	67.7±1.30^{c}	8.3±0.3^{a}	7.8±0.2^{a}
50/4	76.8±0.92^{b}	68.2±1.31^{c}	8.1±0.4^{a}	7.8±0.2^{a}
55/2	76.1±0.91^{b}	68.2±0.87^{c}	8.3±0.3^{a}	7.8±0.3^{a}
55/3	79.9±1.10^{a}	68.3±1.00^{c}	8.2±0.4^{a}	7.9±0.3^{a}
55/4	79.7±1.00^{a}	69.1±0.98^{b}	8.0±0.2^{a}	7.5±0.2^{ab}
60/2	79.6±0.98^{a}	69.3±0.95^{b}	8.0±0.2^{a}	7.6±0.4^{ab}
60/3	79.8±0.96^{a}	70.7±1.51^{a}	7.8±0.2^{b}	7.5±0.3^{ab}
60/4	79.1±0.95^{ab}	70.9±1.07^{a}	7.6±0.2^{b}	7.2±0.3^{b}
65/2	78.9±0.89^{ab}	70.3±1.22^{ab}	7.2±0.2^{c}	6.9±0.2^{c}
65/3	78.7±0.88^{ab}	69.9±1.21^{ab}	7.0±0.3^{c}	6.8±0.2^{c}
65/4	79.0±0.94^{ab}	69.9±1.12^{ab}	6.9±0.2^{bc}	6.6±0.2^{c}

Column values with the same subscripts were not significantly different ($p \leq 0.05$); *:Values are the average of three replicates ± the standard deviation, Means with different subscript in the same column are significant at ($p \leq 0.05$)

Table 2: Quality of olive oil for NB and NM extracted after heating of olive fruits at different temperatures and times

Temperature °C/time min	Acidity(% Oleic)		Peroxide Value(mEqO_2/kg)		Specific extinction at 232 nm and 270 nm $E_{1\,cm}^{1\%}$			
	NB	NM	NB	NM	NB K_{232}	NB K_{270}	NM K_{232}	NM K_{270}
Control	0.31±0.04	0.28±0.06	8.9±1.2	6.1±0.90^{c}	1.71±0.22	0.27±0.08	1.46±0.21^{c}	0.26±0.07
50/2	0.26±0.03	0.24±0.07	8.4±1.2	6.2±1.10^{c}	1.75±0.19	0.30±0.08	1.48±0.21^{c}	0.27±0.07
50/3	0.24±0.03	0.25±0.08	8.1±1.3	5.8±0.84^{c}	1.60±0.17	0.28±0.08	1.52±0.22^{c}	0.27±0.07
50/4	0.23±0.04	0.25±0.10	8.5±1.3	5.6±0.89^{c}	1.74±0.21	0.32±0.09	1.60±0.^{22bc}	0.28±0.07
55/2	0.25±0.02	0.30±0.10	7.9±1.1	5.7±0.90^{c}	1.67±0.25	0.28±0.08	1.52±0.22^{c}	0.27±0.07
55/3	0.23±0.03	0.27±0.11	8.6±1.2	5.9±0.91^{c}	1.55±0.18	0.24±0.06	1.54±0.20^{c}	0.27±0.07
55/4	0.26±0.03	0.23±0.09	9.3±1.3	6.6±0.95^{c}	1.61±0.21	0.32±0.08	1.57±0.22^{bc}	0.25±0.06
60/2	0.25±0.02	0.30±0.08	7.5±1.2	11.6±1.20^{bc}	1.64±0.17	0.25±0.07	1.55±0.21^{bc}	0.25±0.06
60/3	0.24±0.03	0.25±0.10	9.3±1.4	11.9±1.25^{b}	1.54±0.15	0.25±0.07	1.58±0.20^{bc}	0.25±0.06
60/4	0.24±0.03	0.26±0.09	9.8±1.5	12.8±1.31^{a}	1.69±0.17	0.26±0.08	1.86±0.25^{a}	0.27±0.07
65/2	0.25±0.02	0.25±0.10	8.2±1.4	12.9±1.29^{a}	1.59±0.16	0.24±0.06	1.79±0.28^{a}	0.27±0.07
65/3	0.24±0.03	0.24±0.08	9.5±1.4	12.7±1.30^{a}	1.63±0.18	0.28±0.08	1.70±0.29^{b}	0.24±0.05
65/4	0.26±0.02	0.27±0.10	9.8±1.4	12.2±1.26^{b}	1.65±0.18	0.29±0.09	1.64±0.27^{b}	0.26±0.06

Column values with the same subscripts were not significantly different ($p \leq 0.05$); *: Values are average of three replicates ± the standard deviation, Means with different subscripts in the same column are significant at ($p \leq 0.05$)

was 0.9-2.4%, 2.7-9.6%, respectively, when they used different enzymes to maximize olive oil recovery from different olive varieties. In this study, the percentage of yield increase was thought to be linked to both temperature and time heated. The rising yield can be considered a significant value for industrial processing.

As noted, the trend of extractability was declined when heat treatment was greater than 60°C/4 min. This might be due to the denaturated cellular structure of the olive which subsequently formed an emulsion state and thus reduced the oil recovery as Yousfi *et al*. (2010) explained in their work.

The sensory evaluation of each sample was accomplished by a panel of seven trained tasters using a descriptive nine-point intensity scale. As revealed in Table 1, the heat treatments did not significantly adversely affect oil flavor until 60°C/2 min treatment in NB. Comparatively the NM cultivar the heat treatment detrimentally affected at 60°C/4 min. The negative affect after mentioned treatments on gustatory properties of the oil was slight, keeping a good acceptable value. The receding sensory characters were due to deactivated lipoxygenase (LOX) enzymes that happened through the heat treatment process. The LOX-in active state-participates in the biosynthesis process of the olive oil aroma compounds (Luaces *et al*., 2006). This result was consistent with the findings of Cruz *et al*. (2007), Luaces *et al*. (2006) and Pérez *et al*. (2003).

Table 2 shows the chemical alterations of the oil quality obtained from control and pre-heated samples. The variation of acidity, for both NB and NM, were not significant in any heat treatment group. The acidity values were well within the normal reference values and legal limits as established by the International Olive Oil Council (IOOC) (1997). This might be due to inactivation of olive fruit tissue enzymes by heat treatments and the low temperature used throughout the entire extraction process. Similarly, results are in agreement with previous literature reported about exposing olive fruits to warm water and microwave preheats treatments (Cruz *et al*., 2007; García *et al*., 2005; Yousfi *et al*., 2010; García *et al*., 2001; Farag *et al*., 1997).

The peroxide values for NB oils (Table 2), were not affected by heat treatment since the values were in moderate range (7.5-9.8 mEq O_2/kg oil). Comparatively, the peroxide values for NM oils were stable (5.6-6.6 mEq O_2/kg oil) up to 55°C/4 min. They

Table 3: Effect of preheat treatment on olive oil contain of α–tocopherol, total phenolic compound and pigment contents

Treatment temperature °C/time min.	α–Tocopherols mg/ kg		Phenolic compounds (mg gallic acid equiv/kg oil)		Chlorophyll (mg/kg)		Carotene (mg/kg)	
	NB	NM	NB	NM	NB	NM	NB	NM
Control	178±5.9^{a}	159±5.3^{a}	324±8.1^{a}	270±5.5^{a}	14.52±0.31^{e}	11.47±0.22^{d}	9.82±0.15^{d}	10.23±0.22^{d}
50/2	175±5.4^{a}	156±4.7^{a}	299±8.3^{a}	271±5.6^{a}	25.18±0.51^{d}	19.64±0.28^{c}	18.21±0.27^{c}	16.51±0.28^{c}
50/3	169±5.1^{b}	155±3.7^{a}	284±5.3^{b}	263±5.4^{a}	26.02±0.53^{d}	20.89±0.26^{c}	18.68±0.28^{c}	16.36±0.28^{c}
50/4	152±5.2^{c}	148±3.3^{b}	278±5.4^{b}	263±4.9^{a}	29.87±0.58^{d}	24.18±0.30^{c}	18.96±0.28^{c}	17.54±0.30^{c}
55/2	160±6.1^{b}	160±4.8^{a}	279±5.5^{b}	272±5.0^{a}	31.70±0.58^{c}	26.54±0.32^{c}	18.89±0.29^{c}	18.78±0.30^{bc}
55/3	157±5.3^{b}	157±4.1^{a}	278±5.0^{b}	258±5.1^{a}	34.40±0.61^{c}	27.30±0.33^{b}	19.65±0.30^{c}	19.12±0.31^{b}
55/4	155±6.0^{c}	151±4.5^{b}	276±5.1^{b}	256±4.9^{a}	38.63±0.61^{bc}	29.65±0.31^{b}	21.08±0.30^{b}	20.43±0.33^{b}
60/2	159±5.2^{b}	158±3.4^{a}	271±4.9^{b}	269±5.0^{a}	37.48±0.60^{bc}	28.33±0.33^{b}	21.67±0.31^{b}	20.11±0.32^{b}
60/3	152±4.1^{c}	157±3.4^{a}	269±4.9^{b}	261±4.5^{b}	40.82±0.63^{b}	28.45±0.32^{b}	22.31±0.32^{b}	20.06±0.32^{b}
60/4	154±4.6^{c}	145±3.8^{b}	269±5.0^{b}	252±4.6^{b}	45.57±0.69^{b}	28.23±0.36^{b}	23.14±0.32^{a}	21.00±0.35^{b}
65/2	152±5.0^{c}	142±3.0^{c}	267±4.7^{b}	258±5.2^{c}	52.91±0.70^{a}	31.40±0.38	22.94±0.30^{b}	24.39±0.38^{a}
65/3	155±5.1^{c}	144±3.7^{b}	258±4.8^{c}	245±5.3^{c}	52.82±0.71^{a}	32.45±0.39^{a}	24.63±0.33^{a}	25.81±0.41^{a}
65/4	150±4.5^{b}	141±3.2^{c}	260±4.4^{c}	240±5.0^{c}	57.68±0.60^{a}	32.34±0.38^{a}	25.29±0.35^{a}	26.14±0.44^{a}

Column values with the same subscripts were not significantly different ($p \leq 0.05$); *: Values are average of three replicates ± the standard deviation, Means with different subscripts in the same column are significant at ($p \leq 0.05$)

then rose up and reached 12.9 mEq O_2/kg oil at temperature 65°C/2 min. Nevertheless, this value was still lower than the maximum indicated by the International Olive Oil Council (IOOC) (1997). These results were in agreement with the fact, that heat treatment deactivates native enzymes present in the olive tissues, such as lipoxygenase, that are capable of catalyzing the oxidation of heat treatment (Cruz *et al*., 2007). Furthermore, these results reflected on the absorbance at 232 nm and 270 nm, which were not altered through the heat treatment processing in both NB and NM olive oil. The consistency of these absorbances was a result of limited quantity of aldehyde and ketone accumulation due to deactivated native enzymes. Although, there were almost uniform effects of the heat treatment on peroxide and K_{232}in NM oil, these results are consistent with the data reported earlier by other investigators (Cruz *et al*., 2007; García *et al*., 2005; García *et al*., 2001; Farag *et al*., 1997).

Tocopherols are the primary antioxidants that protect oils against oxidative rancidity and α-tocopherol composes 95% of all tocopherol forms (Tasioula-Margari and Okogeri, 2001). Commonly, the amount of tocopherol in oil depends not only on its presence in the olives, but on other various factors such as: olive cultivar, fruit ripening stage, post-harvesting and processing conditions (Beltrán *et al*., 2005). Table 3 depicted the values of α-tocopherol in unheated and heated treatment samples; making it clear that heat treatment caused a slight decrease of tocopherol content. In NB and NM controls, the content of α-tocopherol was 175 and 159 mg/kg oil, respectively. While it was 150 and 141 mg/kg of oil extracted from the olives which were exposed to highest heat treatment. These results are consistent with the data reported earlier (Yousfi *et al*., 2010; Pérez *et al*., 2003; Beltrán *et al*., 2005; Cunha *et al*., 2006). Moreover, these values were in agreement with data previously reported for virgin olive oils ranging from 90 to 300 mg/kg (Tasioula-Margari and Okogeri, 2001).

Phenolic compounds have antioxidant properties. Additionally, they possess other qualities that affect the healthy andorganoleptic aspects of olive oil (Servilli *et al*., 2003). Table 3 shows that, unheated NB and NM olives contain 324 and 270 mg/kg oil of polyphenols, respectively. The declining amount of polyphenol content was apparent as heat treatment increased in both varieties. However, the decreasing amount of polyphenols in the NM variety that occurred throughout increased heat treatment happened with less intensity than in the NB variety. The decrease observed in both varieties was continuous as the heat treatment increased, until they reached 240 and 212 mg/kg of oil, at the final testing parameter (65°C/4 min), in NB and NM, respectively. These results were consistent with the data reported earlier by García *et al*. (2001), Servilli *et al*. (2003) and Ranalli *et al*. (2001). This was probably due to the increased oxidation of phenolic compounds to the polyphenol oxidase and peroxidase activities when the temperature was high (Servilli *et al*., 2003).

The decreasing amount of phenolic compound coincides with a decline in sensory score that occurred to oil extracted from preheated olives as shown in Table 1. However, the phenolic compound content in all olive oil samples studied, throughout all conditions, were in agreement with the data reported by Andrewes *et al*. (2003) which mentioned that the usual value of phenolic compounds for virgin olive oil ranged between 100 and 300 mg gallic acid equiv/kg of oil.

The preheating process of olives significantly increased the intensity of the main pigments (chlorophyll and carotene) present in oil extracted from all levels of heat treatments in both NB and NM. The increase continued as both time and temperature increased. The greatest chlorophyll value was observed at the highest heat treatment, where it reached 4-fold and 3-fold in NB and NM, respectively. The obtained results are in agreement with those obtained by Cruz *et al*. (2007), Yousfi *et al*. (2010) and Lauces *et al*. (2005), who reported that the increase of oil pigment extracts from olives exposed to heat treatment was due to the reduction of the lipoxygenase activity.

Table 4: Determination of resistance to oxidation in olive oil extracted from preheat treatment of olive expressed in hours

Treatment	NB	NM
Control	48.0±0.71[a]	30.2±0.42[a]
50/2	47.3±0.72[a]	30.8±0.41[a]
50/3	47.0±0.70[a]	30.0±0.40[a]
50/4	47.1±0.72[a]	31.0±0.43[a]
55/2	47.5±0.73[a]	29.7±0.38[a]
55/3	47.5±0.72[a]	28.7±0.38[b]
55/4	45.9±0.70[a]	27.5±0.35[b]
60/2	46.1±0.71[a]	27.8±0.36[b]
60/3	45.2±0.69[b]	27.3±0.36[b]
60/4	42.8±0.64[c]	27.6±0.34[b]
65/2	42.2±0.64[c]	25.4±0.33[c]
65/3	40.6±0.61[c]	25.6±0.33[c]
65/4	40.1±0.62[c]	24.3±0.31[c]

Column values with the same subscripts were not significantly different ($p \leq 0.05$); *: Values are the average of three replicates ± the standard deviation, Means with different subscript in the same column are significant at ($p \leq 0.05$)

The color of olive oil impacts consumer purchases. Consumers desire olive oil with a slight intense green color rather than a yellowish-green color (Cruz *et al*., 2007). The oil color we obtained from olives which were exposed to temperatures above 60°C was deep green. This seemed to be a negative response. Chlorophyll acts to promote the oxidation of oil in the presence of light, while it acts as an antioxidant in the presence of dark (Kiritsakis and Dugan, 1985). So the notable oil content of chlorophyll can be considered a positive property if the oil is stored as recommended in a dark container.

Carotene is one of the carotenoids that contribute to olive oil color. In addition, carotene contributes to oil antioxidant properties. For this reason, oils with high content of carotenoids are regarded as healthier and higher in nutritional value since they are precursors to vitamin A. All heat treatment samples showed a rise in carotene content, which was consistent with Yousfi *et al*. (2010) and Lauces *et al*. (2005). In spite of this increase, the oil always exhibited a green color because the green color was more intense than the yellow one. The increase was 2.5-fold higher in both varieties versus the control. This meant that preheat treatment of olives magnified its olive oil value.

Resistance to oxidation changes olive oil shelf-life. Shelf-life is extended or shortened depending the natural antioxidant agent's content. The prime constituents were found to be tocopherols and phenolic compounds (Table 3). These two compounds decreased as time and temperature increased. Additionally, the volatile constituents were also decreased, as shown in the results of organoleptic evaluations (Table 1).

Results in Table 4 indicated a minor decrease of oxidative stability as the heat treatment increased. The induction time in response to forced oxidation was 48h for NB. The oil remained unchanged significantly up to 60°C/4min and the lowest level was 40.1 h at 65°C/4 min. In NM these values were 30h in control, the resistance to oxidation continued up to 55°C/4 min and the lowest level was 24.3 h at 65°C/4 min. The differences of oxidative stability between the two cultivars were due to the differing amounts of anti oxidative agents in each. Nevertheless, the decreased oxidative stability in both cultivars was slight. This might be due to the increased chlorophylls and carotenoids at the advanced heat treatments. Furthermore, our results were in agreement with those obtained by Cruz *et al*. (2007) and Yousfi *et al*. (2010).

CONCLUSION AND RECOMMENDATIONS

The pre-heating for olive fruits increased the oil yield. Considering industrial practices this increase was significant, particularly for the earlier mature stage of olive fruits in which the oil possesses a high quality compared to oil quality properties at the later mature stage. Moreover, these data show that the optimum heat treatment was related to the olive cultivar. Hence, it is important to estimate the optimum heat treatment for the cultivar being processed.

The olive heating process can be carried out through adapting industrial olive oil production whereby a heating system could be added during the fruit-washing process.

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