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The effects of processing aids and techniques on olive oil extractability and oil quality indices

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SUMMARY: This study was conducted to investigate the effects of processing aids and techniques such as talcum powder (2% w/w), calcium carbonate (2% w/w), warm water dipping (45 °C), combined treatment (warm water dipping+2% calcium carbonate) and control (without adding processing aid) on extractability and quality of 'Tarom 7' olive oil as randomized complete block design with three replicates. The results showed that there were no significant differences in the carotenoid content, K_{232}, fatty acid profile or the Cox's value in the oil obtained from untreated and treated fruits with processing aids. The highest chlorophyll content (0.84 mg/kg), total phenolic content (236.94 mg/kg), paste extractability (8.5%) and the lowest peroxide values (0.32 meqO_2/kg), K_{270} (0.38) were obtained from the oil extracted with 2% talc powder. According to the results, it can be suggested that the 2% talc powder treatment could have a positive effect on olive oil quality and paste extractability.

KEYWORDS: *Oil yield; Olive oil; Processing aids; Profile fatty acids; Qualitative characteristics.*

RESUMEN: ***Efectos de los coadyuvantes tecnológicos y técnicas sobre la extractabilidad e índices de calidad del aceite de oliva.*** Este estudio se llevó a cabo para investigar los efectos de los coadyuvantes del procesamiento y técnicas, como talco (2 % p/p), carbonato de calcio (2 % p/p), inmersión en agua tibia (45 °C), tratamiento combinado (inmersión en agua tibia + carbonato de calcio al 2%) y control (sin adición de coadyuvante) sobre la extractabilidad y calidad del aceite de oliva 'Tarom 7' en un diseño de bloques completos al azar con tres repeticiones. Los resultados mostraron que no hubo diferencias significativas en el contenido de carotenoides, K_{232}, perfil de ácidos grasos y el valor de Cox del aceite obtenido de frutos no tratados y tratados con coadyuvantes de procesamiento. El mayor contenido de clorofila (0,84 mg/kg), contenido de fenoles totales (236,94 mg/kg), extractabilidad de la pasta (8,5%) y los valores más bajos de peróxidos (0,32 meqO_2/kg) y K_{270} (0,38) se obtuvieron para el aceite extraído con 2 % de talco. De acuerdo con los resultados, se puede sugerir que el tratamiento con talco al 2% podría tener un efecto positivo sobre la calidad del aceite de oliva y la extractabilidad de la pasta.

PALABRAS CLAVE: *Aceite de oliva; Características cualitativas; Coadyuvantes de elaboración; Perfil de ácidos grasos; Rendimiento de aceite.*

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1. INTRODUCTION

Olive trees, *Olea europaea*, are one of the most important commercial fruit crops in the world which originated from the eastern Mediterranean region (Khaleghi *et al*., 2015). The olive oil is the main product of olive trees and plays a key role in human health, due to its high contents in antioxidants and monounsaturated fatty acids (Altieri *et al*., 2015).

Reviews have shown that only 70-80 % of the oil located in the vacuoles of pulp cells can be extracted during the oil extraction process (Aguilera *et al*., 2010; Caponio *et al*., 2016). The rest of the oil (20-30 %) remains in the form of microgels and emulsion within the vegetable water (Espínola *et al*., 2009; Caponio *et al*., 2014; Al-Okaby *et al*., 2015).

It has been reported that most olive oil-producing countries (such as Spain and Italy) have utilized processing aids (co-adjuvant agents and techniques) to improve oil extraction efficiency (up to 10 to 30%) and to reduce the loss in oil in pomace (García *et al*., 2005; Cruz *et al*., 2007; Canamasas and Ravetti, 2011). Although talc and micronized talc powder (Fernández-Valdivia *et al*., 2008), specific enzymes (Sharma and Sharma, 2007; Najafian *et al*., 2009), common salt (NaCl, KCl), calcium carbonate (Espínola *et al*., 2009) and hot water dipping (García *et al*., 2005; Al-Rousan, 2017), as processing aids and techniques, have been commonly applied in the olive oil industry, nowadays, in Europe, only the use of hydrate magnesium silicate (natural talc) and kaolinitic clays are permitted (Vidal *et al*., 2018). Generally, these aids have improved oil extractability from the olive paste by breaking down the oil-water emulsion (Sadkaoui *et al.,* 2016). Researchers have stated that the effects of processing aids on oil extractability and quality could be different among olive cultivars (Peres *et al*., 2014; Al-Rousan, 2017). So, Espínola *et al*. (2009) reported that the oil extracted from 'Picual', 'Hojiblanca' and 'Arbequina' cultivars treated with 2% calcium carbonate was 1.08, 1.19 and 1.11 times greater than those of untreated extraction, respectively. Several studies have indicated that the application of processing aids enhanced oil extractability by 80.6 to 92.8%, leading to an increase in oil extraction yield from 18.9 to 22.1% (Cruz *et al*., 2007; Carrapiso *et al*., 2013; Caponio *et al*., 2014).

However, no report indicating the negative impacts of processing aids on organoleptic and chemical characteristics of oil upon adding the processing aids has been published (Carrapiso *et al*., 2013; Espínola *et al*., 2015). The sensory analysis results published by Vidal *et al*. (2018), indicated that the bitter, fruity and spicy attributes of olive oil were not significantly altered by using talc. Also, Espínola *et al*. (2009) stated the flavor or taste of olive oil was not changed by adding calcium carbonate. García *et al.* (2005) showed that panel tests of olive oil obtained by heating fruit was no different from that of untreated but the studies demonstrated that the concentration and type of processing aids imposed significant impacts on oil quality. So, Fernández-Valdivia *et al.* (2008) showed that the chlorophyll and carotenoid contents in olive oil did not change upon adding talc powder at either 1 or 2%. Also, the results reported by Cruz *et al.* (2007) and Moya *et al.* (2010) revealed that processing aids would not alter the chlorophyll content in the olive oil. At the same time, Al-Rousan (2017) found that the chlorophyll pigments were higher in the oil obtained from pre-heated fruits than those of untreated. In addition, Moya *et al.* (2010) reported that the amount of extinction coefficient (K_{270}) decreased by adding talc powder. The results published by Caponio *et al.* (2014) showed that adding talc powder at 1 and 2% during the malaxation stage to the 'Coratina' olive cultivar lowered the peroxide value of the extracted oil. García *et al*. (2005) reported that total phenol content was lower in the oil extracted with heated fruit compared to non-heated fruit.

One of the major problems in olive production areas in the Southwest of Iran is the high temperature during the accumulation of oil in the fruit, which causes the amount of oil produced, in particular, 'Tarom 7' cultivar of olive, to be reduced. Considering that there is no study on the effect of the processing aids on increasing oil extractability and improving the chemical properties of oil of 'Tarom 7' olive cultivar, in Iran, the present study aimed to investigate the effects of processing aids on the oil extractability and quality of olive cv. Tarom 7.

2. MATERIALS AND METHODS

2.1. Site of experiment and sampling method

This study was conducted on 15-year-old olive trees of cv. 'Tarom 7' grown with 5×6 m between and within rows in the olive orchard collection at

Shahid Chamran University of Ahvaz, located in the western area of Karun River in Ahvaz city, Iran (31°20´ N, 48°41´ E, 22 m above sea level).

From each tree, 3 kg of healthy fruits were harvested by handpicking in early November, 2016. Fruits of 15 trees were picked according to the maturity index of 4.2 (Espínola *et al*., 2009). Harvested fruits were immediately transferred to the physiology laboratory of horticulture and divided into 5 groups of 9 kg (with three replicates each including 3 kg).

2.2. Oil extraction

30 kg of healthy olive fruits were harvested and divided into 5 groups of 6 kg (three repetitions of 2 kg each). Each group was considered as a treatment. For the extraction of olive oil, an Abencor system (Commercial Abengoa S.A., Sevilla) was used. First, the olive fruits were crushed with a hammer mill, and the paste was malaxed for 30 minutes in the thermoheater at 25 ºC. Then the paste was centrifuged at 5000 rpm for 30 minutes. Finally, the oil samples were separated and stored at 4 ºC in the dark.

2.3. Processing aids and techniques

2.3.1. Solid aids

Talc powder (2% w/w) and calcium carbonate (2% w/w) were added to the paste at the beginning of the malaxation stage. For each replicate, 1.96 kg paste was taken from olive fruits crushed with a hammer mill then 40 g talc powder or calcium carbonate were added at the beginning of the malaxation stage.

2.3.2. Warm water dipping

Healthy olive fruits were immersed in a thermostatic water bath at 45 ° C for 5 min prior to the beginning of the oil extraction process.

2.3.3. Warm water dipping and solid aid

First, fruits were treated with warm water dipping (45 ° C for 5 min) before oil extraction, and then, 1.96 kg paste was taken from olive fruits crushed for each replicate and 40 g calcium carbonate were added to the paste at the beginning of malaxation. Olive oil extracted without any treatments was considered as control.

2.4. Determination of oil quality parameters

2.4.1. Free acidity

Free acidity (% oleic acid per 100 g oil) was determined as described by the European Community Reg. 2568/91 (EEC, 1991). 50 mL of ethanol: chloroform (50:50) were added to 10 g of oil. Next, the solution was titrated with 0.1 N KOH in the presence of phenolphthalein as indicator. Finally, the acidity was calculated according to equation (1)

$$\%\ \text{Free Fatty Acid} = \frac{(\text{mL of titrant})(\text{Nof titrant})(\text{Mwt. of fatty acid})}{(\text{sample wt.})(10)} \quad (1)$$

Where N is normality; Mwt is the molecular weight of oleic acid (282); and M is molarity.

2.4.2. Chlorophyll and carotenoid contents

The chlorophyll and carotenoid contents in the oil were determined using a spectrophotometer (UNICO UV-2100, manufactured in USA) as described by Mínguez-Mosquera *et al*. (1991). For this purpose, 1 g of the olive oil was dissolved in 10 mL of isooctane solution and the absorption spectrum of the solution was captured at wavelengths of 670 nm and 470 nm for the chlorophyll and carotenoid, respectively. Subsequently, the chlorophyll and carotenoid contents of the oil were evaluated in mg/kg of oil using Equations (2) and (3), respectively.

$$\text{Chlorophyll (mg/kg)} = (A670\times 10^6)/(613 \times 100 \times d) \quad (2)$$

$$\text{Carotenoid (mg/kg)} = (A470\times 10^6)/(2000 \times 100 \times d) \quad (3)$$

Where A is the absorption number and d is cell thickness.

2.4.3. UV extinction coefficient

To evaluate the specific extinction coefficient (K_{232} and K_{270}), 250 mg of oil were diluted with 25 mL of cyclohexane (of spectrophotometry grade) and then homogenized with a vortex for 30 seconds. The absorption of the solution was then determined at wavelengths of 232 nm and 270 nm

by a spectrophotometer (UNICO UV-2100, manufactured in the USA) according to the European Commission Regulation EEC/2565/91 (EEC, 1991).

2.4.4. Peroxide value

Peroxide value (milliequivalents of active oxygen per kilogram of oil) was determined according to AOCS (1998) method cd8-53.

Briefly, 30 mL of acetic acid–chloroform solution (3:2 v/v) were added to 5 g of oil. Then, 0.5 mL of saturated potassium iodide solution (KI) was added to the solution and the mixture was left for 1 minute. Subsequently, 30 mL of distilled water were added immediately.

The solution was titrated with 0.1 N sodium thiosulfate until the yellow iodine color almost disappeared. Next, a few drops of starch were added to the solution before being titrated with a 0.02 N thiosulfate solution. Finally, the corresponding peroxide value was obtained from Equation (4).

$$\text{Peroxide value (meq}O_2\text{/kg)} = \frac{(S-B)\times N\times 1000}{\text{mass of sample, g}} \quad (4)$$

Where B is the volume of titrant (mL of blank), S is the volume of titrant, mL of sample, N is the normality of the sodium thiosulfate solution.

2.4.5. Total phenolic content

The total phenolic content in the oil was obtained according to Montedoro *et al*. (1992) using the Folin–Ciocalteu reagent. Total phenolic content was calculated in mg of gallic acid per kg of olive oil. In this method, 2 g of olive oil and 2-3 drops of TWEEN® 20 were added to 10 mL of a methanol: water solution (80:20). The solution was centrifuged at 5000 rpm for 10 minutes, the supernatant was collected and the residual solution was once more added with 10 mL of the methanol: water solution and re-centrifuged under the same conditions before collecting the supernatant. In the next step, 1 mL of the obtained solution was combined with 1 mL of the methanol: water solution and 5 mL of double-distilled water. Then, 0.5 mL of the Folin–Ciocalteu reagent and 2 mL of 15% sodium carbonate were added to the solution. Finally, 1.5 mL of double-distilled water were added to the obtained solution and the mixture was subjected to vortex for 30 seconds. The resultant mixture was left in the dark for 2 hours and then the absorbance of the solution was read at 765 nm by a spectrophotometer (UNICO UV-2100, manufactured in the USA), and total phenol content was obtained from the following equation.

$$\text{Total phenol content}\left(\frac{\text{mg of gallic acid}}{\text{kg of oil}}\right) = \frac{\text{Galic acid (mg/ml)} \times \text{V (ml)} \times 1000}{\text{W (g)}} \quad (5)$$

Where V is the volume of the solution, W is the weight of the oil sample.

2.4.6. Fatty acid profile

The Fatty acid composition was determined according to European Official Methods of Analysis (EEC, 1991). 100 mg oil sample were dissolved in 10 mL n-hexane with 100 µL 2 N methanolic potassium hydroxide solution. Then the sample was vigorously shaken for 30 seconds and centrifuged for 15 minutes. The supernatant phase was used for chromatographic analysis. Chromatographic analyses were performed on Young Lin ACME 6000 (manufactured in South Korea) equipped with a flame ionization detector (FID), a split/splitless injector, and a BPX-70 capillary column (100 m ×0. 25 mm ID×0.2 µm film thicknesses, SGE, Australia). Helium was used as carrier gas. The temperatures of the injector, oven, and detector were set at 230, 200, and 280 °C, respectively.

2.4.7. Cox value

The cox value or oxidation index was obtained based on the content in 18-carbon fatty acids, according to the following equation:

$$\text{Cox value} = \frac{[(\%C18{:}1)1 + (\%C18{:}2)10.3 + (\%C18{:}3)21.6]}{100} \quad (6)$$

Where C18:1, C18:2, and C18:3 are the oleic acid, linoleic acid, and linolenic acid contents, respectively (Fatemi and Hammond, 1980).

2.5. Statistical analysis

These experiments were conducted with a completely randomized design in three replicates. The data were subjected to analysis of variance (ANOVA) using SAS Ver. 9.1 Software. Mean comparison of data was performed using Duncan's multiple range test at 5% significance level.

3. RESULTS

3.1. Chlorophyll and carotenoid contents

As shown in Table 1, a significant difference was observed in chlorophyll content among all treatments. The oil extracted with 2% talc powder (0.84 mg/kg) and 2% calcium carbonate (0.83 mg/kg) exhibited the highest chlorophyll content, while the lowest chlorophyll content (0.4 mg/kg) was related to the combined treatment (warm water dipping+2% carbonate calcium). In addition, chlorophyll contents in the oil extracted with warm water dipping, 2% calcium carbonate, and 2% talc powder were 0.7, 0.83, and 0.84 mg/kg, respectively, which were 1.22, 1.45, and 1.47 times greater than that of the control treatment (0.57 mg/kg). This result showed that there was no significant difference in chlorophyll content between the extracted control oil and combined treatments. Although the carotenoid value ranged from 0.22 to 0.32 mg/kg, the amount of carotenoid was not different among treatments. The processing aids and techniques used did not have significant effect on the carotenoid content (Table 1).

3.2. K_{232} and K_{270} extinction coefficients

No statistically significant difference in the K_{232} extinction coefficient was found between the oil extracted from untreated (control) and treated fruits with processing aids and techniques (Table 1).

Furthermore, the results showed that the processing aids and techniques had a statistically significant effect on the K_{270} extinction coefficient. The lowest value for the K_{270} extinction coefficient (0.33) was observed in the oil extracted with warm water dipping, which did not show a significant statistical difference between the oils extracted with either 2% talc powder (0.38) or the control treatment (0.37). The highest value for the K_{270} extinction coefficient

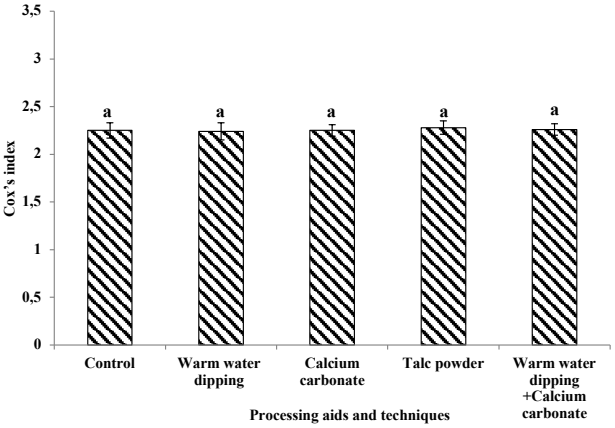

Figure 1. Effect of processing aids and techniques on Cox index. Values (Mean±Standard Deviation) with different letters are significantly different ($p < 0.05$) using Duncan's multiple range test. Number of replicates = 3

Table 1. Effect of different processing aids and techniques on `Tarom 7' olive oil quality indices

Oil quality indices	Processing aids treatments				
	Control	Warm water dipping (45 °C)	Calcium carbonate (2 % w/w)	Talc powder (2% w/w)	Warm water dipping (45 °C)+ Calcium carbonate (2% w/w)
Chlorophyll (mg/kg)	0.41±0.19^{c}	0.70±0.09^{b}	0.83±0.09^{a}	0.84±0.11^{a}	0.57±0.06^{c}
Carotenoid (mg/kg)	0.25±0.07^{a}	0.22±0.12^{a}	0. 31±0.10^{a}	0.32±0.11^{a}	0.32±0.18^{a}
Free acidity (% oleic acid)	0.35±0.01^{a}	0.32±0.01^{b}	0.26±0.01^{c}	0.31±0.01^{b}	0.25±0.01^{c}
K_{270}	0.37±0.06^{b}	0.33±0.07^{b}	0.44±0.02^{a}	0.38±0.05^{b}	0.45±0.03^{a}
K_{232}	2.79±0.06^{a}	2.79±0.07^{a}	2.74±0.05^{a}	2.73±0.08^{a}	2.79±0.08^{a}
Peroxide value (meq O_2/kg oil)	0.40±0.01^{a}	0.37±0.01^{b}	0.29±0.01^{d}	0.32±0.01^{c}	0.32±0.01^{c}
Total phenol content (mg/kg)	174.44±2.03^{d}	181.38±12.51^{c}	222.77±3.08^{b}	236.94±4.21^{a}	193.86±12.38^{c}

Values (Mean± Standard Deviation) in the same row with different superscripts are significantly different ($p < 0.05$) using Duncan's multiple range test.
Number of replicates = 3

(0.45) was related to the combined treatment (warm water dipping+2% carbonate calcium), which indicated no significant difference with the 2% calcium carbonate treatment (0.41) (Table 1).

Moreover, the value for the K_{270} extinction coefficient in the oils treated with 2% calcium carbonate was approximately 18.91% higher than that of the control treatment. The increased rate of the K_{270} extinction coefficient in fruits treated with warm water dipping+2% carbonate calcium was 21.62% compared to the untreated fruits (Table 1).

3.3. Free acidity

A significant difference was observed in free acidity between treated fruits treated with processing aids and untreated fruits. The highest and lowest values for free acidity were obtained for the control (0.35%) and combined treatment (warm water dipping+2% carbonate calcium) (0.25%), respectively (Table 1).

Moreover, the results showed that free acidity was the same in warm water dipping treated and untreated fruits. Also, there was no significant difference in the free acidity in the oil obtained from talc powder and combined treatments (Table 1).

Based on the results, the acidities of the extracted oils with warm water dipping, 2% calcium carbonate, combined treatment (warm water dipping+2% carbonate calcium), and 2% talc powder were 8.57, 25.71, 25.71, and 11.42% higher than that of the extracted oil under control treatment, respectively (Table 1).

3.4. Total phenol content

The data showed that the processing aid treatments had a significant effect on total phenol content. The highest total phenol content (236.94 mg/kg of oil) was related to the oil extracted with 2% talc treatment; while the oil extracted with the combined treatment (warm water dipping+2% carbonate calcium) showed the lowest total phenol content (174.44 mg/kg of oil). Compared to the control treatment, the 2% talc and 2% calcium carbonate treatments exhibited 22.24 and 14.93% higher total phenolic contents, respectively; while the oil extracted with warm water dipping or under the combined treatment (warm water dipping + 2% carbonate calcium) showed lower values for total phenolic content by 6.42 and 10%, respectively.

3.5. Peroxide value

As shown in Table 1, the results indicated that the application of the processing aids tended to attenuate the peroxide value of the extracted oil, as compared to the control treatment. Accordingly, the highest peroxide value (0.40 meqO_2/kg) was that of the oils obtained from untreated fruits; while this index was minimal (0.29 meqO_2/kg) for the oil extracted with 2% calcium carbonate. The peroxide values for the oils obtained with warm water dipping, 2% calcium carbonate, combined treatment (warm water dipping + 2% carbonate calcium), and 2% talc powder were found to be 7.5, 27.5, 20, and 12.5% lower than that of the extracted oil under control treatment, respectively.

3.6. Profile of fatty acids and Cox value

The results indicated that the oils extracted from fruits which were treated and untreated with processing aids showed the same values for palmitoleic acid (C16:1), stearic acid (C18:0), oleic acid (C18:1), linoleic acid (C18:2), linolenic acid (C18:3), oleic acid/linoleic acid ratio (C18:1/C18:2), monounsaturated fatty acids-to-polyunsaturated fatty acids ratio (MUFA/PUFA), unsaturated fatty acids-to-saturated fatty acids ratio (UFA/SFA), and the Cox value; whereas the value for palmitic acid (C16:0) was different between the oils extracted with processing aids and that of the control treatment (Table 2; Figure 2).

The highest and lowest contents in palmitic acid (16.30 and 16.03%, respectively) were related to 2% calcium carbonate and warm water dipping treatments, respectively.

3.7. Paste extractability (%)

The impact of the use of processing aids and technique treatments on paste extractability is shown in Figure 2. A significant difference in paste extractability was found between the control treatment and the treatment with processing aids. The use of processing aids and technique treatments improved paste extractability. The lowest (4.07%) and the highest (9.4%) improvement in paste extractability were related to control and 2% talc powder treatments, respectively. In addition, no significant difference in paste extractability was observed between fruits treated with calcium carbonate (2%) and

TABLE 2. Effect of different processing aids and techniques on the fatty acid composition of `Tarom 7' olive oil.

Fatty acid composition (%)	Processing aids treatments				
	Control	Warm water dipping (45 °C)	Calcium carbonate (2% w/w)	Talc powder (2% w/w)	Warm water dipping (45 °C)+ Calcium carbonate (2% w/w)
Palmitic acid (C16:0)	16.26±0.12[a]	16.03±0.11[b]	16.30±0.19[a]	16.08±0.15[b]	16.09±0.08[b]
Palmitoleic acid C16:1)	1.67±0.31[a]	1.58±0.23[a]	1.59±0.21[a]	1.58±0.19[a]	1.59±0.23[a]
Stearic acid (C18:0)	2.2±0.35[a]	2.22±0.20[a]	2.18±0.18[a]	2.15±0.15[a]	2.19±0.31[a]
Oleic acid (C18:1)	54.3±0.71[a]	54.07±1.37[a]	53.59±0.98[a]	54.53±1.01[a]	53.95±0.96[a]
Linoleic acid (C18:2)	12.95±0.61[a]	12.87±0.40[a]	13.15±0.71[a]	13.26±0.81[a]	13.16±0.92[a]
Linolenic acid (C18:3)	1.72±0.31[a]	1.73±0.22[a]	1.68±0.09[a]	1.71±0.12[a]	1.70±0.26[a]
SFA[c]	18.46±0.35[a]	18.25±0.42[a]	18.48±0.32[a]	18.23±0.52[a]	18.28±0.62[a]
MUFA[d]	54.97±1.31[a]	55.65±1.02[a]	55.18±1.32[a]	56.11±1.41[a]	55.54±0.01[a]
PUFA[e]	14.67±0.91[a]	14.60±0.52[a]	15.18±0.62[a]	14.97±0.71[a]	14.86±0.01[a]
MUFA/PUFA[f]	3.81±0.40[a]	3.82±0.22[a]	3.72±0.33[a]	3.74±0.62[a]	3.73±0.01[a]
UFA/SFA	3.82±0.62[a]	3.84±0.42[a]	3.78±0.34[a]	3.91±0.22[a]	3.85±0.01[a]
Oleic/Linoleic	4.19±0.31[a]	4.21±0.28[a]	4.07±0.21[a]	4.11±0.31[a]	4.11±0.01[a]

[a-b] Values (Mean± Standard Deviation) in the same row with different superscripts are significantly different ($p < 0.05$) using Duncan's multiple range test.
[c]: Saturated fatty acids. [d]: Monounsaturated fatty acids. [e]: Polyunsaturated fatty acids. [f]: Monounsaturated fatty acids / Polyunsaturated fatty acids. Number of replicates = 3

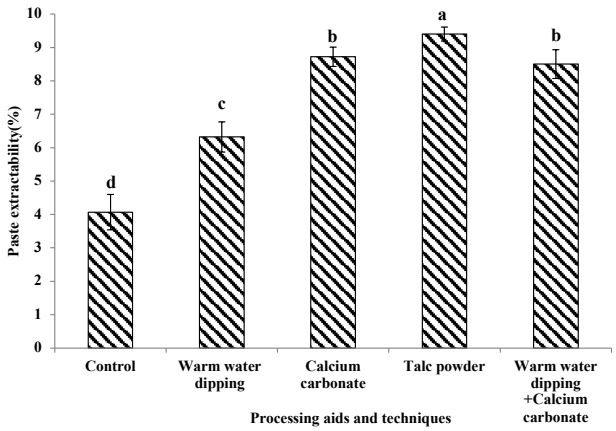

FIGURE 2. Effect of processing aids and techniques on paste extractability (%)
Values (Mean±Standard Deviation) with different letters are significantly different ($p < 0.05$) using Duncan's multiple range test. Number of replicates = 3

combined treatment. Paste extractability was lower in warm water dipping (6.32%) compare to calcium carbonate (2%) and combined treatment.

4. DISCUSSION

4.1. Chlorophyll and carotenoid content

Chlorophyll and carotenoid are the main pigments in olive oil. These pigments play an important role in oxidative activity (Khaleghi *et al*., 2015). Our findings showed that chlorophyll content was affected by the type of processing aids. The amount of chlorophyll was higher in oil extracted from fruits treated with processing aid in comparison to those of untreated fruits. The highest chlorophyll content (0.84 mg/kg) was obtained from oil extracted with 2% talc powder; while the lowest chlorophyll content (0.57 mg/kg) was observed in the control treatment. Results showed there was no significant difference in carotenoid content between control treatment and processing aid treatments. These findings were in agreement with the results reported by several authors who indicated that chlorophyll pigment contents were significantly increased by the addition of processing aids such as pre-heated treatment, or talc powder (García *et al*. 2005; Criado *et al*., 2007; Caponio *et al*., 2014; Al-Rousan, 2017). Reviews showed that a warm water dipping treatment could inactivate enzymes, especially lipoxygenase, which was important to pigment degradation (García *et al*., 2005). Cruz *et al*. (2006) stated that salt aids could increase the solubility of pigments in oil. Our results are not in agreement with the results of Moya *et al*. (2010) for picual olive, Cruz *et al*. (2007), Espínola *et al*. (2015), or Peres *et al*. (2014), who believed that the processing aids imposed no significant effect

on not only chlorophyll pigment and carotenoid contents but also on the xanthophyll content. It seems that this inconsistency could be attributed to genetic and physiological differences between the studied cultivars or differences in the type of processing aids.

4.2. K_{232} and K_{270} extinction coefficients

According to published reports, the extinction coefficient provides a measurement of secondary oxidation processes in the oil that lead to the formation of conjugated dienes (K_{232}), aldehydes, and ketones (K_{270}) (Moya *et al*., 2010; Carrapiso *et al*., 2013; Caponio *et al*., 2014). Our findings revealed no significant difference among the considered treatments in terms of the K_{232} index, but rather significant differences in K_{270}, so that the combined treatment (warm water dipping + 2% calcium carbonate) and also the 2% calcium carbonate treatment could further inhibit the formation of aldehydes and ketones, as compared to other treatments. These results were in agreement with the reports submitted by Moya *et al*. (2010). Also, Caponio *et al*. (2014) and Carrapiso *et al.* (2013) showed that talc powder increased the value of K_{270}, whereas K_{232} was not affected by the addition of talc. These published results are similar to the present study.

4.3. Free acidity

Previous studies have shown that oil acidity is a result of the formation of free fatty acids upon the activity of a particular type of enzyme to decompose triglycerides (Cruz *et al*., 2007; Moya *et al*., 2010). Our findings showed that processing aids tend to affect the activity of the enzyme involved with the process of triglyceride decomposition so that the combined treatment (warm water dipping + 2% calcium carbonate) could further retard the activity of this enzyme.

However, these findings were not in agreement with the results of García *et al*. (2005), Fernández-Valdivia *et al*. (2008), Moya *et al*. (2010), Carrapiso *et al*. (2013), and Caponio *et al*. (2014), who concluded that processing aids such as hot water dipping, talc, and micro talc did not affect free fatty acids. Canamasas and Ravetti, (2014) found that oil extracted with Talc (2.0%) and microtalc had the lower acidity (free fatty acid) than the control. These results are in agreement with the present study. Farag *et al*. (1997) demonstrated that some processing aids such as pre-heated treatment (microwave heating) could reduce the amount of free fatty acids by reducing lipase activity.

4.4. Total phenol content

The results of the present experiment showed that the processing aids affected the release of phenolic compounds and their transmission into the oil phase. Contrary to the pigments which develop in particular parts of the fruit, phenolic compounds are found in most parts of fruit in various forms, i.e. water-soluble or fat-soluble. The processing aids significantly affected the solubility and release of these compounds. Among other treatments, the 2% talc powder imposed the largest impacts on the solubility and release of these compounds.

Although the results of this study were inconsistent with previous reviews about exposing olive fruits of ‘carrascina’, ‘Galega’, ‘Cobrançosa’ and ‘Vulgar’cultivras to warm water and talc (Carrapiso *et al*., 2013; Peres *et al*., 2014), our findings were in agreement with those of Ben-David *et al*. (2010), Caponio *et al*. (2014) and Al-Rousan (2017) on ‘Barnea’, ‘Nabali Baladi’, ‘Nabali Muhassan’ olive cultivars who reported that the total phenolic content f the olive oil was enhanced in the presence of processing aids.

Servili *et al*. (2003) also indicated that pre-heated treatment reduced total phenol compounds in the olive oil by increasing polyphenol oxidase and peroxidase activities.

4.5. Peroxide value

According to existing reports, peroxide value represents the primary oxidation of the oil and serves as an important factor in determining the quality of olive oil, indicating whether the oil is healthy or rather spoiled. When olive oil or olive fruit is exposed to free air where it comes in contact with oxygen in the presence of adverse temperature conditions, primary oxidation of the oil or fruit begins; this process then contributes to increased peroxide content in the oil and the formation of free radicals in the oil (Moya *et al*., 2010; Peres *et al*., 2014). The processing aids tend to inhibit this oxidation process and control the peroxide value of the oil by controlling the oxygenated free radicals. The results of this study showed that the 2%

calcium carbonate lowered the peroxide value of the extracted oil. This finding was in agreement with the findings of Caponio *et al*. (2014), who reported that the presence of processing aids tends to decrease the peroxide value of the extracted oil from the 'Coratina' olive cultivar, which is rather inconsistent with the reports published by Cruz *et al*. (2007).

The difference in the results could possibly be due to the type of cultivar, concentration, and type of processing aids.

4.6. Profile of fatty acids and Cox's value

The results of this study indicated that the profile of fatty acids and Cox value were not affected by processing aids. these findings were similar to the results of Ben Brahim *et al*. (2015), who stated there was no significant difference between the effects of the calcium carbonate (1.5%) treatment and the control treatment on the contents of palmitic acid, palmitoleic acid, oleic acid, linoleic acid, linolenic acid, saturated fatty acids (SFA), mono-unsaturated fatty acids (MUFA), polyunsaturated fatty acids, (PUFA), and unsaturated fatty acids (UFA) as well as the MUFA/PUFA ratio in the 'Chemlali' olive cultivar.

4.7. Paste extractability (%)

According to the results, adding processing aids increased paste extractability. While paste extraction rate was lower in untreated fruits with processing aids. In fact, the highest paste extractability was related to the 2% talc powder treatment. The results of this research were in agreement with the reports published by Carrapiso *et al*. (2013), Koprivnjak *et al*. (2016), Caponio *et al*. (2014), and Moya *et al*. (2014), who observed that physical processing aids tended to enhance the efficiency of the oil extraction process. Vidal *et al.* (2018) reported that an increase greater than 2% of talc proportion can have a negative effect on extraction efficiency. Canamasas and Ravetti, (2014) found that talc powder and calcium carbonate improved paste extractability from 8.2 to 10.2 and 10.7% for the 'Arbequina'olive cultivar, respectively. Cruz *et al*. (2007) indicated that pre-heating the fruit caused cell walls to soften, and release oil from the cells. Other researchers believed that talc powder and calcium carbonate, as solid aids, could help to break oil-water emulsions and improve paste extractability (Canamasas and Ravetti, 2014).

5. CONCLUSIONS

In this experiment, the processing aids and techniques used could facilitate the process of oil extraction and enhance the efficiency of the process compared to the control by contributing to the coalescence of fine and coarse oil droplets and altering the cell wall and membrane to release larger amounts of oil content from the cells and other fruit components. According to the results, the value of oil extracted with 2% talc powder was greater than the other treatments. Furthermore, the values for chlorophyll content and total phenol content in oils extracted with 2% talc powder were higher than the control and other treatments. The amount of acidity and peroxide value were significantly reduced in oils obtained by adding 2% talc powder compared to the control treatment. Therefore, the best processing aid used in this study was the 2% talc powder treatment, which was able to improve oil extraction yield and oil quality parameters.

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