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CHILEANJAR
CHILEAN JOURNAL of AGRICULTURAL RESEARCH

Dehydrated olive-waste cake as a source of high value-added bioproduct: Drying kinetics, physicochemical properties, and bioactive compounds

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Olive (*Olea europaea* L.) oil processing produces significant amount of waste that can be utilized for the production of high value-added ingredients for various industrial applications. In this work, the effects of temperature on drying kinetics and quality indexes of the olive-waste cake during convective dehydration (40-90 °C) were investigated. Results on effective moisture diffusivity, physicochemical parameters, fatty acid profile, total phenolic, flavonoid, and flavanol contents as well as antioxidant capacity are also reported. Most of the fatty acids increased their content with respect to control sample with a temperature increase, i.e. oleic and linoleic acids increased 48% and 43% at 70 and 40 °C, respectively. Total flavanol content increased with temperature (48-62 mg catechin equivalents [CTE] 100 g^{-1} DM) except for 80 °C. Total phenolic and total flavonoid contents were highly correlated to antioxidant capacity ($0.923 < r < 0.992$), except for 70 and 80 °C, the rest of the samples maintained their initial antioxidant capacity by ORAC analysis. Thus, these parameters show that dried olive-waste cake has a high bioactive compounds with potential use as additives for the food or other industries.

Key words: Antioxidant capacity, drying process, fatty acids, *Olea europea*, olive waste, phenolic compounds.

INTRODUCTION

Chile has recently increased the olive oil production due to appropriate climatic conditions in Valparaíso, Libertador General Bernardo O'Higgins, and Maule Regions (ODEPA, 2012). This industry produces relative big amounts of wastes generally known as olive by-products. The different byproducts considered are defined as: a) Olive cakes that consists of olive pulp, skin, stone, and water (Molina-Alcaide and Yáñez-Ruiz, 2008); b) olive mill wastewater is an acidic mixture of water, organic compounds (sugars, N compounds, volatile acids, fats, polyphenols, and fibers) and inorganic compounds (mainly potassium salts and phosphates) and a high biochemical oxygen demand (BOD) (Arjona et al., 2005); and c) wet olive cake made up of mixture of water effluents from the initial fruit cleansing and from secondary centrifugation. This solid residue (olive waste cake) has a high organic matter concentration giving an elevated polluting load and it cannot be easily handled by traditional technology which deals with the conventional three-phase olive cake (Borja et al., 2006). Treatment and disposal of this waste is one of the most serious environmental problems (Doymaz et al., 2004; Lafka et al., 2011); it is often used as a natural fertilizer, as substrate for fermentation and animal feeding, dying agent in biosorbing material and for bio-energy exploitation (Akar et al., 2008; Aboulkas et al., 2008). Due to its high water content, the cake has to be dried or concentrated prior to further processing (Vega-Gálvez et al., 2010).

Convective dehydration is a widely used technology for the production of stabilized materials from a quality point of view. Besides, this type of dehydration is usually applied for by-products from industrial processes (Freire et al., 2001; Doymaz et al., 2004; Ruiz Celma et al., 2008). To control the process itself, mathematical modeling of the process is required. Modeling allows the choice of suitable operating conditions for minimizing drying time and changes in the quality of products when exposed to high temperatures (Di Scala and Crapiste, 2008).

The olive-waste cake contains important amounts of valuable bioactive compounds that can be recovered for possible applications in food, pharmaceutical, and cosmetic industries (Moure et al., 2001; Ranalli et al., 2003). Polyphenols have acquired a great importance because of the multiple benefits generated in their consumers' health, providing antioxidant and anti-inflammatory properties (Mertens-Talcott et al., 2006). A broad range of phenolic compounds have been identified in virgin oil, including flavanols and flavonoids (Suárez

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Received: 30 September 2013.
Accepted: 14 April 2014.
doi:10.4067/S0718-58392014000300007

et al., 2010). Flavonoids are one of the major groups of phenolic compounds present in olive fruits and in their by-products (Vlahov, 1992). Although only about 2% of the total phenols found in olive fruits are transferred to the extracted olive oil, remaining 98% are retained in the olive cake (Suárez et al., 2010).

Nowadays, there is an increasing demand for additional supplementary products in pure forms and for commercial applications that exploit the broad-spectrum bioactivity of these compounds. Agricultural wastes represent a largely ignored source of high-value phytochemicals and value-added industrial products that could contribute to sustainability of objectives with considerable economic benefits (Wijngaard et al., 2009).

The aims of this study were to evaluate the effect of drying temperature on drying kinetics, effective moisture diffusivity, physicochemical properties, fatty acids profile, total phenolic content, total flavonoid and flavanol contents, and antioxidant capacity of the waste olive cake (*Olea europaea* L. 'Frantoio'). Furthermore, simulation of experimental drying curves and mathematical modeling are also presented.

MATERIALS AND METHODS

Raw material

The waste olive cakes were supplied by an agro-food company (Agronoble S.A.) from Ovalle, Chile. The waste resulted from a continuous low temperature process of the olive oil production (Arjona et al., 2005; Borja et al., 2006; Molina-Alcaide and Yáñez-Ruiz, 2008). The olive variety used in this investigation was 'Frantoio', harvested at optimum ripeness (oil content:dry matter) and pressed without delay. Samples used for analysis were packed in polyethylene bags and kept in a freezer at -20 ºC. Before drying, waste olive cake samples were thawed during 24 h under refrigeration conditions at 5 ºC.

Drying process and modeling of drying kinetics

Drying process was carried out at six different air temperatures (40, 50, 60, 70, 80, and 90 ºC) in a batch convective dryer, built at the Department of Food Engineering of Universidad de La Serena, at a constant air flow rate of 2.0 ± 0.2 m s^{-1} (Vega-Gálvez et al., 2010). The load density was 4 kg m^{-2}. The dehydrated samples (1 cm thickness) were packed in polyethylene bags and kept at room temperature in the dark until analysis. All experiments were done in triplicate. To predict drying characteristics, the Fick's second diffusion law (Equation [1]) was applied since it describes dehydration taking place during the falling rate period when water is transported to the surface material by diffusion phenomena (Di Scala and Crapiste, 2008). In this model the dependent variable is the moisture ratio (MR), which relates the gradient of sample moisture content in real time to both initial and equilibrium moisture content (Equation [2]):

$$MR = \frac{8}{\pi^2}\sum_{i=0}^{\infty}\frac{1}{(2i+1)^2}\exp\left[\frac{(2i+1)^2 D_{eff}\,\pi^2 t}{4L^2}\right] \quad [1]$$

$$MR = \frac{X_{wt} - X_{we}}{X_{wo} - X_{we}} \quad [2]$$

where MR is moisture ratio (dimensionless), L is thickness of the slab (m), X_{wt} is moisture content (g water g^{-1} DM), X_{wo} is initial water content (g water g^{-1} DM), X_{we} is equilibrium water content (g water g^{-1} DM), D_{eff} is effective moisture diffusivity (m^2 s^{-1}), t is drying time (s), and L is spatial dimension (m).

Desorption isotherm for the waste olive-cake were determined at 40 °C. This method is recommended by the European project COST 90 (Spiess and Wolf, 1983). Experimental data were fitted by Guggenheim, Anderson and de Boer (commonly known as GAB, Equation [3]) models (Park et al., 2002).

$$X_{we} = \frac{X_m \cdot C \cdot k \cdot a_w}{(1 - k \cdot a_w)\cdot(1 + (C-1)\cdot k \cdot a_w)} \quad [3]$$

where X_m is monolayer water content (g water g^{-1} DM), C and K are GAB's model parameter (dimensionless), and a_w is water activity (dimensionless).

In practice, the effective moisture diffusivity was calculated for each temperature by plotting experimental drying data in terms of ln(MR) *vs*. drying time and the D_{eff} value obtained from the straight line's slope. Drying curves were fitted to five thin layer drying models namely, Midilli-Kukuk (Equation [4]), Logarithmic (Equation [5]), Verma (Equation [6]), Weibull (Equation [7]), and Modified Page (Equation [8]) (Akgun and Doymaz, 2005; Meziane, 2010; Vega-Gálvez et al., 2010). The mathematical expressions for these models are the following:

$$MR = aexp(-kt^n) + bt \quad [4]$$

$$MR = a\,exp(-kt^n) + c \quad [5]$$

$$MR = a\,exp(-kt) + (1-a)exp(-ct) \quad [6]$$

$$MR = exp\left[-(t/\beta)^{\alpha}\right] \quad [7]$$

$$MR = exp\left(-(kt)^n\right) \quad [8]$$

where k is kinetic parameter (min^{-1}); a, b, c, and n are empirical parameters (dimensionless); α is Weibull's shape parameter (dimensionless); β is Weibull's scale parameter (min).

The temperature dependence of the effective diffusivity can be represented by an Arrhenius relationship (Akgun and Doymaz, 2005). Both kinetic parameters (E_a and D_o) can be estimated from the slope and intercept of the plot ln(D_{eff}) *vs*. T^{-1} (Vega-Gálvez et al., 2010):

$$D_{eff} = D_o\,exp\left(\frac{-E_a}{RT}\right) \quad [9]$$

where T is absolute temperature (°K), D_o is Arrhenius factor (m^2 s^{-1}), Ea is activation energy (kJ mol^{-1}), and R is universal gas constant (J mol^{-1} K^{-1}).

Statistical evaluation of the models

Fit quality of the proposed models for simulating the drying kinetics data was evaluated by means of statistical

test including determination correlation coefficient (r^2), Chi-square (χ^2) (Equation [10]), and root mean square error (RMSE) (Equation [11]). Values RMSE and χ^2 closer to zero, and r^2 value closer to 1, indicate a better fit to drying kinetics:

$$\chi^2 = \frac{\sum_{j=1}^{N}(MR_{ei} - MR_{ci})^2}{N - z} \quad [10]$$

$$RMSE = \left[\frac{1}{N}\sum_{j=1}^{N}(MR_{ci} - MR_{ei})^2\right]^{1/2} \quad [11]$$

where *ei* are experimental data, *ci* are calculated data, *j* is number of terms, *z* is number of parameters, and *N* is number of data.

Physicochemical analysis and determination of fatty acids

The crude protein content was determined using the Kjeldahl method with a conversion factor of 6.25. The lipid content was obtained gravimetrically following Soxhlet extraction, using petroleum ether as solvent according to method described in AOAC method nr 920.39 (AOAC, 1990). The crude fiber content was estimated through an acid/alkaline hydrolysis of insoluble residues as described in AOAC method nr 962.09. The crude ash content was estimated by incineration in a muffle furnace (FE-341, Felisa, Jalisco, Mexico) at 550 ºC. The available carbohydrate was estimated by difference. The water content was determined by means of AOAC method nr 934.06. The pH-value was measured directly on fresh sample as described in AOAC method nr 945.10 using a microcomputer (pH-vision 246072, Extech Instruments, Waltham, Massachusetts, USA) and the level of titrimetric acidity at final pH 8.2 was expressed as oleic acid as described in AOAC method nr 939.05. Water activity was measured in a water activity meter (Novasina AW Sprint, Pfäffikon, Switzerland). All methodologies were done in triplicate and followed the recommendations of the Official Methods of Analysis (AOAC, 1990).

The fatty acid composition was determined by gas chromatography. Fatty acid methyl esters (FAME) were prepared by UNE-EN ISO 5509, with 0.5 M NaOH used for saponification, isooctane as solvent and BF_3-CH_3OH as esterifying agent. The FAME were analyzed using Perkin Elmer gas chromatograph fitted with SGE capillary column, BPX70 bonded phase in fused silica 60 m length, 0.25 mm internal diameter and 0.25 mm film thickness with flame ionization detector (FID). The injection and detector temperatures were set at 250 ºC and the carrier gas was He at 1.0 mL min^{-1} constant flow. The initial oven temperature was 160 ºC and then increased to 190 ºC at a rate of 3 ºC min^{-1} and held for 10 min, again increased to 220 ºC at rate of 1 ºC min^{-1} for a final time of 10 min. The injection volume was 0.5 µL. Peaks were identified by comparison with their retention times with appropriate FAME standards (Sigma). The FAME standards were run under the same conditions and the subsequent retention times were used to identify fatty acids. The fatty acids were expressed as g 100 g^{-1} DM.

Preparation of extract and determination of total phenolic, flavonoid, and flavanols

The extraction was performed as described by Que et al. (2008), with some modifications. Analytically weighed 2 to 3 g dried and finely crushed sample, to which 40 mL of absolute methanol were added. Mixture was homogenized during 24 h in 50 mL Falcon tubes on orbital shaker (Orbital Shaker OS-20, Boeco, Hamburg, Germany) at ambient temperature and darkness. Then, it was filtered through a Whatman filter nr 1. The filtered cake was washed twice with 20 mL methanol and the filtrate was evaporated to dryness under reduced pressure at 40 °C on a rotary evaporator (Rotavapor R-210, Büchi, Flawil, Switzerland). Extract was then dissolved in 50 mL methanol in volumetric flask and kept refrigerated until further analysis.

The total phenol content of the olive waste cake extracts was determined using the spectrophotometric analysis with Folin-Ciocalteu's phenol reagent (Merck, KGaA, Darmstadt, Germany) described by Chen et al. (2008) with some modifications. In short, an aliquot of 100 μL methanolic solution of olive waste cake extract was added, diluted with 400 μL methanol in a 15 mL falcone tube, 0.5 mL Folin-Ciocalteau reagent was added after 5 min followed by a neutralization with 2 mL Na_2CO_3 solution 200 mg mL^{-1}. The sample was then mixed on a vortex (Velp 2X3, Milan, Italy) and allowed to stand for 15 min at ambient temperature. Ultra-pure water (10 mL) was then added and the precipitate formed was removed by centrifugation for 5 min at 5000 rpm. Finally, absorbance was measured at 725 nm in a spectrophotometer (Spectronic 20 Genesys, New York, USA) and compared to a gallic acid (UCB, Brussels, Belgium) calibration curve (25-500 μg gallic acid [GA] mL^{-1}). Results were expressed as mg GA 100 g^{-1} DM. All measurements were done in triplicate.

Total flavonoid content of olive waste cake extracts was performed following the protocol described by Kim et al. (2003) slightly modified. A 0.1 mL aliquot of methanolic extract was mixed with 2.4 mL deionized water in a 5 mL microcentrifuge tube, added 0.15 mL $NaNO_2$ (50 mg mL^{-1}), and allowed to react for 5 min. Following this, 0.15 mL $AlCl_3$ (100 mg mL^{-1}) was added and the mixture allowed standing for further 6 min. Finally, 1.0 mL 1 M NaOH and 1.2 mL deionized water were added to the reaction mixture and the absorbance at 510 nm was obtained against a blank, by replacing the extract with deionized water. Total flavonoid content was calculated from a calibration curve using catechin as standard, and expressed as mg catechin equivalents (CTE) 100 g^{-1} DM. All measurements were done in triplicate.

Flavanols were determined after derivatization with p-dimethylaminocinnamaldehyde (p-DMACA reagent,

Fluka, Sigma-Aldrich, St. Gallen, Switzerland) using an optimized methodology (Nigel and Glories, 1991). An aliquot of methanolic extract (0.2 mL) was introduced into a 2 mL microcentrifuge tube, 0.5 mL HCl (0.24 N in methanol) and 0.5 mL p-DMACA solution (0.2% in methanol) were added. The mixture was allowed to react for 5 min at room temperature, and the absorbance was measured at 640 nm. A blank was prepared by replacing sample with methanol. Total flavanols were calculated from a calibration curve using catechin as standard, and expressed as mg CTE 100 g^{-1} DM. All measurements were done in triplicate.

Radical scavenging activity and oxygen radical absorbance capacity (ORAC) assay

Free radical scavenging activity of samples was determined using the 2,2-diphenyl-2-picrylhydrazyl (DPPH) method (Lafka et al., 2011) with some modifications. Different dilutions of methanolic extracts were prepared in triplicate. An aliquot of 3.9 mL of 0.51 mM DPPH (Sigma-Aldrich) radical in methanol was added to a test tube with 20 µL sample extract and 80 µL methanol. The reaction mixture was vortex-mixed for 30 s and left to stand at room temperature in the dark for 30 min. The absorbance was measured at 515 nm. 6-Hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox, Sigma-Aldrich) was used as the standard for the calibration curve (0.1 ~ 1.0 mM) and the DPPH radical-scavenging activities were expressed as µmol Trolox equivalents (TE) mg^{-1} DM.

Antioxidant activity was determined using an ORAC procedure described by Guorong et al. (2009). Extract was obtained using 2 to 3 g dried and crushed sample, to which 20 mL of mixture acetone water (50:50 v/v) were added. Mixture was homogenized during 1 h on an orbital shaker to 200 rpm. Extracts were centrifuged (Centrifuge 5804R, Eppendorf, Hamburg, Germany) for 15 min at 4000 rpm. The supernatant was filter through a Whatman filter nr 1. A second extraction was taken and the filtrate was evaporated under reduced pressure at 40 °C on a rotary evaporator (Büchi R-210). Extract was then dissolved in 50 mL buffer phosphate pH 7.4 in volumetric flask and kept refrigerated until further analysis. Trolox standard solutions were prepared from 6.25-200 μM for the construction of calibration curve; 40 μL trolox standard solution, 40 μL buffer phosphate pH 7.4 and blanks containing only 40 μL extraction solvent were added to a 96-welled microplate. A solution of 100 nM fluorescein (200 μL) was added to each well and plates were covered and incubated for 20 min in a Multimode Plate Reader (Victor X3, Perkin Elmer, Hamburg, Germany) preheated to 37 °C. A solution of 0.36 M AAPH (35 μL) was added to each well to generate peroxyl radicals. Fluorescence readings of the plate were taken every minute using an excitation wavelength of 485 nm and an emission wavelength of 535 nm until all fluorescence readings declined to less than 5% of initial values. Samples and standards were determined in triplicate.

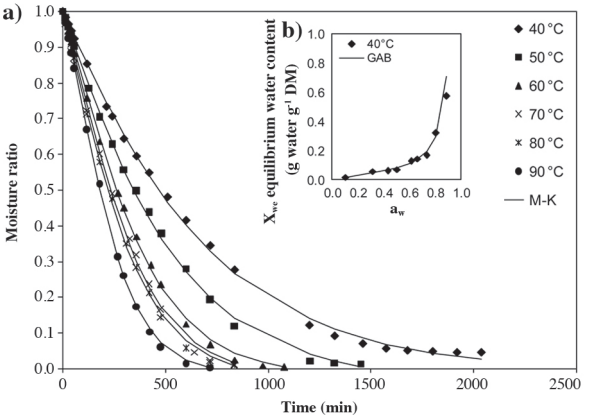
Statistical analysis

The experimental design only evaluated the effect of air temperature on drying kinetics and quality parameters. One-way ANOVA with one factor (k = temperature) and six levels (n = 40, 50, 60, 70, 80, and 90 °C) using Statgraphics Plus5.0 (Statistical Graphics Corp., Herndon, Virginia, USA) was performed (n^k). Differences among media were analyzed using the least significant difference (LSD) test with a significance level of $\alpha = 0.05$ and a confidence interval of 95%. In addition, the multiple range test (MRT) included in the statistical program was used to demonstrate the existence of homogeneous groups within each of the parameters.

RESULTS AND DISCUSSION

Drying kinetics analysis and mathematical modeling

The changes in the experimental moisture ratio as a function of time of olive-waste cake at different air drying temperatures (40-90 °C) are presented in Figure 1a. The equilibrium moisture content in the drying process for 40 °C was X_{we} = 0.020 g water g^{-1} DM, at 50 °C was X_{we} = 0.016 g water g^{-1} DM, at 60 °C was X_{we} = 0.009 g water g^{-1} DM, at 70 °C was X_{we} = 0.007 g water g^{-1} DM, at 80 °C was X_{we} = 0.004 g water g^{-1} DM and at 90 °C was X_{we} = 0.003 g water g^{-1} DM. As an example, an average temperature of air drying outlet (40 °C) is shown and modeled by the GAB equation (Figure 1b). The drying time required to reduce initial moisture 190.95 ± 1.21% (DM) to final moisture content was 2040, 1576, 1311, 1080, 960, and 848 min at air drying temperatures of 40, 50, 60, 70, 80, and 90 °C, respectively. The final moisture contents were: 10.46 ± 0.23%, 3.84 ± 0.22%, 1.29 ±



MR: moisture ratio (dimensionless, Equation [2]); X_{we}: equilibrium water content; a_w: water activity (dimensionless); GAB: Guggenheim, Anderson and de Boer model; M-K: Midili-Kucuk model.

Figure 1. a) Experimental and predicted moisture ratio of olive-waste cake ('Frantoio') as function of drying time and temperature, and b) experimental and predicted desorption isotherm at 40 °C.

0.53%, 1.08 ± 0.40%, 2.51 ± 0.31%, and 0.47 ± 0.97%, respectively.

The drying air temperature had a significant effect on the drying kinetics of olive-waste cake (Figure 1a). Thus, the moisture ratio decreased continuously with temperature following a decreasing exponential pattern (Doymaz et al., 2004). Drying rate is a function of air drying temperature since high temperature (e.g. 90 °C) led to shorter processing time to reach final water content. Comparable effects of temperature on drying kinetics were reported during drying of similar products (Doymaz et al., 2004; Göğüs and Maskan, 2006; Meziane, 2010; Vega-Gálvez et al., 2010).

Effective moisture coefficient and activation energy

As expected, effective moisture diffusivity values increased with a drying temperature increase between 40 and 90 °C. Values were 1.160×10^{-9} m^2 s^{-1} (40 °C), 2.056×10^{-9} m^2 s^{-1} (50 °C), 2.999×10^{-9} m^2 s^{-1} (60 °C), 3.699×10^{-9} m^2 s^{-1} (70 °C), 3.896×10^{-9} m^2 s^{-1} (80 °C), and 5.183×10^{-9} m^2 s^{-1} (90 °C). Comparable results were reported in previous works: $4.89\text{-}9.98 \times 10^{-10}$ m^2 s^{-1} in the range 80-110 °C (Doymaz et al., 2004), $1.50\text{-}2.20 \times 10^{-9}$ m^2 s^{-1} at 125 °C (Freire et al., 2001); $0.66\text{-}1.39 \times 10^{-9}$ m^2 s^{-1} in the range 40-70 °C (Milczarek et al., 2011); $1.84\text{-}3.94 \times 10^{-7}$ m^2 s^{-1} in the range 60-80 °C (Göğüs and Maskan, 2006), and $1.71\text{-}2.03 \times 10^{-9}$ m^2 s^{-1} in the range 50-90 °C (Vega-Gálvez et al., 2010).

ANOVA results obtained comparing mean diffusivity values showed a significant influence of drying air temperature on this parameter ($p < 0.05$). The influence of temperature on effective moisture coefficient has been described using Arrhenius type relationship. The activation energy (E_a) is the energy barrier that must be overcome to activate moisture diffusion and the diffusivity constant (D_o) equivalent to the diffusivity at infinitely high temperature. Thus, for this work the logarithm of D_{eff} as a function of reciprocal of the absolute temperature shows a linear relationship or an Arrhenius-type relationship with $r^2 = 0.9355$. Values of D_o and E_a calculated from the linear regression were 3.33×10^{-5} m^2 s^{-1} and 26.24 kJ mol^{-1}, respectively. The E_a values found for olive-waste cake were in agreement with data reported in previous studies: 26.71 kJ mol^{-1} (Doymaz et al., 2004), 17.97 kJ mol^{-1} (Akgun and Doymaz, 2005), and 25.4 kJ mol^{-1} (Göğüs and Maskan, 2006).

Mathematical modeling and statistical analysis

Several mathematical models were used to evaluate the best for representing experimental olive-waste cake drying data. Therefore, experimental data obtained were fit to these models and correlation parameters are presented in Table 1. Furthermore, Table 2 shows statistical test values (r^2, RMSE and χ^2) for the six selected models; where regarding to analysis resulting, it was showed that the Midilli-Kucuk (M-K) model gave a better fit to experimental data ($r^2 > 0.9987$, RMSE < 0.0012, $\chi^2 < 0.0002$). This good fit quality on experimental data can be explained because the Midilli-Kucuk model presents four terms, which provides a better mathematical

Table 1. Kinetic parameters of drying models as affected by temperature.

Temperature (°C)	Models	Constants							
	Midilli-Kucuk	*a*	0.99363 ± 0.0006	k	0.00075 ± 0.00018	b	-2.1E-06 ± 2.4E-06	n	1.1116 ± 0.0388
	Logarithmic	*a*	1.05353 ± 0.0125	k	0.00140 ± 0.00000	c	-0.0441 ± 0.0079		
40	Verma	*a*	0.00015 ± 0.0001	k	0.00227 ± 0.00116	c	0.0015 ± 0.0001		
	Weibull	*β*	641.30 ± 12.1550	α	1.0847 ± 0.02589				
	Modified Page	*k*	0.00156 ± 2.9E-05	n	1.0847 ± 0.02589				
	Midilli-Kucuk	*a*	0.99433 ± 0.0020	k	0.00096 ± 0.00048	b	-1.8E-05 ± 3.5E-06	n	1.1348 ± 0.0694
	Logarithmic	*a*	1.08717 ± 0.0263	k	0.00190 ± 0.00010	c	-0.0734 ± 0.0164		
50	Verma	*a*	0.00022 ± 0.0002	k	0.00437 ± 0.00199	c	0.0021 ± 0.0001		
	Weibull	*β*	443.06 ± 27.7168	α	1.1644 ± 0.06190				
	Modified Page	*k*	0.00226 ± 0.0001	n	1.1644 ± 0.06190				
	Midilli-Kucuk	*a*	0.98883 ± 0.0078	k	0.00061 ± 0.00018	b	-1.5E-05 ± 5.1E-06	n	1.2633 ± 0.0547
	Logarithmic	*a*	1.10367 ± 0.0215	k	0.00263 ± 0.00040	c	-0.0736 ± 0.0206		
60	Verma	*a*	0.00079 ± 0.0006	k	0.00310 ± 0.00061	c	0.0029 ± 0.0003		
	Weibull	*β*	317.31 ± 40.7699	α	1.2698 ± 0.03450				
	Modified Page	*k*	0.00319 ± 0.0004	n	1.2698 ± 0.03450				
	Midilli-Kucuk	*a*	0.98973 ± 0.0081	k	0.00066 ± 0.00005	b	-2.0E-05 ± 7.3E-06	n	1.2719 ± 0.0198
	Logarithmic	*a*	1.11270 ± 0.0197	k	0.00293 ± 0.00021	c	-0.0796 ± 0.0155		
70	Verma	*a*	0.00123 ± 0.0007	k	0.00337 ± 0.00015	c	0.0033 ± 0.0002		
	Weibull	*β*	286.97 ± 29.7716	α	1.2612 ± 0.01796				
	Modified Page	*k*	0.00351 ± 0.0003	n	1.2612 ± 0.01796				
	Midilli-Kucuk	*a*	0.98387 ± 0.0016	k	0.00082 ± 0.00034	b	-2.7E-05 ± 1.5E-05	n	1.2525 ± 0.0748
	Logarithmic	*a*	1.11993 ± 0.0083	k	0.00297 ± 0.00006	c	-0.0975 ± 0.0042		
80	Verma	*a*	0.00120 ± 0.0005	k	0.00363 ± 0.00032	c	0.0035 ± 0.0001		
	Weibull	*β*	267.65 ± 7.2373	α	1.1998 ± 0.03692				
	Modified Page	*k*	0.00374 ± 1.0E-04	n	1.1998 ± 0.03692				
	Midilli-Kucuk	*a*	0.98577 ± 0.0021	k	0.00061 ± 0.00024	b	-2.2E-05 ± 8.3E-06	n	1.3561 ± 0.0920
	Logarithmic	*a*	1.13510 ± 0.0037	k	0.00383 ± 0.00057	c	-0.0923 ± 0.0131		
90	Verma	*a*	0.00167 ± 0.0003	k	0.00443 ± 0.00046	c	0.0043 ± 0.0005		
	Weibull	*β*	228.91 ± 26.3634	α	1.2665 ± 0.07417				
	Modified Page	*k*	0.00441 ± 0.0005	n	1.2665 ± 0.07417				

Table 2. Statistical analysis of drying models as affected by temperature.

Temperature (°C)	Models	r^2	RMSE × 10^{-1}	χ^2
40	Midilli-Kucuk	0.9990	0.0111	0.00013
	Logarithmic	0.9986	0.0135	0.00020
	Verma	0.9966	0.0204	0.00045
	Weibull	0.9987	0.0124	0.00016
	Modified Page	0.9987	0.0124	0.00016
50	Midilli-Kucuk	0.9990	0.0109	0.00013
	Logarithmic	0.9983	0.0146	0.00023
	Verma	0.9933	0.0282	0.00089
	Weibull	0.9966	0.0218	0.00053
	Modified Page	0.9966	0.0218	0.00053
60	Midilli-Kucuk	0.9990	0.0105	0.00012
	Logarithmic	0.9966	0.0209	0.00049
	Verma	0.9891	0.0378	0.00159
	Weibull	0.9925	0.0302	0.00102
	Modified Page	0.9925	0.0302	0.00102
70	Midilli-Kucuk	0.9987	0.0125	0.00017
	Logarithmic	0.9962	0.0225	0.00057
	Verma	0.9875	0.0415	0.00193
	Weibull	0.9736	0.0244	0.00067
	Modified Page	0.9736	0.0216	0.00052
80	Midilli-Kucuk	0.9989	0.0120	0.00016
	Logarithmic	0.9967	0.0205	0.00048
	Verma	0.9886	0.0383	0.00168
	Weibull	0.9920	0.0279	0.00089
	Modified Page	0.9920	0.0279	0.00089
90	Midilli-Kucuk	0.9994	0.0088	0.00009
	Logarithmic	0.9945	0.0275	0.00087
	Verma	0.9834	0.0482	0.00268
	Weibull	0.9958	0.0168	0.00032
	Modified Page	0.9958	0.0185	0.00032

approximation on the drying curves with exponential tendency. Consequently, this model can provide a satisfactory description of the drying characteristics of olive-waste cake for whole range of drying temperatures (Figure 1a). Similar findings have been reported by Meziane (2010). Depending on drying data other models have been reported to give satisfying results on similar waste (Doymaz et al., 2004; Akgun and Doymaz, 2005; Göğüs and Maskan, 2006).

Physicochemical analysis and fatty acid composition

Table 3 shows the proximate composition of the samples dried at different temperatures. A clear influence of the drying temperature on the parameters determined with respect to the fresh samples was observed ($p < 0.05$). Parameters evaluated were moisture, crude fiber (CF), protein, fat, ash, and carbohydrate content was calculated by difference. Water activity is also presented in Table 3. The first two parameters had higher values than the fresh ones that in the dehydrated samples to different temperatures. The values of the proximate analysis of fresh olive cake differed from those reported in previous works (Molina-Alcaide and Yáñez-Ruiz, 2008; Sellami et al., 2008). The moisture and crude protein content of fresh olive cake used in drying experiments was 190.95 ± 1.21 and 6.83 ± 0.52 g 100 g^{-1} DM, respectively. Then, moisture and protein content varied from 0.63-3.24 and 6.76-7.76 g 100 g^{-1} DM, respectively, for dried olive cake samples. These values are similar to those reported by other authors (Hepbasli et al., 2003; Alhamad et al., 2012). The major constituent of fresh olive cake was CF with a mean value of 43.88 ± 1.66 g 100 g^{-1} DM showing that high percentage of CF is typical to olive cake, which is comparable to results presented by Alhamad et al. (2012). The CF contents of dehydrated olive cakes were also significantly different to that of the fresh sample ($p < 0.05$). A decreased CF in dehydrated samples was observed; at 50 °C decreased a 30%, at 90 °C decreased only 18%, showing a significant difference. An important aspect of the composition of olive cake is its high content of fiber, owing to the fact this product is composed of skin, flesh, and bone and lesser amounts of olive leaves. Regarding this aspect, this by-product is a good source of animal feeding (Chiofalo et al., 2004). A significant increase ($p < 0.05$) in fat content can be observed with regard to fresh sample (Table 3). The proximate analysis also showed significant differences ($p > 0.05$) between ash content of fresh and dehydrated samples.

Table 4 shows changes in individual fatty acids occurring during dehydration of the olive-waste cake. The oleic acid (18:1, 65.8%) was the major acid in the fresh sample followed by palmitic (C16:0, 13.6%) and linoleic (C18:2, 13.1%) acids. Most of the fatty acids of the waste were unsaturated fatty acids. Resistance of polyunsaturated oils to oxidation depends on factors such as exposure to oxygen, exposure to light, presence of pigments, and heavy metals as well as saturation degree (Stewart et al., 2003). Most of fatty acids increased their content with respect to control sample due to temperature increase ($p < 0.05$). The extent of degradation of some of them is dependent on the drying time and process temperature (Stewart et al., 2003). At 70 °C, oleic and palmitic acids of dehydrated samples showed the highest values. In particular, the oleic acid is nowadays considered as the preferred fatty acid for edible purposes, because it combines healthy benefits increasing of several diseases as well as a high oxidative stability (Chiofalo et al., 2004).

Table 3. Proximate analysis of fresh and dehydrated samples of olive-waste cake ('Frantoio'). Results expressed in g 100 g^{-1} DM.

Components	Fresh	40 °C	50 °C	60 °C	70 °C	80 °C	90 °C
Moisture	190.95 ± 1.21a	3.24 ± 0.07b	2.62 ± 0.14bc	1.81 ± 0.09cd	1.71 ± 0.02d	0.70 ± 0.09e	0.63 ± 0.06e
Fat	4.80 ± 0.10a	5.79 ± 0.17bc	5.85 ± 0.03bc	5.74 ± 0.27b	6.75 ± 0.13d	5.99 ± 0.10c	5.40 ± 0.03e
Ash	5.02 ± 0.33a	6.33 ± 0.33bde	7.61 ± 0.36c	6.76 ± 0.05d	6.02 ± 0.33b	6.24 ± 0.08bd	6.52 ± 0.14de
Protein	6.83 ± 0.52ac	6.76 ± 0.41a	6.78 ± 0.79a	7.03 ± 0.12ac	7.76 ± 0.2b	7.52 ± 0.14bc	7.03 ± 0.15ac
Crude fiber	43.88 ± 1.66a	33.95 ± 1.01bde	30.63 ± 0.46c	32.35 ± 0.47bc	34.72 ± 1.31de	33.07 ± 1.03bd	35.91 ± 1.87e
Carbohydrates	17.72 ± 0.16a	77.99 ± 0.39bc	77.21 ± 1.26b	78.70 ± 0.18cd	77.79 ± 0.5bc	79.56 ± 0.14de	80.42 ± 0.13e
a_w	0.93 ± 0.002a	0.065 ± 0.002b	0.056 ± 0.002c	0.048 ± 0.001d	0.051 ± 0.000e	0.029 ± 0.001f	0.026 ± 0.002g

Values are mean ± SD (n = 3). Different letter in rows indicates significant differences ($p < 0.05$).

Table 4. Effect of temperature on fatty acids content of olive-waste cake.

Fatty acids	Fresh	40 °C	50 °C	60 °C	70 °C	80 °C	90 °C
	g 100 g^{-1} dm						
Palmitic	0.622 ± 0.010a	0.858 ± 0.043b	0.741 ± 0.041cd	0.715 ± 0.025cd	0.859 ± 0.093b	0.791 ± 0.055bc	0.670 ± 0.014ad
Palmitoleic	0.127 ± 0.032a	0.127 ± 0.029a	0.276 ± 0.093ab	0.242 ± 0.075ab	0.302 ± 0.137bc	0.221 ± 0.076ab	0.454 ± 0.117c
Estearic	0.136 ± 0.014a	0.134 ± 0.042a	0.136 ± 0.016a	0.165 ± 0.022a	0.146 ± 0.007a	0.141 ± 0.014a	0.132 ± 0.017a
Oleic	3.016 ± 0.114a	3.724 ± 0.130b	3.913 ± 0.057bc	3.817 ± 0.192bc	4.469 ± 0.101d	3.997 ± 0.073c	3.472 ± 0.083e
Linoleic	0.602 ± 0.027a	0.863 ± 0.070b	0.661 ± 0.014a	0.668 ± 0.023a	0.834 ± 0.055b	0.752 ± 0.041c	0.605 ± 0.022a
Linolenic	0.074 ± 0.023a	0.064 ± 0.007a	0.104 ± 0.027b	0.113 ± 0.004b	0.119 ± 0.016b	0.074 ± 0.010a	0.055 ± 0.002a
Arachidonic	0.007 ± 0.002a	0.012 ± 0.003ab	0.013 ± 0.004b	0.016 ± 0.004b	0.016 ± 0.005b	0.011 ± 0.001ab	0.007 ± 0.001a
Eicosanoic	0.002 ± 0.000a	0.005 ± 0.001a	0.004 ± 0.000a	0.003 ± 0.001a	0.004 ± 0.002a	0.001 ± 0.000a	0.002 ± 0.001a

Means sharing different letters in rows are significantly different for each product type ($p < 0.05$).

Total phenolic, flavonoid, and flavanol contents

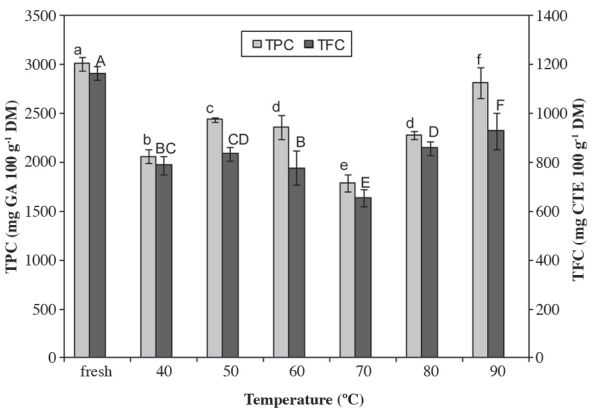
The initial content of total phenolic (TPC), total flavonoid (TFC) and flavanol contents of the olive-waste cake were 3003.34 mg GA 100 g^{-1} DM and 1163.34 mg catechin 100 g^{-1} DM, respectively. The initial TPC value was higher than those reported for residues of acerola, fruit passion, and pineapple (Cabral de Oliveira et al., 2009). The influence of drying temperature on TPC and TFC for dehydrated olive-waste cake can be observed in Figure 2. TPC and TFC decrease with increasing temperature ($p < 0.05$). At 70 ºC, the lowest retention of TPC (40.6%) and TFC (43.9%) were observed. These findings are in agreement with previous investigations (Martín-Cabrejas et al., 2009; Qu et al., 2010).

Some authors reported degradation of flavonoids and tannins (i.e., polymeric forms of flavanoids) as a result of drying treatments (Gu et al., 2004; Kofink et al., 2007; Hurst et al., 2011). Therefore, an optimum combination of drying time/temperature should be established for each product's polyphenolic profile to minimize the degradation of these bioactive compounds during the dehydration leading to recover phenolic compounds and derivatives (Ahmad-Qasem et al., 2013).

Several subcategories of flavonoids can be found, which are common in the human diet, including flavanols, flavonols, iso-flavones, flavones, and anthocyanidins. High levels of flavanols are found in cocoa, grapes, green and black teas, red wine, and apples (Gu et al., 2004). In recent years there has been an increasing interest in the health benefits of flavanol-containing foods. Initial content of flavanols in olive-waste cake was 39.31 mg catechin 100 g^{-1} DM. The influence of drying temperature on total flavanols is observed in Figure 3. Except, for 80 °C temperature increased the initial content of these compounds. Some authors reported an increase in flavanols during drying process which could be induced by high temperature (Kofink et al., 2007; Hurst et al., 2011). Waste olive cake, in some cases, exceeds the content of several food flavanols, thus this waste could be a rich source of these bioactive compounds (Francene et al., 2003).

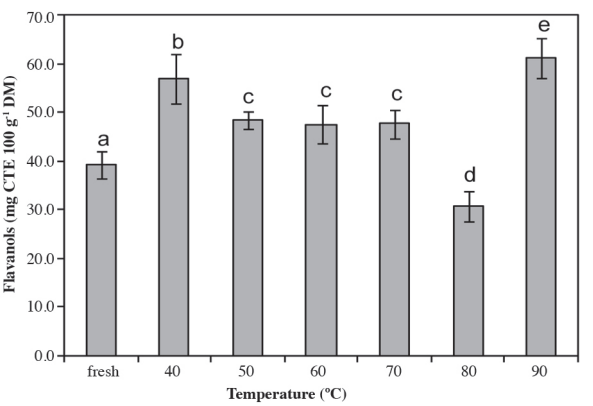
Determination of antioxidant capacity (ORAC and DPPH)

Antioxidant capacities of food/waste extracts not only depend on extract composition but also on the conditions of the test being used (Wu et al., 2004). Figure 4 presents the antioxidant capacity of olive waste cake by means of ORAC and DPPH analyses. The ORAC and DPPH values for fresh sample were 447.69 and 72.35 μmol TE g^{-1} DM, respectively. Effect of drying temperature on DPPH showed



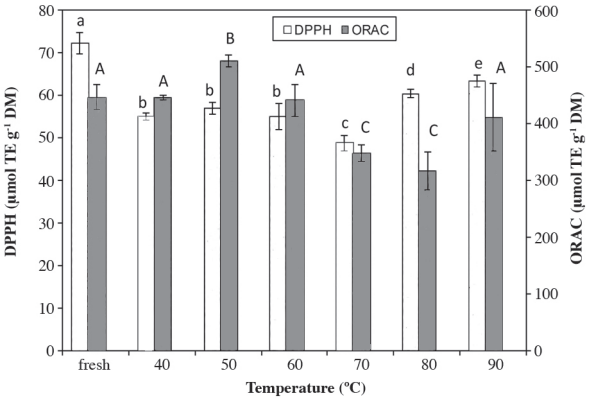
GA: gallic acid; CTE: catechin equivalents.
Values are mean ± SD (n = 3). Different letter indicates significant differences ($p < 0.05$).

Figure 2. Total phenolic (TPC) and total flavonoid (TFC) contents of olive-waste cake 'Frantoio' as affected by drying temperatures.



Values are mean ± SD (n = 3). Different letter indicates significant differences ($p < 0.05$).
CTE: catechin equivalents.

Figure 3. Total flavanols contents of olive-waste cake ('Frantoio') as affected by drying temperatures.



Values are mean ± SD (n = 3). Different letter indicates significant differences ($p < 0.05$).
TE: Trolox equivalents.

Figure 4. Antioxidant capacity (oxygen radical absorbance capacity [ORAC] and 2,2-diphenyl-2-picrylhydrazyl [DPPH] method) of the olive-waste cake ('Frantoio') as affected by drying temperatures.

significant results ($p < 0.05$). A decrease in antioxidant activity was observed with a minimum retention value of 67% at 70°C. These results are consistent with those reported in previous investigations and concluded that not only thermal treatment but also interactions between flavonoids could affect food antioxidant capacity (Hidalgo et al., 2010). Regarding to ORAC, drying temperature showed significant differences in samples ($p < 0.05$). The antioxidant capacity of dehydrated samples maintained or increased their initial value, with exception at 70 and 80 °C ($p < 0.05$). In particular, at 50 °C an increase of 14% of antioxidant activity was observed compared to fresh samples. Comparable results were reported by Miletić et al. (2013) working with plums.

To explore the influence of the TPC on antioxidant capacity in olive waste cake extracts, correlation between the antioxidant capacity and TPC was determined (Amro et al., 2002). Results of the ORAC assay were positive but weakly correlated to the TPC and TFC ($r = 0.382$; $r = 0.264$, respectively). On the other hand, DPPH results were positively correlated to TPC and TFC ($r = 0.923$; $r = 0.992$, respectively), indicating these compounds might be responsible for the antioxidant capacity exerted by the olive-waste cake. Additional studies are needed to characterize active compounds and biological activities of these active extracts so that they may be included in nutraceutical formulations.

CONCLUSIONS

Drying kinetics and quality indexes of olive waste-cake during convective dehydration were evaluated. The effective moisture diffusivity increased as drying temperature increased. Most of the fatty acids increased their content with increasing temperature. Regarding flavanols, except for 80 °C temperature increased the initial content of these compounds. 2,2-Diphenyl-2-picrylhydrazyl (DPPH) method results were positive correlated to the total phenolic content, total flavonoid content and flavanol content indicating that these compounds might be responsible for the antioxidant capacity of the dehydrated waste. These results indicated that the dehydrated olive-waste cake could be a source of compounds with bioactive function with potential use as additives for the food or other industries.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge the financial support provided by FONDECYT Regular N°1120101 Project and Research Department of Universidad de La Serena (DIULS), La Serena, Chile, for publication of this research.

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