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Quality Characterization of Waste Olive Cake During Hot Air Drying: Nutritional Aspects and Antioxidant Activity

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Abstract Olive cake, a by-product of the olive oil industry, was characterised through a drying process, where the influence of air drying temperature on physicochemical properties and antioxidant activity was investigated. A comparison of fresh and dehydrated olive cake showed that drying led mainly to denaturation of crude protein. Crude fibre content showed a slight increase during drying and may have undergone some alterations in its structure due to Maillard reactions. Fatty acid analysis revealed that olive cake was especially rich in oleic acid and fatty acid composition did not significantly change during drying. Ash content also showed a slight variation but may be considered as practically unchanged. Potassium and sodium were respectively the most and the least abundant minerals found in olive cake. Total phenolic content showed a direct relationship to DPPH radical scavenging activity. Overall antioxidant activity, highest in fresh olive cake, was affected by air drying temperatures being more evident at 90 °C. Vitamin E showed an increasing trend at all drying temperatures. According to this investigation, convective dehydration can lead not only to a dried olive cake that can be used as a material for many processing industries (e.g. food and cosmetic) but also can contribute to minimize the environmental impacts of this agro-industrial waste.

Keywords Olive cake · Convective dehydration · Phenolic compounds · DPPH · Antioxidant activity · Vitamin E

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Introduction

Favoured by appropriate climatic conditions in its central regions (Regions of Coquimbo, O'Higgins and Maule), Chile has steadily increased its olive oil production (ODEPA 2008) to join Spain, Italy, Greece, Turkey and Tunisia, large producers who together have marketed 97% of the world olive oil production (Doymaz et al. 2004). These Mediterranean countries still remained during 2007–2009 as the main producers of olive, with an average of 79.7% of world olive production, which then reached a total of 17.5 million metric tons (ODEPA 2010). Along with mechanical olive oil extraction, the

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olive industry has to face an important issue with accumulation of solid and liquid residues. With increasing emphasis on cost reduction of industrial processes and value addition to agro-industrial residues, oil cakes are seen as an ideal source of proteinaceous nutrients and could also find use as a support matrix for various biotechnological processes. Furthermore, several oil cakes—in particular, edible oil cakes—offer potential benefits when utilized as substrate for bioprocesses (Ramachandran et al. 2007; Akar et al. 2008; Canet et al. 2008; Thassitou and Arvanitoyannis 2001). The residual olive cake, being an important by-product of oil extraction process, could be used in the development of value-added products, which makes systematic studies indispensable.

Olive cakes consist of olive pulp, skin, stone, water and a remaining quantity of oil (Azbar et al. 2004, Roig et al. 2006; Hachicha et al. 2008). Due to its high water content, the cake needs to be dried or concentrated prior to further processing (Weinberg et al. 2008; Akar et al. 2008; Aboulkas et al. 2008; Krokida et al. 2002). Conventional air drying is one of the most frequently used operations for by-product treatments (Krokida et al. 2002; Doymaz et al. 2004; Ruiz-Celma et al. 2008). The drying process generally involves high energy costs, and the use of proper operating conditions is crucial to warrant the quality of final products and low environmental impact (Liébanes et al. 2008; Vega-Gálvez et al. 2010) since the effect of temperature on quality attributes may be maintained under control and a more efficient use of energy could be included in process design. The native physical state of product is also altered when materials are exposed to drying conditions, leading to changes in the quality and safety of food materials (Chen and Patel 2008). Thus, evaluation of waste utilization techniques needs to be performed together with characterisation of olive cake quality attributes and their changes during processing. The nutritional evaluation of olive wastes has been determined using chemical, physicochemical, biological and indirect methods. These by-products are usually characterised in terms of chemical and nutritional composition; however, compounds like phenols should be determined quantitatively as their content may fluctuate considerably during oil extraction process itself or during exposure to environmental conditions. Phenolic compounds, ubiquitous in plants, are an essential part of the human diet and are of considerable interest due to their antioxidant properties (Balasundram et al. 2006). The use of such wastes as nutrient sources for animals may further enhance the efficiency of industrial vegetal and animal production, consequently increasing profitability (Weinberg et al. 2008; Mussatto et al. 2011), considering that this waste product represents a cheap raw material with a high potential to be converted in valuable products after proper treatment. Some of these residues may also be used as a natural fertilizer or as a dye biosorbing material (Weinberg et al. 2008; Akar et al. 2008). In addition, it has also a great potential for bio-energy exploitation in many parts of the world since its energy content is over 15 MJ/kg dry matter and can therefore be used for direct burning after drying (Aboulkas et al. 2008; Krokida et al. 2002; Akgun and Doymaz 2005).

The aim of this study was to evaluate the effect of air temperature on changes occurring in the physicochemical and nutritional aspects of the olive cake during convective dehydration. The most relevant quality attributes of this important food by-product, namely, fatty acid composition, mineral and vitamin E contents, antioxidant activity and total phenolics, were analysed.

Materials and Methods

Raw Material and Drying Process

The olive cake was supplied by an agro-food company (Razeto) from the city of Quillota, Valparaiso, Chile. The waste resulted from a continuous cold process of olive oil production. The olive varieties used in this process were Frantoio, Leccino, Racimo, Barnea and Picual, harvested at optimum ripeness and pressed without delay. The samples used for analysis were packed in polyethylene bags and kept in a freezer at −20 °C. Before drying, olive cake samples were thawed during 24 h under refrigeration conditions at 5 °C.

Drying process was carried out at five different temperatures (50, 60, 70, 80 and 90 °C) in a convective dryer, built at the Department of Food Engineering of Universidad de La Serena, at a constant air flow rate of $2.0 \pm 0.2\ m s^{-1}$ (Vega-Gálvez et al. 2010). The dehydrated samples were packed and kept in the dark in polyethylene bags until analysis. All experiments were done in triplicate.

Physicochemical Analysis

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 (Merck, p.a.) as solvent according to the method described in AOAC method no. 920.39 (AOAC 1990). The crude fibre content was estimated by Weende method through an acid/alkaline hydrolysis of insoluble residues as described in AOAC method no. 962.09 (AOAC 1990). The crude ash content was estimated by incineration in a muffle furnace (Felisa, FE-341, Jalisco, Mexico) at 550 °C. All methodologies followed the recommendations of the Official Methods

of Analysis (AOAC 1990). The available carbohydrate was estimated by difference. The moisture level was determined by means of AOAC method no. 934.06 (AOAC 1990). The pH-value was measured directly on moist sample as described in AOAC method no. 945.10 (AOAC 1990) using an EXTECH Instruments micro-computer pH-vision 246072 (Waltham, MA, USA) and the level of titrimetric acidity at final pH 8.2 was expressed as oleic acid according to AOAC method no. 939.05 (AOAC 2000). All measurements were done in triplicate.

Determination of Minerals

Mineral elements (Na, Fe, K, Ca and Mg) were determined by atomic absorption using an AA spectrophotometer (Shimadzu Instruments, Inc., SpectrAA-220, Kyoto, Japan) after digestion in a mixture of H_2SO_4, HNO_3 and $HClO_4$. All determinations were done in triplicate. The mineral content was expressed in g 100 g^{-1} dry matter.

Determination of Fatty Acid Composition

For determination of fatty acid composition, methyl ester preparation and analysis were carried out in accordance to International Union of Pure and Applied Chemistry method (IUPAC 1987, no. 2.301 and 2.302). A gas chromatographer 6890N with flame ionization detectors from Agilent (Palo Alto, CA, USA), equipped with an automatic liquid sampler model 7683B, was used.

Determination of Vitamin E (α-Tocopherol)

The vitamin E content was determined by means of HPLC/fluorescence method as described by Ubaldi et al. (2005). A liquid chromatograph (Shimadzu Instruments, Inc., Shimadzu LC-10AD) was used for all determinations with methanol and acetonitrile (50:50) as mobile phase at a flow rate of 1.2 mL min^{-1} using a chromatography column Symmetry C18/10 cm. As standard, α-tocopherol (≥96%) from Sigma (St. Louis, MO, USA) was used. Monitoring of α-tocopherol was performed with a fluorescence detector (Shimadzu Instruments, Inc., Shimadzu RF-10 A xL). All measurements were done in triplicate. The vitamin E content was expressed in mg 100 g^{-1} dry matter.

Determination of Total Phenolic Content

Total phenolic content (TPC) was determined colorimetrically using Folin–Ciocalteu reagent (FC) according to Chuah et al. (2008) with modifications. Extract was obtained using 3 to 5 g dried and finely crushed sample, to which 40 mL of absolute ethanol was added. Mixture was homogenized during 24 h on a magnetic stirrer before filtering through a Whatman filter no. 1. The filter cake was washed twice with 40 mL of absolute ethanol and filtrate was evaporated under reduced pressure at 40 °C on a rotary evaporator (Büchi RE 121, Switzerland). Extract was then dissolved in 100 mL absolute ethanol and kept refrigerated until further analysis.

A 0.5-mL aliquot of the olive cake extract was transferred to a glass tube; 0.5 mL of reactive FC was added after 5 min followed by 2 mL of Na_2CO_3 solution (200 mg mL^{-1}). The sample was then mixed on a vortex mixer and the reaction proceeded for 15 min at ambient temperature. A total of 10 mL of ultra-pure water was then added and the precipitate formed was removed by centrifugation for 5 min at 4,000 × *g*. Finally, absorbance was measured at 725 nm in a spectrophotometer (Spectronic™ 20 GenesysTM131, IL, USA) and compared to a gallic acid (GA) calibration curve. Results were expressed as mg GA 100 g^{-1} dry matter. All reagents were purchased from Merck (Merck KGaA, Darmstadt, Germany) and all measurements were done in triplicate.

Determination of DPPH Radical Scavenging Activity

Free radical scavenging activity of the samples was determined using the 2,2,-diphenyl-2-picryl hydrazyl (DPPH) method (Turkmen et al. 2005) with some modifications. Extracts were obtained using 5 g of dried and finely crushed sample to which 50 mL of 80% (*v*/*v*) ethanol, was added and agitated for 30 min in an ultrasound water bath (Ultrasonik™ Cleaner, model 19H, Yupaica, CA, USA). The mixture was filtered (Whatman filter no.1) and filter cake was washed twice with 50 mL 80% (*v*/*v*) ethanol. The filtrates were mixed and evaporated under reduced pressure at 40 °C on a rotary evaporator (Büchi RE 121, Switzerland). The residue left after evaporation was used to prepare solutions of extract at required concentration (50 μg extract mL^{-1}) by adding 80% (*v*/*v*) ethanol in a 100-mL flask. The extract solution was kept refrigerated at 4 °C until analysis of antioxidant activity.

Dilutions of the extracts were prepared in triplicate. An aliquot of 2 mL of 0.15 mM DPPH radical in ethanol was added to a test tube with 1 mL of the sample extract. The reaction mixture was vortex-mixed for 30 s and left to stand at room temperature in the dark for 20 min. Absorbance was measured at 517 nm using a spectrophotometer (Spectronic® 20 Genesys®, IL, USA) at a fixed concentration of 50 μg extract mL^{-1}. The spectrophotometer was equilibrated with 80% (*v*/*v*) ethanol. Control sample was prepared without adding extract. All solvents and reagents were purchased from Sigma (Sigma Chemical CO., St. Louis, MO, USA).

The DPPH radical scavenging rate of sample was calculated as percent inhibition relative to control using the following equation and expressed as percentage inhibition:

$$\text{Inhibition}, \% = \left(1 - \frac{\text{Abs}_{\text{sample}}}{\text{Abs}_{\text{control}}}\right) * 100$$

where $\text{Abs}_{\text{sample}}$ and $\text{Abs}_{\text{control}}$ are, respectively, absorbance with and without sample extract.

Statistical Analysis

The effect of air drying temperature on quality parameters was estimated using Statgraphics® Plus 5.0 (Statistical Graphics Corp., Herndon, VA, USA). One-way ANOVA with five levels and three replicates was performed. Differences among the media were analysed using the least significant difference test with a significance level of $\alpha = 0.05$ and a confidence interval of 95% ($p<0.05$). In addition, the multiple range test included in the statistical programme was used to demonstrate the existence of homogeneous groups within each of the parameters.

Results and Discussion

Physicochemical Properties

Table 1 shows the mean values and standard deviations of proximate analysis of both fresh and dehydrated samples, including moisture, crude protein, fat, crude fibre and ash contents. Carbohydrate content can be calculated by difference. In samples of fresh olive cake, pH value and tritimetric acidity were 6.09±0.03 and 1.04±0.05 g oleic acid 100 g^{-1} dry matter, respectively. A proximate analysis of fresh and dehydrated samples showed that the drying process did cause some changes in the overall composition of the olive cake. Significant differences in respective contents in fresh and dehydrated samples were obtained together with homogeneous groups within the analysed samples ($p<0.05$). The values of the proximate analysis of the fresh olive cake differ from those reported in previous works (Molina-Alcaide and Yáñez-Ruiz 2008; Sellami et al. 2008). Factors such as proportion of different physical components (stone, skin, pulp and water), residual oil extraction, harvest year, geographic origin of olives and contamination with soil would cause great variability in chemical composition (Molina-Alcaide and Yánez-Ruiz 2008). The differences in chemical composition may also be due to the oil extraction process and degree of extraction (Mioč et al. 2007). The oil extraction method and extent of de-stoning also affect both the nutritional value and the preservation characteristics of the olive cake (Weinberg et al. 2008).

The moisture content of fresh olive cake used in drying experiments was 191.0±11 g 100 g^{-1} dry matter. The crude protein content of the fresh olive cake was also similar to those reported by other authors (Alburquerque et al. 2004; Hepbasli et al. 2003; Molina-Alcaide and Yáñez-Ruiz 2008). The main component of fresh olive cake was crude fibre with a mean value of 35.37±0.83 g 100 g^{-1} dry matter, showing that a high percentage of crude fibre is typical to olive cake, which is comparable to the results presented by Chiofalo et al. (2004). This high fibre content, associated with the presence of skin, pulp and stones, is an important quality attribute of olive cake. As a by-product of the olive oil industry, it would be a good source of animal feeds (Chiofalo et al. 2004; Weinberg et al. 2008). It could even be a potential source of dietary fibres, which are known for their health-promoting properties, for example, in reducing colon cancer, lowering serum cholesterol levels and preventing hyperglycaemia in diabetics (Garau et al. 2007). The crude fibre contents of the dehydrated olive cake were also significantly different to that of the fresh sample ($p<0.05$), being slightly higher in the dried samples. The variation of the crude fibre content in dehydrated samples presented values from 40.91±0.62 at 80 °C to 43.64±0.88 g 100 g^{-1} dry matter at 60 °C. This slight variation may be due to the formation of melanoidins, high molecular weight polymers, through Maillard reaction between sugars and amino acids (Adams et al. 2009; Wang et al. 2011). It should be noted that, during the drying process, protein content decreased as crude fibre content increased.

The proximate analysis also showed that ash content, which represents all minerals in the dry matter, remained as expected, practically unaltered, and no significant difference was found between the ash content of fresh and dehydrated cakes, respectively ($p>0.05$). As can be seen further in Table 1, the dehydrated olive cake samples showed, on the contrary, decreased levels of crude protein with respect to the fresh sample. The loss of protein could probably be the result of denaturation or changes in solubility during drying (Miranda et al. 2009). Denatured protein probably took part in the Maillard reaction to form melanoidins (Adams et al. 2009; Wang et al. 2011). With respect to crude protein content, the dehydrated samples obtained were not significantly different from one another, showing that the temperature level of the drying assays would not significantly affect the denaturation process. On the other hand, a decrease in fat content would be due to enzymatic hydrolysis during the drying period or to lipid oxidation through thermal treatment (Miranda et al. 2010). However, the fat content of samples did not show any significant change during drying. Only at higher temperatures (80 and 90 °C) was fat content observed to be slightly higher than that found in samples dried at lower temperatures (50, 60 and 70 °C), with a significant difference ($p<0.05$). This may be related to

Table 1 Proximate analysis of fresh and dehydrated samples of olive cakes (g 100 g^{-1} dry matter)

Parameters	Fresh	50 °C	60 °C	70 °C	80 °C	90 °C
Moisture	191.0±11.0 a	29.5±0.5 b,c	28.8±0.6 b	30.2±0.5 b, c	29.6±0.8 b, c	28.6±0.2 c
Crude protein	6.03±0.33 a	2.29±0.15 b	2.19±0.07 b	2.07±0.10 b	2.21±0.14 b	2.20±0.10 b
Fat	7.97±0.44 a, b	8.19±0.25 a, b	8.68±0.14 a	7.57±0.42 b	11.12±1.07 c	10.51±0.71 c
Crude fibre	35.37±0.83 a	42.22±0.73 b	43.53±0.15 c	43.64±0.88 c	40.91±0.62 d	41.40±0.78 b, c
Ash	5.70±0.36 a	5.47±0.39 a	5.56±0.08 a	5.49±0.41 a	5.40±0.36 a	5.48±0.38 a

Different letters in the same row indicate that values are significantly different ($p<0.05$)

the different fibre content of each respective sample (Table 2) or to an overall physical change in structure of the dry matter. This would consequently change the relative proportion of fat content in the dry matter. Some physical characteristics of the dry matter may have suffered some structural alterations, for example, during protein denaturation. The amino acids released would then react with other compounds such as sugars to produce dark brown-coloured polymers, known as melanoidins, via the Maillard reaction (Lee and Shibamoto 2002; Perera 2005). Browning reactions change colour, decrease nutritional value and solubility, create off-flavours and induce textural changes (Rahman 2008). Consequently, a probable overall alteration in the structure of the dry matter could have occurred.

Drying Behaviour

The experimental results of the drying process of olive cake can be seen in Fig. 1, where variation of moisture content is represented as a function of time for drying at five different air temperatures. The moisture content decreased steadily with time and more rapidly as temperature was increased. Drying rate was clearly a function of air drying temperature with a higher temperature leading to lower process time to reach equilibrium moisture content at a mean value of 0.293±0.005 $g g^{-1}$ d.m. All drying curves showed an exponential tendency that was modelled in a previous study (Vega-Gálvez et al. 2010). Similar effects of temperature on drying kinetics were reported during drying of olive cake by Arjona et al. (1999), Freire et al. (2001), Krokida et al. (2002), Doymaz et al. (2004), Akgun and Doymaz (2005) and Gogŭs and Maskan (2006). A constant rate period was not observed in the range of the air drying temperatures studied. Thus, the critical moisture content is equal to the initial moisture content. Therefore, the entire drying process occurred in the falling rate period, during which internal molecular diffusion is the predominant mechanism of mass transfer (Vega-Gálvez et al. 2010). After 300 min drying at 50 and 90 °C, MR reached a value of 0.20 and 0.14, respectively. Comparable results can be found in previous works (Arjona et al. 1999; Freire et al. 2001; Göğüş and Maskan 2006).

Effects on Some Essential Minerals

Table 2 shows the composition of five essential minerals, namely, iron, sodium, potassium, calcium and magnesium, in fresh and dehydrated olive cakes at different drying temperatures. ANOVA revealed a significant difference in the mineral content of dehydrated olive cake when compared to fresh olive cake ($p<0.05$) at each drying temperature. In fresh olive cake, potassium was found to be the most abundant mineral with a value of 36.85±0.454 mg 100 g^{-1} dry matter, while sodium was the least abundant with a value of 0.43±0.004 mg 100 g^{-1} dry matter. This was in close agreement with the observation of Alburquerque et al. (2004) that indicated olive cake to be especially rich in potassium, which is a common characteristic of olive mill

Table 2 Some essential minerals of fresh and dehydrated olive cake (mg 100 g^{-1} dry matter)

Mineral	Fresh	50 °C	60 °C	70 °C	80 °C	90 °C
Iron	4.49±0.075 a	5.91±0.006 b	4.05±0.501 c	3.31±0.024 d	3.05±0.024 d	5.25±0.139 e
Sodium	0.43±0.004 a	0.18±0.002 b	0.19±0.005 b,c	0.21±0.025 c	0.23±0.013 d	0.24±0.062 d
Potassium	36.85±0.454 a	12.87±0.061 b	12.78±0.012 b	12.80±0.060 b	12.62±0.143 b	12.79±0.046 b
Calcium	9.36±0.123 a	6.48±0.022 b	6.31±0.107 b	8.25±0.076 c	6.84±0.848 b	8.24±0.804 c
Magnesium	15.16±0.351 a	5.26±0.414 b	4.92±0.317 b	8.11±0.181 c	5.77±0.110 d	7.91±0.152 c

Identical letters in the same row indicate no significant difference ($p<0.05$)

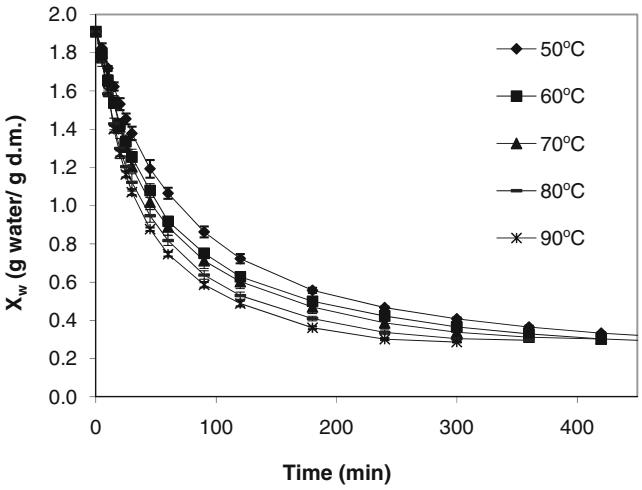

Fig. 1 Experimental drying curves of the olive cake dehydrated at different air temperatures

wastes and by-products. The potassium content in the fresh olive cake was also more than twice the amount found in the dehydrated samples, similar to the level of magnesium in the fresh sample that was also twice or thrice the amount found in the dehydrated samples. As for sodium, its content in the fresh sample is very near to twice the amount found in any of the dehydrated samples. On the other hand, the iron and calcium contents of dehydrated samples are almost of the same order as that found in the fresh sample. A decrease in contents of some specific minerals with respect to the fresh samples was observed and may be due to the diffusion of these micronutrients into intercellular spaces especially at high temperatures (Miranda et al. 2010), resulting in a random and non-homogeneous distribution of these minerals. Consequently, some differences in concentration of these minerals were observed in the analysed samples. Furthermore, the mineral content may vary widely among vegetables, depending on several factors such as ripeness, variety, soil type, the use of fertilizers, intensity and exposure time to sunlight, temperature, rain and cultivation area (Costa et al. 2003; Garcia-Hernandez et al. 2006).

Effects on Fatty Acid Composition

In Table 3, a summarized data of the qualitative and quantitative composition of fatty acids as determined in this work can be seen. Statistical analysis (ANOVA) of the data indicated that some of the fatty acids found in the fresh and the dehydrated olive cakes have significant differences in their contents ($p<0.05$). However, overall, only slight differences were observed. The fatty acid profile of the olive cake showed the lipids to be a good source of the nutritionally essential oleic and linoleic acids. Most of the fatty acids were unsaturated fatty acids, with oleic acid (73.07–73.08 g 100 g^{-1} fat) as the predominant fatty acid. Unsaturated linoleic acid (9.69–10.42 g 100 g^{-1} fat) had content almost similar to that of saturated palmitic acid (11.27–11.98 g 100 g^{-1} fat). The other fatty acids were found in small quantities (Table 3). The high amount of unsaturated fatty acids, especially the high amount of oleic acid, makes olive cake suitable for applications in animal nutrition, which will indirectly benefit human nutrition, too. It was found that ewe fed with olive cake mixed in their diet produced milk with an increased content of oleic acid and total monounsaturated fatty acid together with a drop in saturated fatty acids (Molina-Alcaide and Yáñez-Ruiz 2008). Furthermore, oleic acid is interesting in human nutrition for its beneficial effects on blood cholesterol and other health-related outcomes (Chiofalo et al. 2004).

Table 3 Fatty acid composition of the fresh and dehydrated olive cake (g 100 g^{-1} fat)

Fatty acids	Fresh	50 °C	60 °C	70 °C	80 °C	90 °C
Saturated fatty acid						
Palmitic (C16:0)	11.85±0.06 a	11.98±0.12 b	11.68±0.31 c	11.300±0.07 d	11.27±0.06 d	11.60±0.05 c
Estearic (C18:0)	2.65±0.01 a, b	2.64±0.02 a	2.69±0.04 b	2.860±0.02 c	2.79±0.02 d	2.77±0.01 d
Behenic (C22:0)	0.18±0.01 a	0.17±0.01 a, b	0.10±0.01 c	0.19±0.01 b, c	0.21±0.01 d	0.21±0.01 d
Unsaturated fatty acid						
Palmitoleic (C16:1)	0.68±0.01 a	0.66±0.01 b	0.64±0.01 c	0.58±0.01 d	0.64±0.01 c	0.68±0.01 a
Oleic (C18:1)	73.07±0.09 a	72.91±0.24 a	73.08±0.12 a	72.60±0.09 b	72.89±0.21 a	72.23±0.06 c
Linoleic (C18:2)	10.02±0.05 a	9.69±0.01 b	9.89±0.02 c	10.21±0.11 d	10.14±0.02 d	10.42±0.04 e
Gamma linolenic (C18:3)	0.43±0.01 a	0.43±0.01 a	0.45±0.01 b	0.46±0.01 b	0.48±0.01 c	0.49±0.01 c
Alpha linolenic (C18:3)	0.62±0.01 a	0.64±0.01 b	0.66±0.01 c	0.73±0.01 d	0.67±0.01 e	0.68±0.01 d
Eicosenoic (C20:1)	0.26±0.01 a	0.25±0.01 a,b	0.26±0.01 a, b	0.25±0.01 b	0.29±0.01 c	0.30±0.01 d
Nervonic (C24:1)	0.24±0.02 a	0.25±0.06 a	0.16±0.01 b	0.29±0.07 a,c	0.22±0.02 a, b	0.35±0.02 c

Different letters in the same column indicate that the values are significantly different ($p<0.05$)

Olive cake is also a potential raw material for extraction of oleic acid, which can be used in many products, as an excipient in pharmaceuticals or as emulsifying or solubilising agent in aerosol products (Smolinske 1992). Linoleic acid, on the other hand, can be used in quick-drying oil in oil paints and varnishes since it readily reacts with oxygen in air, leading to crosslinking and formation of a stable film. It is also used in the cosmetic industry due to its beneficial properties, having anti-inflammatory, acne-reductive and moisture-retentive properties when applied topically on the skin (Letawe et al. 1998). The nutritional value of linoleic acid is also recognized due to its metabolism at tissue levels which produce the hormone-like prostaglandins. The activity of these prostaglandins includes lowering of blood pressure and constriction of smooth muscle (Zia-Ul-Haq et al. 2007). Linoleic and linolenic acids are also important essential fatty acids required for growth, physiological functions and maintenance (Zia-Ul-Haq et al. 2007).

In general, the drying method used in this study did not cause substantial changes in the qualitative and quantitative composition of the fatty acids. However, total fat content decreased during the drying process, implying also a decrease in quantity of total fatty acids present per gram of waste olive cake, although the quantitative composition per gram of fat did not change. The preservation of the fatty acid profile may be due to the application of relatively low heating temperature (maximum 90 °C), showing that all the fatty acids present in the olive cake were stable within this temperature range. Any decrease in fatty acid content may be ascribed to enzymatic hydrolysis or to oxidation due to thermal treatment (Miranda et al. 2010), whereas any apparent increase may be due to probable structural changes of the dry matter as explained previously in the case of fat content.

Antioxidant Activity

Antioxidants can inhibit or retard oxidation in two ways: either by scavenging free radicals, in which case the compound is described as a primary antioxidant, or by a mechanism that does not involve direct scavenging of free radicals, in which case the compound is a secondary antioxidant. Primary antioxidants include phenolic compounds of the lipophilic group such as α-tocopherol, well known as a component of vitamin E (Ibañez et al. 2000). Loss of this food component by radical-catalysed reactions may often accompany lipid oxidation (Maestri et al. 2006). Figure 2 shows the effect of drying temperature on α-tocopherol or vitamin E for the fresh and dehydrated olive cake samples. The initial content of vitamin E was 2.207±0.07 mg 100 g^{-1} dry matter. All rehydrated samples showed significant changes in the vitamin E content compared to the fresh olive cake ($p<0.05$). Although α-tocopherol is heat-stable, it is susceptible to oxidation, leading to the formation of products with low water activity (e.g. dimers, trimers and α-tocopherol quinines) at temperatures above 60 °C (Kumar et al. 2001; Miranda et al. 2009). However, a comparison of the data for the dehydrated samples with that for the fresh reference sample indicated that none of the drying conditions employed had any destructive effect on tocopherols ($p<0.05$). On the contrary, an increase in vitamin E was observed at all drying temperatures. The enhancement in vitamin E content could be due to tocopherols scavenging lipid peroxy radicals, yielding a tocopheroxyl radical that can be recycled back to the corresponding tocopherols by reacting with ascorbate or other antioxidants through different chemical reactions (Sattler et al. 2004). Depending on the food matrix, a significant amount of vitamin E linked to proteins or phospholipids could be released by heat treatment breakdown. Thus, a higher content of this vitamin can be obtained compared to the non-processed olive cake (Casal et al. 2006).

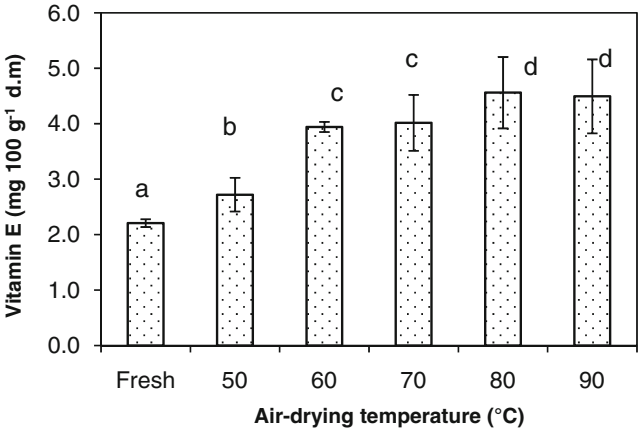

Fig. 2 Effect of air drying temperature on vitamin E content of fresh and dehydrated olive cake. *Identical letters above the bars* indicate no significant difference ($p<0.05$)

Vitamin E also acts as an antioxidant at the cell membrane level, protecting the fatty acids of the membranes against damage caused by free radicals (Repo-Carrasco et al. 2003). Drying process did not show relevant changes in composition of fatty acids in the olive cake, which coincide with the results for vitamin E. This may be a reflection of the general correlation between the degree of unsaturation of lipids in natural materials and their vitamin E content (Chow and Draper 1969). The drying process of olive cake can therefore be carried out to enhance vitamin E content, which would prevent degradation of the unsaturated fatty acids.

Polyphenols are other bioactive compounds present in solid olive waste (Roig et al. 2006; Aludatt et al. 2010). They are potent antioxidants and play an important role in the chemical, organoleptic and nutritional properties of

virgin olive oil and table olives. Olive fruits have a characteristic phenolic composition, which depends qualitatively and quantitatively on the type of olives, stage of maturity, season, storage time, processing technique and/or climatic conditions (Aludatt et al. 2010). During the mechanical extraction process of olive oil, the major proportion of the phenolic compounds will flow into the aqueous phase so that only a minor percentage (<1%) will be found in the olive oil. This explains why a large fraction of them can be found in the solid residues. The use of two-phase centrifugal decanters in olive oil extraction compared to the conventional three-phase extraction mode leads generally to a virgin olive oil that has a greater concentration of phenolic compounds; however, the resulting solid residue, known also as "alperujo", will retain about 98% of the phenolics (Fernández-Bolaños et al. 2006).

In this study, TPC was determined according to Folin–Ciocalteu method with GA as standard; a linear relationship was obtained between absorbance due to TPC and GA concentration (r^2=0.99, y=0.0047x+0.0635). TPC was found in a range between 185.07±34.5 and 1,910.24±62.67 mg GA 100 g^{-1} dry matter. TPC in the fresh olive cake was 1,910.24 mg GA 100 g^{-1} dry matter, which was less than that reported by DeJong and Lanari (2009), who reported a mean value of 2,680 mg GA 100 g^{-1} dry matter, but greater than the values between 4 and 437 mg GA 100 g^{-1} dry matter as reported by Aludatt et al. (2010), who worked on olive cake from Jordan.

The influence of drying temperature on TPC for fresh and dehydrated olive cakes can be seen in Fig. 3 (p<0.05). An increase in drying temperature caused a notable loss in TPC, leading to an evident reduction of these components, in particular, above 70 °C. This observation may be explained by the fact that most phenolic compounds are heat-sensitive or labile under heat treatment (Vashisth et al. 2011). Loss of TPC due to thermal degradation has been also reported by other authors (Miranda et al. 2010; Vashisth et al. 2011). In addition, decrease in TPC during dehydration may also be ascribed to binding of the polyphenols with other compounds like proteins or to alterations in the chemical structure of the polyphenols that cannot be extracted and determined by available methods (Qu et al. 2010).

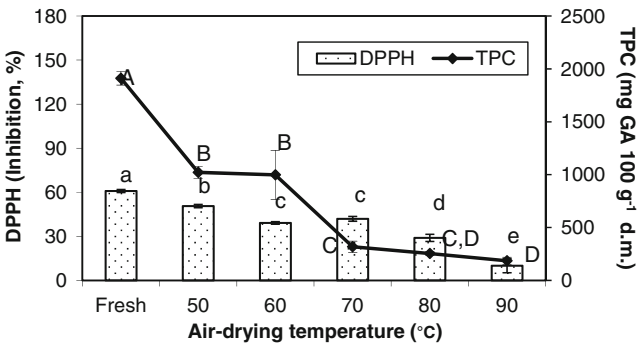

Fig. 3 Effect of air drying temperature on TPC and DPPH-free radical scavenging activity of fresh and dehydrated olive cake. *Identical letters above the bars* indicate no significant difference (p<0.05)

The stable DPPH radical, which has a maximum absorption at 517 nm, is widely used to evaluate the free radical scavenging activity of hydrogen-donating antioxidants found in many plant extracts (Pugalenthi et al. 2004; Turkmen et al. 2005; Katsube et al. 2009; Murthy and Naidu 2010). The DPPH radical scavenging activity of olive cake samples shown as % inhibition is presented also in Fig. 3. The fresh sample (60.86%) showed a relatively higher radical scavenging activity than the dehydrated samples (p<0.05). Samples dehydrated at 90 °C presented retention of 16% of the initial antioxidant capacity of the cake. These results are consistent with those reported by Bennett et al. (2011) who concluded that variations in drying and processing conditions would affect food antioxidant capacity.

Amro et al. (2002) concluded that the presence of the phenolic compounds in the olive cake would explain their activity as antioxidants and radical scavenging agents. The reduced levels of the phenolic compounds found in the analysed olive cake correlated well to the antioxidant activity (Fig. 3), indicating that the decrease of antioxidant activity in olive cake air-dried at different temperatures resulted from the degradation of the phenolic compounds. Between DPPH radical scavenging activities and the levels of phenolic compounds in the olive cake air-dried at various temperatures, a relatively good correlation was obtained (r^2=0.68, y=0.02x+21.51). However, the relationship between TPC and antioxidant activity of foods during dehydration is still a complex issue due to various factors, including drying method, type of extraction solvent, antioxidant assays, nature of phytochemicals and interactions of multiple antioxidant reactions (Miranda et al. 2010). These results suggest TPC to be directly related to the level of measurable antioxidant activity. Although some of the endogenous antioxidants in a sample may be destroyed during drying, the overall antioxidant properties of food might be enhanced by the release or generation of new antioxidant species as a consequence of processing. This behaviour would occur for two reasons: First, at a high temperature, new compounds can be generated as a result of non-enzymatic browning or Maillard reaction (Adams et al. 2009; Wang et al. 2011). These compounds, referred to as melanoidins or Maillard reaction products (MRPs), possess antioxidant activity and function as an antioxidant via a chain-breaking mechanism. Several authors have observed that the antioxidant activity afforded by the generation of MRPs does not necessarily compensate for the loss resulting from the degradation of the phenolic compounds (Lee and Shibamoto 2002; Perera 2005;

Vashisth et al. 2011). Second, during oxidation of polyphenolics, the oxidation products formed during the intermediate stages have shown to possess greater antioxidant activity than the endogenous polyphenolics; however, these intermediate compounds are only temporary. At the same time, constituents with moieties possessing antioxidant behaviour and bound to different components of the food or plant matrix can be released or cleaved from cell walls during thermal operations, thereby allowing them to exhibit antioxidant activity (Vashisth et al. 2011).

Conclusion

In this study, olive cake was subjected to convective drying and the effect of air temperature between 50 and 90 °C on physicochemical properties as well as antioxidant activity due to total phenolic compounds was investigated. A proximate analysis of fresh and dehydrated olive cakes showed that drying within the given temperature range did cause some changes in overall composition ($p<0.05$). Besides the obvious decrease in moisture, denaturation of crude protein was the most noticeable change. However, only slight differences were observed in crude protein contents of the dehydrated samples so that the drying temperature did not cause any drastic alteration. The high content of crude fibre was shown to be typical in olive cake, making it a potential source of dietary fibre and useful in animal nutrition. Variation in crude fibre content may be due to protein denaturation and formation of Maillard reaction products which probably caused some alteration in the structure of the dry matter. Fat content and fatty acid composition did not show any significant change during drying. A slight increase in fat content at 80 and 90 °C seemed to be related to some physical changes in dry matter. Ash content, as expected, remained practically unaltered, although some of the essential minerals showed a decrease in content with respect to the fresh samples, which may be due to the diffusion of these micronutrients into intercellular spaces especially at high temperatures. Potassium was the most abundant mineral while sodium was the least. It was also observed that vitamin E destruction did not occur; on the contrary, its content was lower in fresh olive cake, which may be attributed to a less efficient extraction from a high moisture sample. DPPH radical scavenging activity of olive cake samples significantly decreased, showing the minimum value at 90 °C ($p<0.05$). Between DPPH radical scavenging activities and the levels of phenolic compounds in the olive cake air-dried at various temperatures, a relatively good correlation was obtained. The observed decrease of antioxidant activity in the air-dried olive cake resulted from the degradation of the phenolic compounds. The reported results revealed that this by-product is an important material which can be used for the food and cosmetic industries because it is a very promising source of value-added substances as well as a relief to environmental issues of the olive oil processing industry. In the light of the reported results, drying at 70 °C at a constant air flow rate of 2.0 ± 0.2 ms^{-1} may give a dried olive cake of high quality at a reasonable energy input.

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