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Influence of air temperature on drying kinetics and antioxidant potential of olive pomace

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ABSTRACT

This work aims to evaluate the influence of olive pomace drying (a solid by-product of the olive oil industry) on both antioxidant potential and drying kinetics. The two main fractions of olive pomace (pits, PI and pulps + peels, P + P) were characterized by image analysis and density measurement. The drying process was analyzed in experiments carried out at different temperatures (from 50 to 150 °C) and mathematically described from the diffusion and Weibull models. The antioxidant potential of the extracts (ethanol–water 80:20 v/v, 22 ± 1 °C, 170 rpm for 24 h) obtained from the dry product was analyzed by measuring the total phenolic content and antioxidant capacity and the main polyphenols were quantified by HPLC–DAD/MS–MS.

The drying behavior of olive pomace was well described by considering the diffusion in the PI and P + P fractions separately and the influence of temperature on effective moisture diffusivities was quantified by an Arrhenius type equation. The antioxidant potential was only mildly influenced by the drying temperature. However, long drying times at the highest temperature tested (150 °C) significantly ($p < 0.05$) increased the antioxidant potential.

1. Introduction

The olive (*Olea europea*) is an evergreen tree traditionally cultivated for the production of oil and table olives. As regards both wealth and tradition, the olive oil industry is a relevant one, especially in the Mediterranean countries where 97% of the world's olive production is harvested. Spain is the leading country in terms of the total crop surface and the number of productive trees (Niaounakis and Halvadakis, 2004).

Nowadays, the olive oil industry generates a great environmental impact due to the production of high polluting residues (Baeta-Hall et al., 2005). Several studies have stated the negative effects of these forms of waste on soil's microbial populations (Paredes et al., 1987), aquatic ecosystems (DellaGreca et al., 2001) and even on the air (Rana et al., 2003). However, olive polyphenols, such as oleuropein, verbascoside or hydroxytyrosol, are present not only in olive oil but also in oil waste products, exhibiting among other things, antiviral, antitumoral and antioxidant activities (Della Ragione et al., 2000; Liu et al., 2003; Micol et al., 2005). One of the most problematic olive oil waste products is pomace (the solid byproduct made up from pieces of pit, skin and pulp), also known as cake. Actually, it is used for animal feed, residual oil extraction, energy recovery, soil amendment or the extraction of valuable polyphenols (Roig et al., 2006). A previous dehydration stage reduces the pomace water content to 5–6% (wet basis), aiming to stabilize the byproduct and so avoiding undesirable degradation during storage. Moreover, in the particular case of bioactive compound extraction, drying avoids the interference of water in the polyphenol release (Soysal and Öztekin, 2001), improving the extraction yield. For industrial purposes, hot air drying is the most widely used method, since it allows an accurate control of the process variables. Traditionally, low air temperatures are used as a means of better protecting the bioactive compounds from degradation during drying. However, drying at low temperatures constitutes a slow process in which metabolic reactions may be long lasting, leading to quality loss (Fennell et al., 2004). Thereby, certain studies also suggest the use of high temperatures for the industrial drying of olive pomace (Göğüs and Maskan, 2006). High temperatures speed up the drying kinetics, which could be interesting for the purpose of increasing productivity on an industrial scale (Ahmad-Qasem et al., 2013a), but at the same time it could promote the oxidative degradation of polyphenols (Gomes and Caponio, 2001) and requires the use of a great amount of energy.

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For this reason, the main aim of this work was to assess the influence of the air temperature on the drying kinetics and antioxidant potential of olive pomace, two aspects which have not previously been considered together.

2. Materials and methods

2.1. Raw material

The raw material used in this work was olive pomace from a traditional pressing system for obtaining olive oil, provided by an oil factory located in Altura (Castellón, Spain) The pomace was collected just after the pressing operation and immediately vacuum packaged and stored at 4 °C. The initial moisture content was determined by drying in a vacuum chamber at 70 °C until reaching constant weight (AOAC method n° 934.06, AOAC, 1997).

It could be considered that olive pomace is mainly composed of two main fractions: pits (PI) and pulps + peels (P + P). Homogeneous samples of olive pomace were taken, both fractions were separated by hand and their corresponding mass fraction (X) calculated and characterized by image analysis (Table 1). RGB images were taken (Fig. 1a and c) and processed using Image J software (Research Service Branch, National Institute of Mental Health, US, available as freeware from http://rsbweb.nih.gov/ij/). Images were converted to the binary system (Fig. 1b and d) using an automatic threshold. Finally, the particles were counted and their surface (S, mm^2) calculated considering the scale reference. From another set of experiments, the initial moisture content of both fractions was also determined, as already explained for the olive pomace.

The bulk density (ρ) of both the PI and P + P fractions, as well as that of the fresh olive pomace, was determined at 20 °C by liquid displacement using water, a volumetric standard picnometer (48.89 mL) and an analytical balance (PB 303-S, Mettler Toledo).

2.2. Drying experiments

Drying experiments were conducted in a forced air laboratory drier (FD, Binder, Tuttlingen, Germany), using a horizontal air flow of 0.094 m^3/s and an air velocity of 0.683 m/s. Each run was carried out with an initial mass load of 40 g of olive pomace, uniformly distributed in a monolayer (4 ± 1 mm thick, 0.083 g/cm^2).

Two different sets of experiments were designed. In the first one, the variable to be considered was that of the air temperature in order to determine its influence on both the drying kinetics and antioxidant potential of the extracts obtained from the dried product. For this purpose, the drying experiments were carried out at different air drying temperatures: 50, 70, 90, 120 and 150 °C. During the process, the samples were weighed (XS204, Mettler Toledo, Barcelona, Spain) at pre-set times. The drying experiments finalized when the sample weight loss reached 30 ± 1%. This fact was established by previous experiments, ensuring that the water activity was below 0.4 and the obtained product was stable.

In the second set of experiments, the variable to be studied was the drying time. For that purpose, drying experiments were carried out at 150 °C and for different drying times: 5, 10, 20, 30 and 60 min. It should be highlighted that, in this set of experiments the effective drying period took place between 5 and 10 min, from which mass transfer could be considered negligible. Therefore, this involves overexposing the olive pomace to a high temperature (150 °C).

The drying experiments for each experimental condition tested were carried out three times, at least.

Table 1
Characterization of pit (PI) and pulp + peel (P + P) fractions of olive pomace.

	PI	P + P
ρ (kg/L)	1.30 ± 0.05	1.5 ± 0.2
W_0 (g w/g d.w.)	0.234 ± 0.004	0.66 ± 0.05
X	0.424 ± 0.005	0.576 ± 0.005
r_m (mm)	1.80 ± 0.02	0.311 ± 0.015
Y_1 (S_p > 10 mm^2)	0.584	0.502
Y_2 (1 < S_p < 10 mm^2)	0.381	0.384
Y_3 (0.25 < S_p < 1 mm^2	0.021	0.063
Y_4 (S_p < 0.25 mm^2)	0.015	0.051

ρ = density, W_0 = initial moisture content, X = mass fraction, r_m = characteristic dimension (thickness in PI and radius in P + P fraction), Y = sub-fraction of particles with a specific surface (S_p, $Y_1 + Y_2 + Y_3 + Y_4 = 1$).

2.3. Modeling of hot air drying kinetics

The experimental drying kinetics were determined from the initial sample mass and the weight loss measured during drying. Previous approaches to the modeling of the drying kinetics of olive pomace have been addressed through the use of deep beds, assuming in the modeling that the sample is as thick as the bed is high and that it behaves like an infinite slab (Göğüs and Maskan, 2006). In addition, Vega-Gálvez et al. (2010) molded olive cake into a rectangular form and conducted drying experiments in monolayer at different temperatures in order to identify an effective moisture diffusivity in this particular body. However, the drying of the individual particles of olive pomace has not previously been addressed. This could be considered a complicated issue, since olive pomace is a heterogeneous material made up mainly of pits, peels and pulp pieces, which represents a handicap when using a diffusion model where the samples are assumed to be homogeneous. In this work, therefore, the monolayer drying of this byproduct has been studied in order to identify the drying behavior of olive pomace at particle level, and further studies should be performed to address the drying of the bulk of the olive pomace. For that purpose, two different approaches were considered.

On the one hand, a diffusion model for the olive pomace was used by considering the diffusion in both fractions of the olive pomace to be different: Pits (PI) and pulps + peels (P + P). It was assumed that pits could be considered as geometrically spherical particles, while peels + pulps could behave like infinite slabs. Eqs. (1) and (2) show the solution of diffusion models for spheres and infinite slabs, respectively, considering:

- Homogenous and isotropic solids.
- Constant effective diffusivity.
- Negligible shrinkage.
- Uniform initial moisture and temperature.
- The solid surface at equilibrium with the drying air.
- Solid symmetry.

$$W_{\mathrm{PI}} = W_e + (W_c - W_e)\left[\sum_{n=1}^{\infty}\frac{6}{n^2\pi^2}\exp\left(-\frac{D_e^{\mathrm{PI}}}{R^2}n^2\pi^2 t\right)\right] \quad (1)$$

$$W_{\mathrm{P+P}}(t) = W_e + (W_c - W_e) \times \left[\sum_{n=0}^{\infty}\frac{8}{(2n+1)^2\pi^2}\exp\left(-\frac{D_e^{\mathrm{P+P}}(2n+1)^2\pi^2 t}{4L^2}\right)\right] \quad (2)$$

where W is the average moisture content (dry basis), subscripts c and e refer to the critical and equilibrium states, t (min) the drying time, D_e is the effective moisture diffusivity (m^2/s), which was considered to be different in both PI and P + P fractions. The characteristic diffusion paths, radius (R) and thickness (L), were experimentally determined. The average radius of pit pieces was obtained from image analysis. For that purpose, the radius of