Review of the Drying Kinetics of Olive Oil Mill Wastes: Biomass Recovery

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The drying kinetics of olive oil mill wastes was analyzed based on experiments carried out by various researchers utilizing different drying systems. A critical review of the literature was done, and mathematical models of drying curves proposed by investigators were evaluated. A comparison between the best mathematical models of fit in the drying curves used in past experiments and a two-term Gaussian model was performed. This model improved all the results of fit in each experiment. Drying rates and drying stages were obtained and discussed. An average drying rate for each experiment from the two-term Gaussian model was calculated. This value allowed for visualizing and comparing the average speed of evaporated water in each experiment for the different dryers. Finally, and after having verified that almost all drying occurs mainly by a diffusion phenomenon, an analysis on the effective moisture diffusivity and activation energy values was performed. The results indicated that there was no dependency of these quantities on independent variables such as the drying air temperature, the drying air velocity, and the sample thickness. It follows that drying of olive oil mill wastes is a very complex physical process that depends heavily on aspects such as pieces of pit, pulp, skin, vegetation water, olive oil content, sugars and organics compounds of different nature.

Keywords: Drying kinetics; Drying rate; Olive oil mill waste; Biomass; Modeling; Effective moisture diffusivity

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INTRODUCTION

Olive oil is one of the most important foods in the Mediterranean diet, particularly extra virgin olive oil, which has excellent health and nutritional benefits. The annual average production of olive oil is around 2.8 millions of tonnes (International Olive Council (IOC) 2014b). Approximately two million tonnes are produced in the European Union, which is 70% of the world production. Spain, Italy, and Greece are the largest producers of olive oil with annual average productions of 1.215, 0.456, and 0.318 million tonnes, respectively (International Olive Council (IOC) 2014a).

Throughout the 20th century, traditional pressing methods dedicated to olive oil extraction have gradually been replaced by new technologies based on different procedures such as milling and crushing through the use of stainless steel hammers, thermo-mechanical beating, and horizontal centrifugation in continuous decanting systems

(Boncinelli *et al.* 2009; Altieri *et al.* 2013). Nowadays, there are two decanter systems used for olive oil extraction: the three-phase system and the two-phase system.

Since the early seventies and through the nineties, the extraction process of olive oil was carried out in decanters of three phases: virgin olive oil, olive cake (in Spanish called “orujo”), and vegetation water with organic compounds (in Spanish called “alpechín”). The olive cake was a by-product formed from the skin, pieces of pit, and pulp of the olives and a small amount of olive oil, between 5% and 8%; however, this system presented several environmental problems. First, large quantities of water were necessary before the decantation to separate the three phases and, second, the “alpechín” that was stored in reservoirs constructed near the olive oil mills, presented a serious environmental problem due to its high biochemical oxygen demand (BOD). Currently, the three-phase system is still being used in several countries such as Turkey, Greece, and Tunisia.

A two-phase extraction system was instituted in the early nineties with the objective of eliminating the environmental problems caused by the three-phase system. In this system only two phases are obtained: olive oil and a by-product formed by a mixture of olive cake and vegetation water (in Spanish called “alpeorujo”, mixture of “orujo” and “alpechín”); however, this system produces a by-product with a high moisture content– between 60% and 70% (wet basis)–in comparison with the moisture content of olive cake obtained in the three-phase system–between 35% and 45%. The two-phase system is currently fully implemented in Spain and Italy.

Olive oil mill wastes need to be dried for three main reasons: to avoid a hazardous environmental problem, to extract the remaining olive oil, and finally to obtain a biomass product (in Spanish called “orujillo”). In order to extract the olive oil in these by-products, both “orujo” and “alpeorujo” need to be dried to achieve values close to the equilibrium moisture content, about 7.5% (wet basis) (Moral and Méndez 2006). Under these conditions the solvent used in the olive pomace oil extraction process is much more effective. After this process, a biomass product with a significant net calorific value, about 17.5 MJ/kg, is obtained, the “orujillo”. This abundant green energy source has special interest as biofuel for generating electrical energy in cogeneration and generation plants (Jurado *et al.* 2003; Cruz-Peragón *et al.* 2006), and for generating thermal energy in space heating (García-Maraver *et al.* 2012; Rosúa and Pasadas 2012).

Presently, the physical drying process is a unique method used to treat olive oil mill wastes; the vast majority is dried in co-current rotary dryers (Gómez-de la Cruz *et al.* 2015b). Large quantities of these by-products are annually treated in secondary extraction factories. For drying, hot air flows at high temperatures (between 600 ºC and 1000 ºC), and its study is focused on knowing the drying air temperatures, drying air flows, and moisture content of these wastes at the inlet and outlet of the rotary dryer (Casanova-Peláez *et al.* 2012). However, there have been no studies on the drying kinetics in rotary dryers. Furthermore, this sector has been relentlessly buffeted by the possible risk of the development of carcinogenic substances (polyaromatic hydrocarbons like benzopyrene) in the olive pomace oil due to high temperatures used in the drying process.

These circumstances have boosted the development and study of new drying systems. The drying kinetics of “alpeorujo” and “orujo” have been studied in other alternative drying systems such as solar dryers (Celma *et al.* 2007; Montero *et al.* 2010; Montero *et al.* 2011), fluidized bed dryers (Liébanes *et al.* 2006; Meziane 2011), convective dryers (Arjona *et al.* 1999; Krokida *et al.* 2002; Doymaz *et al.* 2004; Akgun and Doymaz 2005; Gögüs and Maskan 2006; Vega-Gálvez *et al.* 2010; Casanova-Peláez *et al.* 2015), microwaves with convection-assisted dryers (Gögüs and Maskan 2001; Milczarek *et al.* 2011), and infrared dryers (Ruiz Celma *et al.* 2008). Generally, drying kinetics are carried out from the variation of mass of the sample studied for different conditions of temperature (isothermal experiments), velocity, and sample thickness. Analysis of experimental data makes it possible to understand the drying behavior of olive oil mill by-products and allows for improving and optimizing the drying processes in each type of dryer.

The present work provides a review of drying olive oil mill wastes. First, the different drying systems are described, and the advantages and disadvantages of each system are discussed. Next, the drying curves from a wider selection of drying experiments conducted by researchers are analyzed. Thereafter, the drying rate for each drying curve proposed is obtained, and the drying stages for olive oil mill wastes are discussed. An average drying rate value is obtained for each experiment, followed by analysis of the effective moisture diffusivity and activation energy values obtained in the literature. Several considerations are then made. Lastly, the main findings are presented.

DRYING SYSTEMS

Solar Dryers

Solar dryers consist mainly of a drying chamber, a fan, a chimney, and a flat plate collector. Solar energy is used for heating the airflow. Both natural convection and forced convection are used to dry the olive oil mill wastes. The use of solar dryers is economically viable; however, drying requires long periods of time, resulting in low drying rates. In some applications, solar dryers work closely with other auxiliary heat sources, such as electric resistors or infrared lamps.

Several works have reported on the drying of "alpeorujo" (Celma *et al.* 2007) and "orujo" (Montero *et al.* 2010; Montero *et al.* 2011) in solar dryers. The drying kinetics were studied for several heat transfer configurations: natural convection, forced convection, and hybrid mode (solar energy and heat generated by resistances). Experiments were carried out in a temperature range of 20 to 80 ºC and drying air velocities between 1 and 7 m/s. The sample thickness studied was between 6.2 and 40 mm.

Fluidized Bed Dryers

The fluidization phenomenon consists of passing hot air up through a bed of granular solids. Drying air velocity should be high enough to balance the pressure drop from the fluid drag force and the weight of the granular solid. Generally, fluidized bed dryers are formed in a drying chamber that consists of a cylindrical column, a centrifugal fan, and electrical resistances.

Drying kinetics have been studied for both "alpeorujo" (Liébanes *et al.* 2006) and "orujo" (Meziane 2011). Fluidized bed dryers are characterized by large contact areas between the olive oil mill waste phases. This results in high drying rates at relatively low drying air temperatures. Furthermore, sample homogeneity is the one of the major features in these systems; however, in the case of "alpeorujo" there is a drawback. The samples cannot be fluidized directly and have to be dried to reach a moisture content less than 50% (wet basis) due to their nature of having a high fluid content. "Orujo" has a more granular nature and is ideal for this drying system. Drying air temperatures used in the experiments were between 50 and 130 ºC and air velocities were in the range of 1 to 2.5 m/s. The thickness of the samples studied was between 41 and 63 mm.

Convective Dryers

Convective dryers have been the most popular drying system for the study of drying kinetics of olive oil mill wastes. Generally, these dryers consist of three fundamental elements: a blower, a drying chamber or drying tunnel, and air heaters or electrical resistances. The system operates by transporting hot air to the sample at different temperatures and velocities, providing fast and uniform drying.

Several convective dryers have been constructed for the drying of “alpeorujo” (Arjona *et al.* 1999; Krokida *et al.* 2002; Vega-Gálvez *et al.* 2010; Casanova-Peláez *et al.* 2015) and “orujo” (Doymaz *et al.* 2004; Akgun and Doymaz 2005; Gögüs and Maskan 2006) according to their respective requirements. These types of dryers have a good drying rate-to-energy cost ratio. Experiments conducted in convective dryers used drying air temperatures from 50 to 350 ºC and air velocities of 1.2 to 3 m/s. The samples dried had thicknesses between 4 and 72 mm.

Microwave-Convective Dryers

Microwave-convective dryers utilize microwave energy as the primary drying system and hot air convection dryers as the auxiliary drying system. Generally, this type of dryer consists of a microwave cavity where the sample is dried and ancillary equipment with air heaters and centrifugal fans. The main advantage of microwave-convective dryers is the application of two drying methods. High drying rates are obtained when the power of microwaves and drying air temperatures are high; nevertheless, the energy cost of microwave-convective systems is, on some occasions, very high.

Two works report on the drying kinetics of olive oil mill wastes. In the first work, “alpeorujo” was studied at low drying air temperatures, between 40 and 70 ºC, with a hot air velocity of 4 m/s and a sample thickness of 7 mm (Milczarek *et al.* 2011). In the second work, “orujo” was dried in a temperature range of 100 to 225 ºC for different sample thicknesses, between 6 and 14 mm (Gögüs and Maskan 2001).

Infrared Dryers

Infrared drying systems consist of a drying chamber with several halogen lamps. Drying is mainly accomplished in the sample by a diffusion phenomenon. Infrared wavelength radiation increases the sample temperature from the inner layer to outer layer. The infrared dryer features lower air flows that surround the sample and a reasonable drying rate-to-energy cost ratio; however, for high temperatures the energy cost can be high.

“Orujo” has previously been dried at temperatures between 80 and 140 ºC for a sample thickness of 7 mm (Ruiz Celma *et al.* 2008).

DRYING CURVES

Establishing drying curves is the first step in studying the drying kinetics of olive oil mills wastes. The experiments analyze the variation of mass, *i.e.* loss in moisture, with respect to time under isothermal conditions, with or without air velocity and a specific sample size. To plot drying curves a dimensionless variable is used, the moisture ratio, as calculated by Eq. 1,

$$MR = \frac{M_t - M_e}{M_0 - M_e} \quad (1)$$

where M_t is the moisture content at time t, M_0 is the initial moisture content, and M_e is the equilibrium moisture content, with all variables in dry basis. The vast majority of authors, however, express the moisture ratio as $MR = M_t/M_0$, because the equilibrium moisture content is small with respect to the other variables.

To estimate and predict the optimum drying conditions for future applications, the experimental data has been fitted, moisture ratio *vs.* drying time, using different thin-layer mathematical models. Non-linear regression analysis is usually used to fit the experimental data. Table 1 indicates the mathematical models proposed in the drying of "alpeorujo" and "orujo". Because drying is a complex physical phenomenon that is governed by heat and mass transfer mechanisms, various theoretical, empirical, and semi-empirical mathematical models have been studied for the drying of such by-products.

Table 1. Theoretical, Empirical, and Semi-Empirical Thin-Layer Mathematical Models in the Drying of Olive Oil Mill Wastes

Model Name	Equation	Reference
Lewis	$MR = \exp(-kt)$	(Lewis 1921)
Page	$MR = \exp(-kt^n)$	(Page 1949)
Modified Page	$MR = \exp(-(kt)^n)$	(Overhults *et al.* 1973)
Henderson and Pabis	$MR = a \cdot \exp(-kt)$	(Henderson and Pabis 1961)
Logarithmic	$MR = a \cdot \exp(-kt) + c$	(Akgun and Doymaz 2005)
Wang and Singh	$MR = 1 + at + bt^2$	(Wang and Singh 1978)
Two-Term	$MR = a \cdot \exp(-k_0 t) + c \cdot \exp(-k_1 t)$	(Noomhorm and Verma 1986)
Two-Term Exponential	$MR = a \cdot \exp(-kt) + (1-a) \cdot \exp(-kat)$	(Doymaz 2011)
Approach of Diffusion	$MR = a \cdot \exp(-kt) + (1-a) \cdot \exp(-kbt)$	(Yaldiz *et al.* 2001)
Modified Henderson and Pabis	$MR = a \cdot \exp(-kt) + b \cdot \exp(-k_2 t) + c \cdot \exp(-k_3 t)$	(Vega-Gálvez *et al.* 2010)
Midilli *et al.*	$MR = a \cdot \exp(-kt^n) + bt$	(Midilli *et al.* 2002)
n-Order	$MR = [1-(1-n)kt]^{\frac{1}{1-n}}$	(Chávez-Méndez *et al.* 1995)
Weibull	$MR = \exp\left(-\left(\frac{t}{\beta}\right)^n\right)$	(Corzo *et al.* 2008)
Simplied Fick's Diffusion	$MR = a \cdot \exp\left(-\frac{\delta t}{L^2}\right)$	(Diamante and Munro 1993)
Two term Gaussian	$MR = a \cdot \exp\left[-\left(\frac{t-b}{c}\right)^2\right] + d \cdot \exp\left[-\left(\frac{t-e}{f}\right)^2\right]$	(Gómez-de la Cruz *et al.* 2014a,b)

Table 2. List of Experiments in the Drying of Olive Oil Mill Wastes and a Comparison between Mathematical Models

Experiments and By-product	**Drying Conditions**			**Moisture Content (w.b) Initial-Final**	**Model proposed by authors**			**Two-Term Gaussian Model**		**Reference**
	Velocity (m/s)	**Sample thickness (mm)**	**Temperature (ºC)**		R^2	**RMSE**	**Model**	R^2	**RMSE**	
Solar Dryers										
Test 1- *Alpeorujo*	1	6.2	80	66%-7.5%	0.9985	0.0109	Midilli *et al.*	0.9989	0.0098	(Celma *et al.* 2007)
Test 2-*Alpeorujo*	1	6.2	40	66%-7.5%	0.9990	0.0081	Midilli *et al.*	0.9991	0.0079	(Celma *et al.* 2007)
Test 3-*Orujo*	4.5	30	32	55%-20%	-	-	-	0.9995	0.0086	(Montero *et al.* 2010)
Test 4-*Orujo*	-	30	20	55%-20%	-	-	-	0.9999	0.0033	(Montero *et al.* 2011)
Test 5-*Orujo*	-	30	3	55%-20%	-	-	-	0.9997	0.0070	(Montero *et al.* 2011)
Fluidized Bed Dryers										
Test 6-*Alpeorujo*	1.8 m/s	-	100	50%-0%	0.9999	-	n-order	1	0.0031	(Liébanes *et al.* 2006)
Test 7-*Alpeorujo*	1.8	-	130	50%-0%	0.9999	-	n-order	1	0.0044	(Liébanes *et al.* 2006)
Test 8-*Alpeorujo*	2.1	-	100	50%-0%	0.9999	-	n-order	1	0.0047	(Liébanes *et al.* 2006)
Test 9-*Alpeorujo*	2.5	-	100	50%-0%	0.9999	-	n-order	1	0.0073	(Liébanes *et al.* 2006)
Test 10-*Orujo*	1	41	50	48.6%-4.75%	0.9995	0.0067	Midilli *et al.*	0.9997	0.0057	(Meziane 2011)
Test 11-*Orujo*	1	41	70	48.6%-4.75%	0.9984	0.0002	Midilli et al.	0.9997	0.0001	(Meziane 2011)
Test 12-*Orujo*	1	63	50	48.6%-4.75%	0.9998	0.0032	Midilli et al.	0.9999	0.0023	(Meziane 2011)
Test 13-*Orujo*	1	63	70	48.6%-4.75%	0.9998	0.0037	Midilli et al.	0.9999	0.0021	(Meziane 2011)
Convective Dryers										
Test 14-*Orujo*	1.2	8	110	45%-5%	0.9998	0.0092		0.9999	0.0020	(Akgun and Doymaz 2005)

Test 15-*Orujo*	1.2	8	80	45%-5%	0.9992	0.0249		0.9999	0.0038	(Akgun and Doymaz 2005)
Test 16-*Orujo*	1.2	12	90	45%-5%	0.999	0.0057		0.9999	0.0045	(Doymaz *et al.* 2004)
Test 17-*Orujo*	1.2	4	80	45%-5%	-	-	-	0.9999	0.0025	(Doymaz *et al.* 2004)
Test 18-*Orujo*	1.2	6	80	36.7%-0%	-	-	-	0.9996	0.0085	(Gögüs and Maskan 2006)
Test 19-*Orujo*	1.5	9	70	36.7%-0%	-	-	-	0.9996	0.0074	(Gögüs and Maskan 2006)
Test 20-*Alpeorujo*	2	72	250	76%-0%	-	-	-	0.9998	0.0041	(Arjona *et al.* 1999)
Test 21-*Alpeorujo*	3	72	250	76%-0%	-	-	-	0.9998	0.0046	(Arjona *et al.* 1999)
Test 22-*Alpeorujo*	2	13	50	65.6%-27%	0.999	-	Herderson and Pabis	0.9996	0.0072	(Vega-Gálvez *et al.* 2010)
Test 23-*Alpeorujo*	2	13	70	65.6%-27%	0.999	-	Herderson and Pabis	0.9991	0.0111	(Vega-Gálvez *et al.* 2010)
Test 24-*Alpeorujo*	1.2	-	135	55%-0%	-	-	-	0.9996	0.0088	(Krokida *et al.* 2002)
Microwave-Convective Dryers										
Test 25-*Alpeorujo*	4	7	40	67.65%-0%	-	-	-	0.9998	0.0052	(Milczarek *et al.* 2011)
Test 26-*Alpeorujo*	4	7	60	67.65%-0%	-	0.0056	Midilli *et al.*	0.9999	0.0034	(Milczarek *et al.* 2011)
Test 27-*Orujo*	-	14	160	45%-4.7%	0.999	-	Lewis	0.9998	0.0097	(Gögüs and Maskan 2001)
Test 28-*Orujo*	-	14	225	45%-4.7%	0.983	-	Lewis	0.9999	0.0080	(Gögüs and Maskan 2001)
Infrared Dryers										
Test 29-*Orujo*	-	7	120	48%-8%	0.9999	0.0019	Midilli *et* al.	1	0.0016	(Ruiz Celma *et al.* 2008)
Test 30-*Orujo*	-	7	140	48%-8%	0.9999	0.0019	Midilli *et al.*	1	0.0016	(Ruiz Celma *et al.* 2008)

A broad cross-section of experiments carried out by researchers on the drying of these wastes for different drying systems has been examined in this review. In total, 30 drying tests have been reproduced. Drying curves have been newly fitted from a recent mathematical model proposed for the drying of “alpeorujo” (Gómez-de la Cruz *et al.* 2014a) and olive stone (Gómez-de la Cruz *et al.* 2014b), the two-term Gaussian model. Table 2 shows the characteristics of the drying experiments, the statistical criteria of the best mathematical models of fit presented by authors in their work, and the statistical criteria obtained with the two-term Gaussian model. To compare the quality of fit, the coefficient of determination (R^2) and root mean square error (RMSE) were used. High values of R^2 and low values of RMSE are needed to show a suitable mathematical model that explains the drying of these by-products. The two-term Gaussian model improved the statistical parameters of fit in all drying experiments as shown in Table 2.

The experiments presented a wide range of drying air temperatures, drying air velocities, and samples thicknesses. The initial moisture content also varied for the same by-product; “Orujo” had an initial moisture content between 36.7% and 55% (wet basis), and “alpeorujo” had values between 55% and 76% (wet basis). In tests 6 through 9, a pretreatment of centrifugation was carried out in the “alpeorujo” to reduce the moisture content to 50%. The final moisture content is shown as well. Figures 1 through 5 show the drying curves of the detailed experiments in Table 2 for each drying system. Furthermore, these curves were fitted to the Two-Term Gaussian model. As shown, this model seems to provide adequate approximation of experimental data of the drying curves.

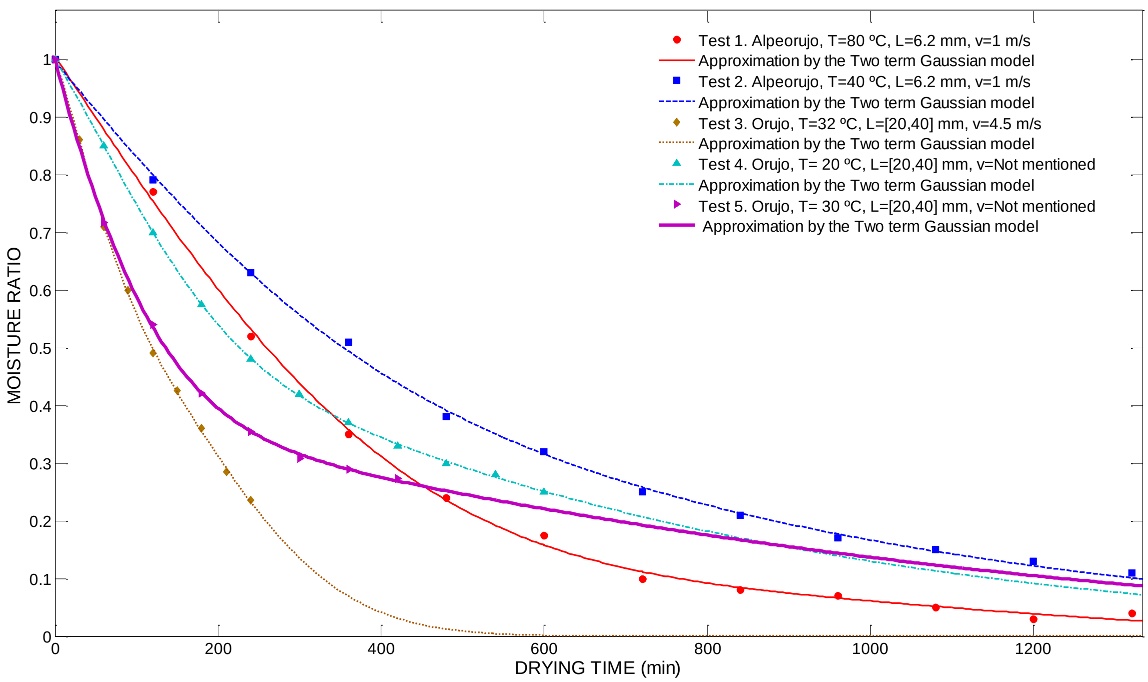

Fig. 1. Drying curves of the olive oil mill wastes dried with solar dryers. Experiments performed by various authors and approximation by the Two-Term Gaussian model

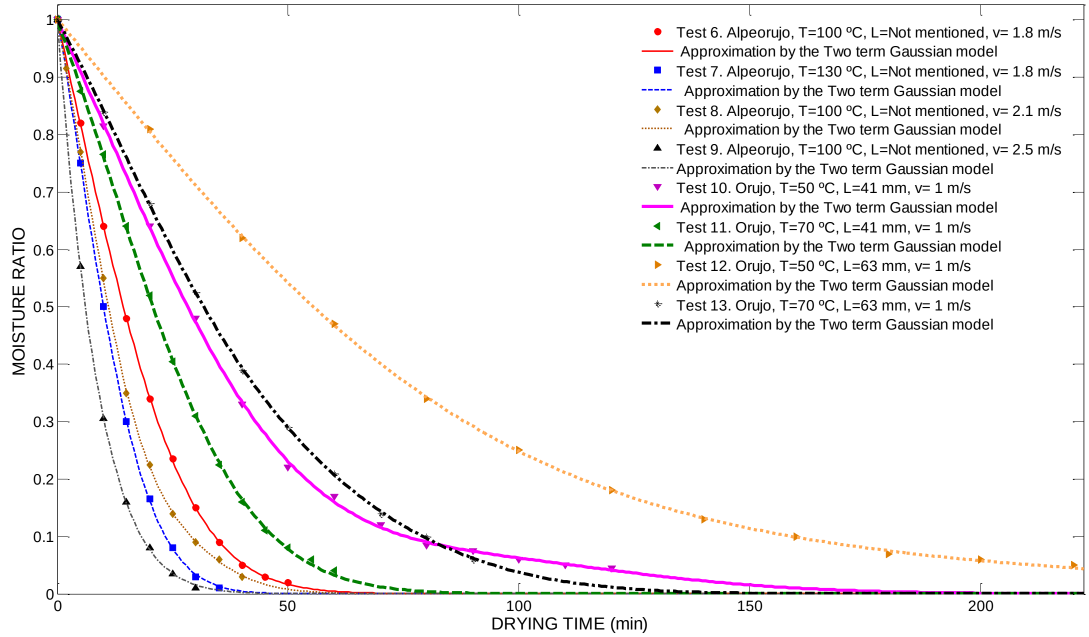

Fig. 2. Drying curves of the olive oil mill wastes dried with fluidized bed dryers. Experiments performed by various authors and approximation by the Two-Term Gaussian model

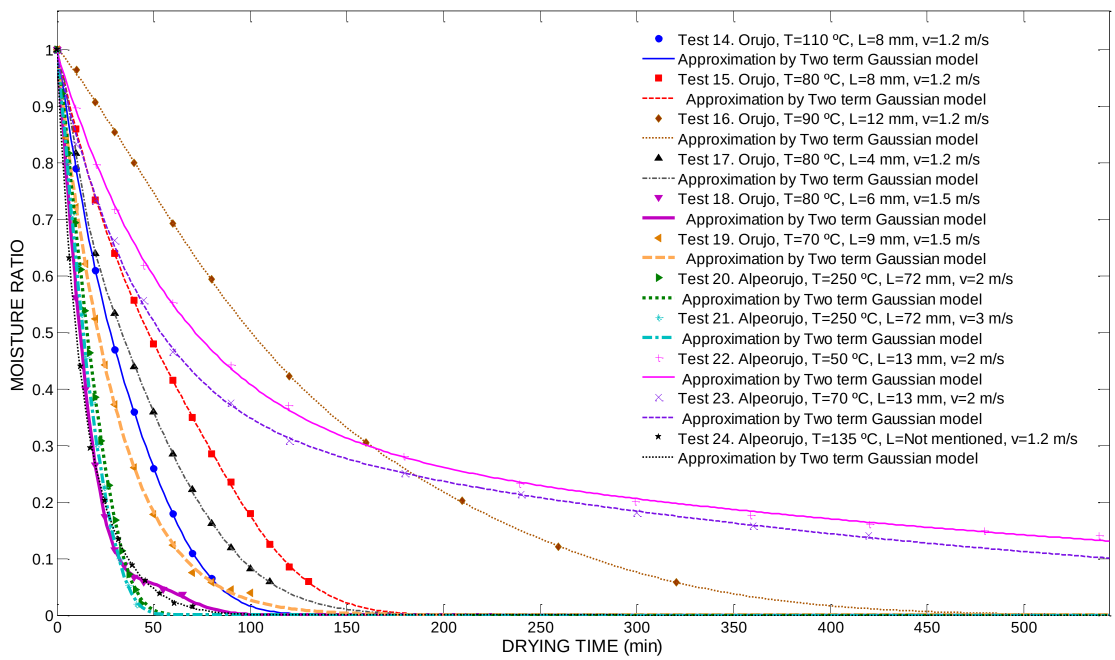

Fig. 3. Drying curves of the olive oil mill wastes dried with convective dryers. Experiments performed by various authors and approximation by the Two-Term Gaussian model

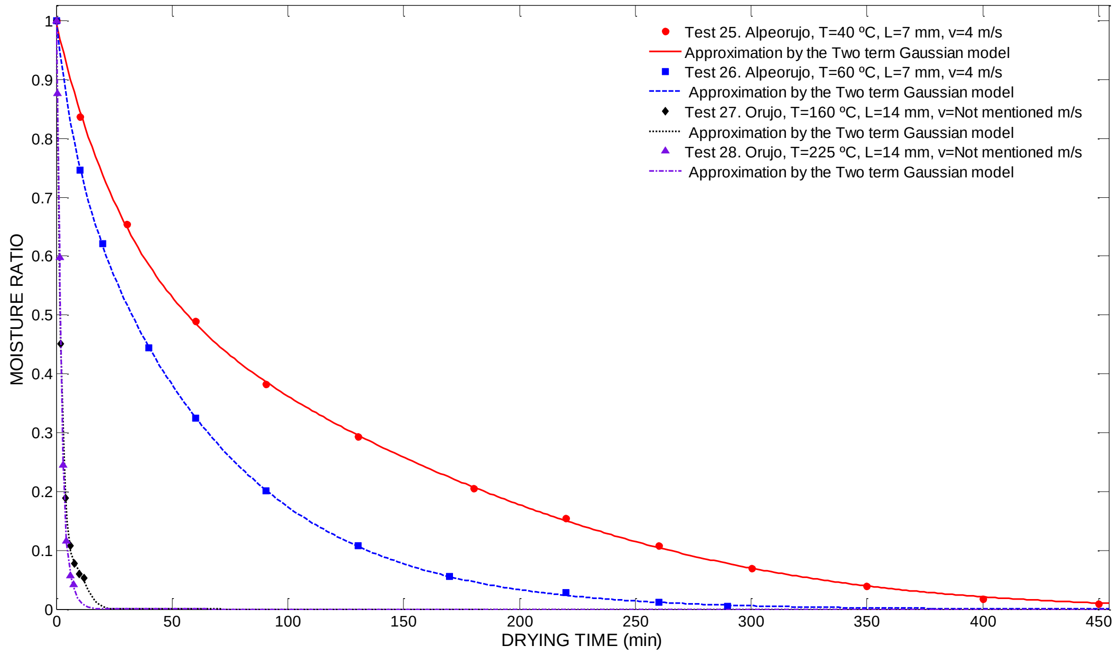

Fig. 4. Drying curves of the olive oil mill wastes dried with microwave-convective dryers. Experiments performed by various authors and approximation by the Two-Term Gaussian model

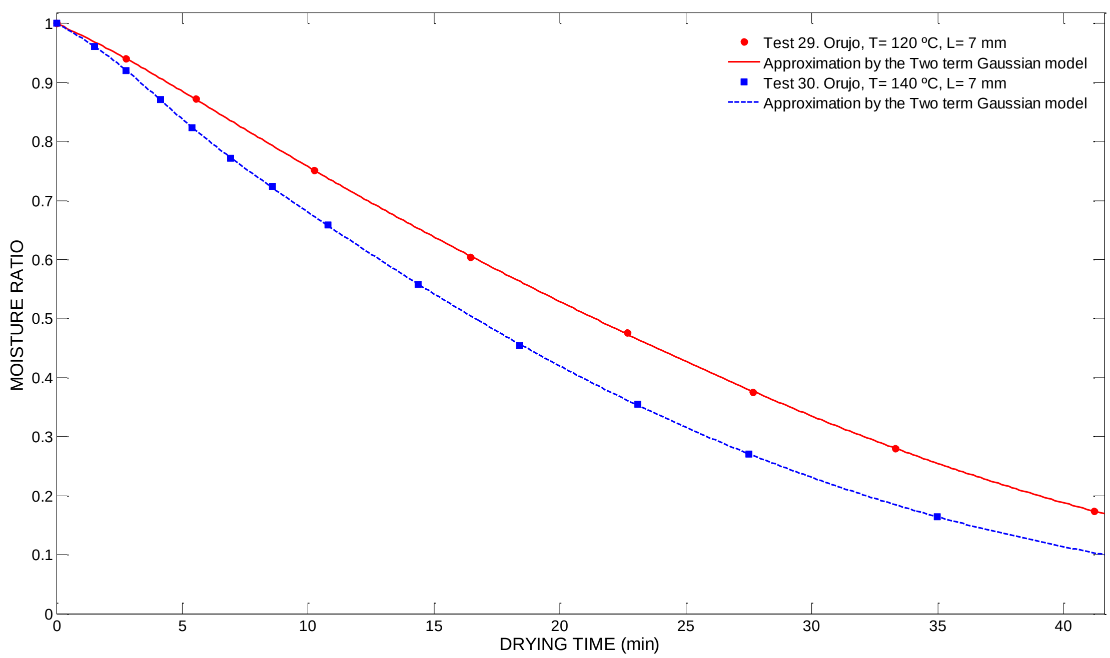

Fig. 5. Drying curves of the olive oil mill wastes dried with infrared dryers. Experiments performed by various authors and approximation by Two-Term Gaussian model

A general analysis of these curves shows that the main guiding force in the drying of these by-products is the drying air temperature; however, high drying air velocities substantially decreased the drying time, and sample size is another important factor. For similar conditions of temperature and velocity, it is vital to decrease the sample thickness to reduce the drying time. The figures indicate that drying depends on the moisture content of the by-product, as the moisture content increases, the drying time increases as well; thus, the drying times for "alpeorujo" are longer than those for "orujo", under similar drying conditions.

Figure 1 shows that drying times in the tests carried out in solar dryers were the longest (above 20 h) because of the low drying air temperature and drying air velocity (tests 1 and 2, "alpeorujo"). When the air velocity was increased, however, the drying time decreased to less than 7 h (test 3, "orujo"). A sample thickness between 20 and 40 mm increased the drying time, approximately one day (tests 4 and 5, "orujo").

The drying curves of the fluidized bed dryers are shown in Fig. 2. Similar initial moisture contents are found for both the "alpeorujo" and "orujo". As previously mentioned, the "alpeorujo" moisture content was reduced up to 50% (wet basis). In this type of dryer, low drying air temperatures, low air velocities, and several considerable sample thicknesses increased the drying time, between 1 and 4 h (tests 10 through 13, "orujo"), while drying air temperatures higher than 100 °C and air velocities between 1.8 and 2.5 m/s achieved a faster drying, between 25 and 50 min (tests 6 through 9, "alpeorujo").

In Fig. 3, the drying curves of the 11 experiments conducted in convective dryers are depicted. Tests performed at 250 °C resulted in very fast drying, below 1 h (tests 20 and 21, "alpeorujo"). A set of experiments at temperatures between 70 and 135 ºC had sample thicknesses between 4 and 9 mm, similar air velocities, and drying times in the range of 50 to 150 min (tests 14 and 15, tests 17 through 19, "orujo" and test 24, "alpeorujo"). When the sample thickness is greater than 10 mm, at drying air temperatures from 50 to 90 ºC and drying air velocities of 1.2 (test 16, "orujo") and 2 m/s (tests 22 and 23, "alpeorujo"), the drying time is longer than 5 h.

Figure 4 illustrates the four experiments performed in microwave-convective dryers. Drying times in this type of dryer were long when the drying air temperatures ranged from 40 to 60 ºC, for drying time greater than 150 min, even at a high velocity, 4 m/s, and a relatively small sample thickness, 7 mm (tests 25 and 26, "alpeorujo"); however, for greater thicknesses in the temperature range of 160 to 225 ºC, the drying experiments were completed in less than 15 min (tests 27 and 28, "orujo").

The drying curves of "orujo" carried out in an infrared dryer without drying air flow are plotted in Fig. 5. Test 29, at 140 ºC, presented a drying time less than test 30, which was performed at 120 ºC with the same sample thickness. In both cases, the drying time was greater than 1 hour.

DRYING RATES

The drying rate was calculated to better understand the drying behavior, drying stages, and the efficiency of the process. This value represents the amount of evaporated water per time unit. Experimentally, drying rate can be calculated from the moisture content variation with respect to time as shown by Eq. 2,

$$DR \approx -\frac{M_{t+\Delta t}-M_t}{\Delta t} \tag{2}$$

where DR is the drying rate ($kg_{water}/kg_{dry\ solid}\cdot s$), t is the drying time (s), $M_{t+\Delta t}$ represents the moisture content at time $t+\Delta t$ ($kg_{water}/kg_{dry\ solid}$), and M_t represents the moisture content at time t ($kg_{water}/kg_{dry\ solid}$). Drying rates can also be calculated from the derivative with respect to time of the thin-layer mathematical models proposed in Table 1 using Eq. 3:

$$x_v = -\frac{d(MR)}{dt} \tag{3}$$

In this equation x_v indicates that the drying rate is expressed as the reciprocal of unit of time; however, errors can be significant in the drying rate when the derivative function of these mathematical models is obtained, even for high values of R^2 and low values of RMSE. The two-term Gaussian model presents an excellent quality of fit: practically, R^2 values equal to 1 and RMSE values less than 0.01 are obtained. In this sense, the derivative of this model can be used to calculate the drying rate curves for olive oil mill wastes with a good approximation, as other works have confirmed (Gómez-de la Cruz *et al.* 2014a,b; Casanova-Peláez *et al.* 2015). In the vast majority of works, drying rate curves have been plotted as a function of moisture ratio instead of drying time. Figures 6 through 10 show the drying rate for each drying system: solar dryers, fluidized bed dryers, convective dryers, microwave-convective dryers, and infrared dryers, respectively.

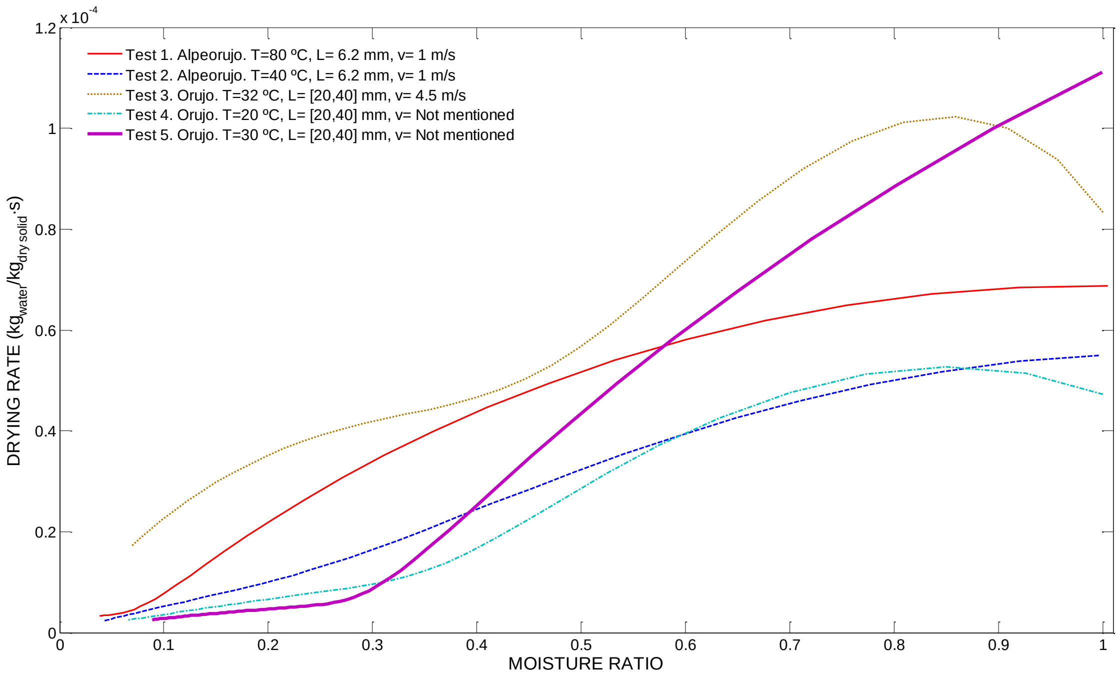

Fig. 6. Drying rate curves of the olive oil mill wastes dried with solar dryers calculated from the two-term Gaussian model

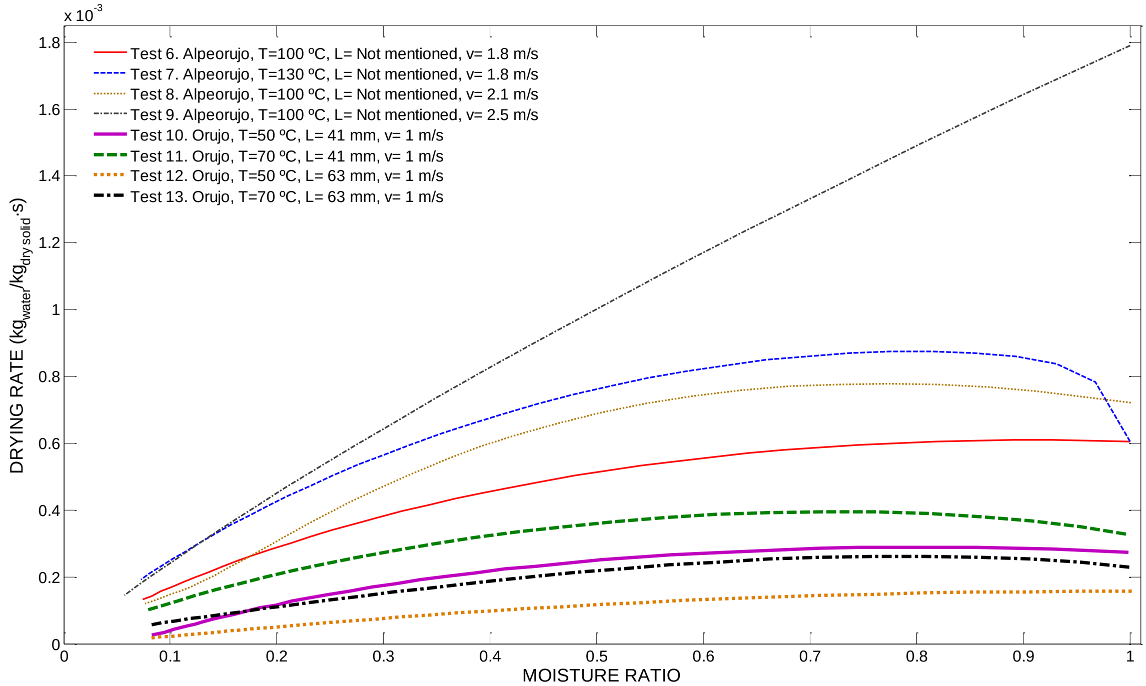

Fig. 7. Drying rate curves of the olive oil mill wastes dried with fluidized bed dryers calculated from the two-term Gaussian model

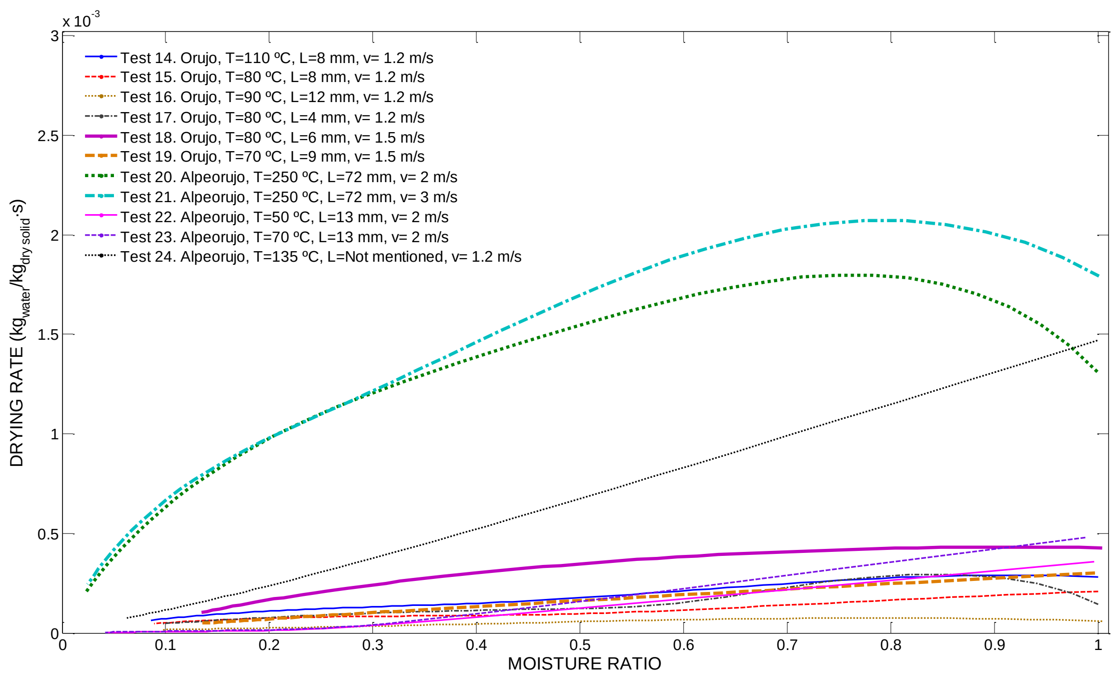

Fig. 8. Drying rate curves of the olive oil mill wastes dried with convective dryers calculated from the two-term Gaussian model

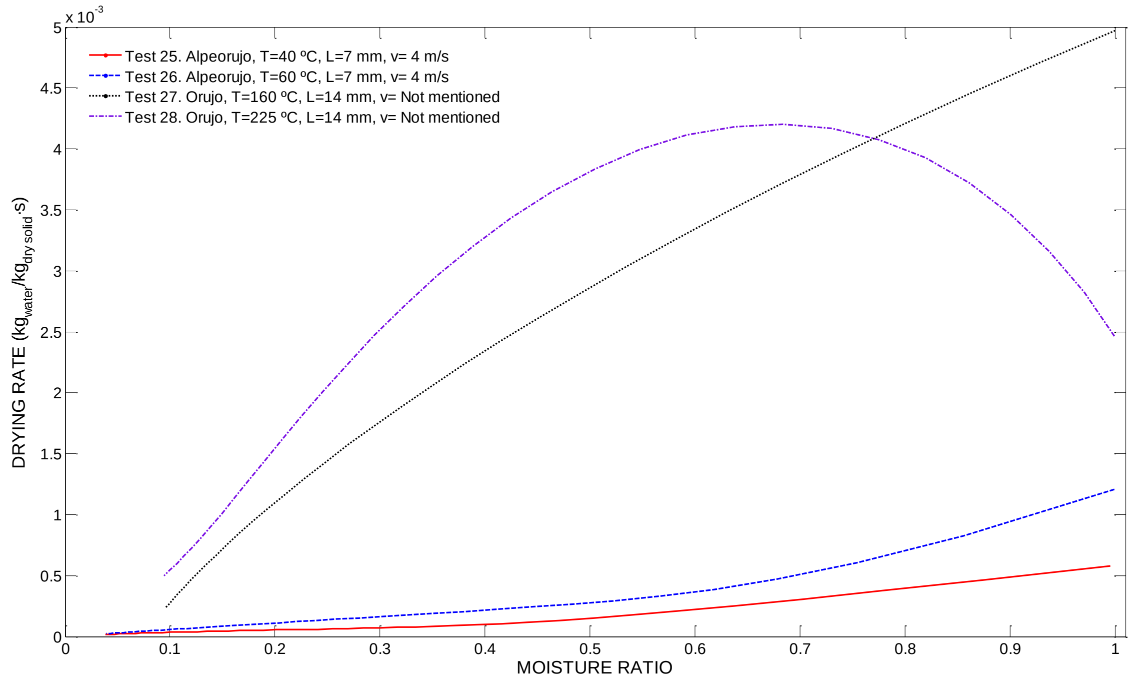

Fig. 9. Drying rate curves of the olive oil mill wastes dried with microwave-convective dryers calculated from the two-term Gaussian model

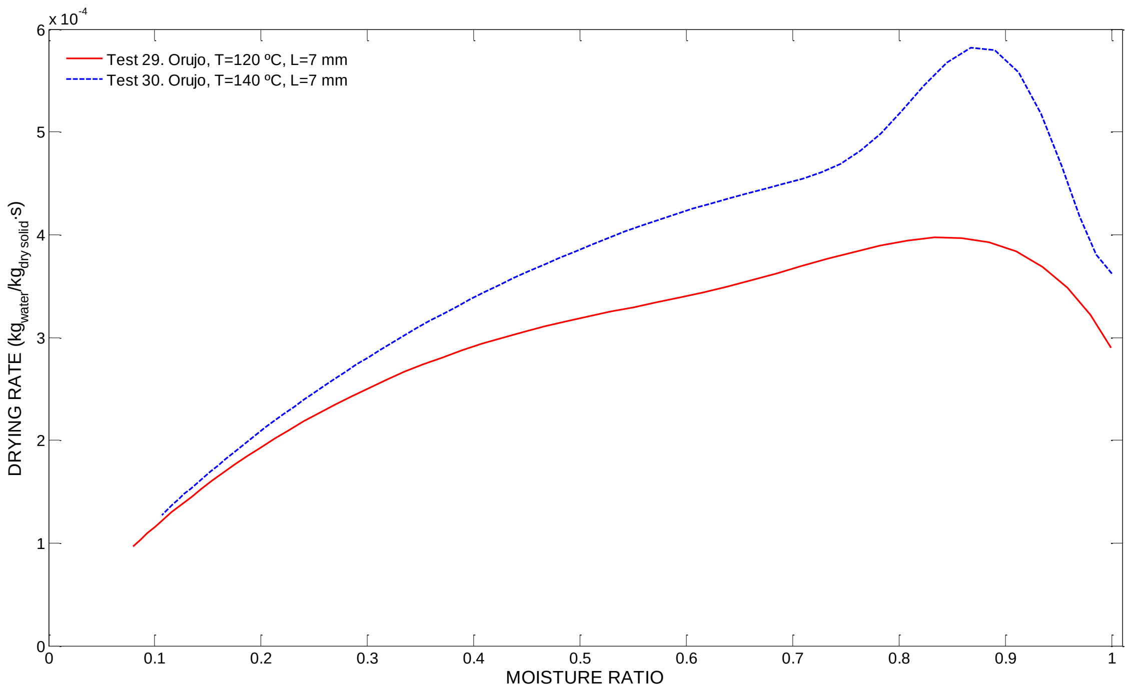

Fig. 10. Drying rate curves of the olive oil mill wastes dried with infrared dryers calculated from the two-term Gaussian model

These curves provide evidence regarding the mechanisms of heat and mass transport phenomena and allow for the study of the variables that intervened in the drying process of the olive oil mill by-products. Several works investigated the influence of variables such as drying air temperature, air velocity, sample thickness, and moisture content in the drying stages on the drying rate (Arjona *et al.* 1999; Ruiz Celma *et al.* 2008;

Meziane 2011; Gómez-de la Cruz *et al.* 2014b; Casanova-Peláez *et al.* 2015). There exist, however, other important factors that occur in the drying of olive oil mill wastes that are very difficult to investigate. Some of these factors are: physical and chemical phenomena, composition of the by-products, capillary diffusion, porosity, and heterogeneity of the particles.

By analyzing the drying rate curves, several drying stages can be differentiated: warm-up period, first falling rate period, and second falling rate period. Some researchers have defined a fourth stage at temperatures higher than 200 ºC, where water removal and volatile release take place simultaneously, mainly when the moisture content tends to equilibrium moisture content (tests 20 and 21, "alpeorujo" and test 28, "orujo") (Arjona *et al.* 1999). As shown by the curves, the drying of olive oil mill wastes does not manifest a constant rate period. These drying phases are exhibited too in other agricultural products such as apples (González-Fésler *et al.* 2008), pears (Guiné *et al.* 2007), potatoes (Wang *et al.* 2004), and pumpkins (Wang *et al.* 2007).

The warm-up period is characterized by rapid heating of the entire sample surface. In this phase, the total surface of the sample is saturated. All water on the surface is practically removed, but capillary suction is not sufficient enough to transport the water content from the inside to the outside of the sample. The drying rate increases as sample temperature increases. As the temperature approaches 100 ºC, water removal is produced by higher vapor pressures (ebullition), and the drying rate is raised until a maximum value is reached (Fig. 8: tests 20 and 21, Fig. 9: test 28, Fig. 10: tests 29 and 30). At temperatures less than 100 ºC, this period tends to be constant (Fig. 6: test 4, Fig. 7: tests 10 through 13, Fig. 8: tests 16 and 17). The maximum value of drying rate moves in a downward direction of the dimensionless moisture ratio. Nevertheless, when the sample is unsaturated, this period may disappear entirely, even at temperatures greater than 100 ºC (Fig. 7: test 9, Fig. 8: test 24, Fig. 9: test 27). This indicates that all drying processes occur in a falling rate period. Drying air velocity is important too in this stage, for the same conditions of temperature and sample thickness (Fig. 8: tests 20 and 21). Furthermore, for high drying air velocities, this period can be appreciated due to the fast drying by convection phenomenon, even at low temperatures (Fig. 6: test 3).

The first falling rate period is manifested at low temperatures when small quantities of water in the sample surface still exist (Fig. 6: tests 3 through 5, Fig. 9: tests 27 and 28). At higher temperatures (>100 ºC), continuous movement of water from within the porous medium to the sample surface is observed, which is mainly motivated by temperature (Fig. 10: tests 29 and 30). This period is called the *funicular state* (Strumillo and Kudra 1986), in which drying rate decreases as the moisture content at the surface decreases. The length of this stage may vary depending on the complex composition of the by-products. In some instances this period can hardly be differentiated, being very short or negligible (Figs. 7 and 8). At very high temperatures this period tends to disappear because all the water at the surface is rapidly eliminated in the warm-up period.

In the second falling rate period, liquid and vapor (<100 ºC, the *pendular state* (Strumillo and Kudra 1986)) and vapor (>100 ºC) migration is mainly produced by a diffusion phenomenon. In this stage the sample surface is completely dry and water is transported from inside the sample to the surface. The second falling rate period can be appreciated in all the reported drying tests.

Average Drying Rate

To measure the average speed of evaporated water in the different drying systems, the average drying rate was calculated. This value is calculated as the total quantity of water, eliminated from initial moisture content to equilibrium moisture content and divided by the drying time until the equilibrium moisture content is achieved. Mathematically, this value can be calculated by the integration of Eq. 3 utilizing the two-term Gaussian model between $t = 0$ and $t = t_{M_e}$ as:

$$\overline{x_v} = \frac{\int_{t=0}^{t=t_{M_e}} -\frac{d(MR)}{dt}dt}{t_{M_e}} \tag{4}$$

Average drying rate can be used to design and optimize future drying experiments and allow for proper estimation of the drying time. Furthermore, it can serve as a starting point to obtain the drying efficiency in each experiment. Table 3 specifies this value for each drying experiment, both “alpeorujo” and “orujo”.

The highest average drying rates corresponded to the drying experiments carried out in microwave-convective dryers for “orujo” at elevated drying air temperatures (tests 27 and 28). The combination of microwaves and high drying air temperatures is essential to ensure fast drying, but, as stated previously, a considerable amount of energy should be taken into account. High average drying rates were achieved in tests 20 and 21 performed in convective dryers for “alpeorujo”. In these experiments, high drying air velocities and temperatures were used. Fluidized bed dryers had good values for temperatures equal to or greater than 100 ºC, taking into account air velocities between 1.8 and 2.5 m/s. Even without air flows, experiments performed in infrared dryers presented good results when drying temperatures exceeded 100 ºC (tests 29 and 30); however, at low temperatures this value declined sharply for all drying systems, especially for solar dryers (tests 1 through 5), in which the average drying rates were the lowest of all experiments. Nevertheless, the great advantage of this dryer is the utilization of renewable energy.

EFFECTIVE MOISTURE DIFFUSIVITY AND ACTIVATION ENERGY

In the previous section it was confirmed that the falling rate period is the longest stage in the drying of olive oil mill wastes (Figs. 6 through 10). During this period, the main mass transfer mechanism is diffusion, which includes phenomena such as molecular diffusion, Knudsen diffusion, non-Fickian or stress driven diffusion, liquid diffusion through solid pores, and vapor diffusion in air-filled pores (Efremov and Kudra 2004). However, there are other mechanisms of mass transport such as capillarity, hydrodynamic flow, and evaporation-condensation that are manifested as well. In this sense, effective moisture diffusivity is usually defined as an overall mass transport property of moisture that includes the main mechanisms of mass transport, and it can explain the mass transfer process during the drying of these by-products (Vasić *et al.* 2014; Gómez-de la Cruz *et al.* 2015c). This variable is associated with the diffusion of mass into the medium during the changing of moisture content with the drying time and is obtained from Fick’s second law of diffusion (Eq. 5),

$$\frac{\partial(MR)}{\partial t} = D_{eff} \frac{\partial^2(MR)}{\partial x^2} \tag{5}$$

Table 3. Average Drying Rates of the Experiments Performed in the Drying of Olive Oil Mill Wastes

Experiments and By-products	Velocity (m/s)	Sample thickness (mm)	Temperature (ºC)	Initial moisture content (dry basis)	Drying time at M_e=0.08 (min)	Average drying rate $(min^{-1})\cdot 10^3$	Average drying rate $(g_{water} / kg_{dry\ matter}\cdot s)$	Reference
Solar Dryers								
Test 1-*Alpeorujo*	1	6.2	80	1.94	1178	0.819	0.026	(Celma *et al.* 2007)
Test 2-*Alpeorujo*	1	6.2	40	1.94	1860	0.514	0.017	(Celma *et al.* 2007)
Test 3-*Orujo*	4.5	30	32	1.22	364.5	2.564	0.052	(Montero *et al.* 2010)
Test 4-*Orujo*	-	30	20	1.22	1383	0.676	0.014	(Montero *et al.* 2011)
Test 5-*Orujo*	-	30	3	1.22	1526	0.612	0.012	(Montero *et al.* 2011)
Fluidized Bed Dryers								
Test 6-*Alpeorujo*	1.8 m/s	-	100	1	36.3	25.36	0.423	(Liébanes *et al.* 2006)
Test 7-*Alpeorujo*	1.8	-	130	1	24.9	37.01	0.617	(Liébanes *et al.* 2006)
Test 8-*Alpeorujo*	2.1	-	100	1	31.5	29.25	0.487	(Liébanes *et al.* 2006)
Test 9-*Alpeorujo*	2.5	-	100	1	19.7	46.6	0.777	(Liébanes *et al.* 2006)
Test 10-*Orujo*	1	41	50	0.94	83	11.02	0.177	(Meziane 2011)
Test 11-*Orujo*	1	41	70	0.94	49	18.62	0.293	(Meziane 2011)
Test 12-*Orujo*	1	63	50	0.94	171	5.364	0.085	(Meziane 2011)
Test 13-*Orujo*	1	63	70	0.94	83.1	11.01	0.174	(Meziane 2011)
Convective Dryers								
Test 14-*Orujo*	1.2	8	110	0.82	72.5	12.43	0.169	(Akgun and Doymaz 2005)
Test 15-*Orujo*	1.2	8	80	0.82	117.5	7.685	0.105	(Akgun and Doymaz 2005)
Test 16-*Orujo*	1.2	12	90	0.82	279.2	3.246	0.044	(Doymaz *et al.* 2004)
Test 17-*Orujo*	1.2	4	80	0.82	96	9.405	0.128	(Doymaz *et al.* 2004)
Test 18-*Orujo*	1.2	6	80	0.58	27.6	31.37	0.303	(Gögüs and Maskan 2006)
Test 19-*Orujo*	1.5	9	70	0.58	57.5	14.91	0.144	(Gögüs and Maskan 2006)

Test 20-*Alpeorujo*	2	72	250	3.17	44.4	21.93	1.157	(Arjona *et al.* 1999)
Test 21-*Alpeorujo*	3	72	250	3.17	40.9	23.85	1.259	(Arjona *et al.* 1999)
Test 22-*Alpeorujo*	2	13	50	1.91	1162	0.826	0.026	(Vega-Gálvez *et al.* 2010)
Test 23-*Alpeorujo*	2	13	70	1.91	877	1.076	0.034	(Vega-Gálvez *et al.* 2010)
Test 24-*Alpeorujo*	1.2	-	135	1.22	44.3	21.15	0.430	(Krokida *et al.* 2002)
Microwave-Convective Dryers								
Test 25-*Alpeorujo*	4	7	40	2.09	352.8	2.706	0.094	(Milczarek *et al.* 2011)
Test 26-*Alpeorujo*	4	7	60	2.09	191.2	4.997	0.174	(Milczarek *et al.* 2011)
Test 27-*Orujo*	-	14	160	0.82	6.2	144.4	1.969	(Gögüs and Maskan 2001)
Test 28-*Orujo*	-	14	225	0.82	4.9	185.7	2.532	(Gögüs and Maskan 2001)
Infrared Dryers								
Test 29-*Orujo*	-	7	120	0.92	50.9	17.94	0.276	(Ruiz Celma *et al.* 2008)
Test 30-*Orujo*	-	7	140	0.92	43.4	21.06	0.324	(Ruiz Celma *et al.* 2008)

where, D_{eff} is the effective moisture diffusivity (m^2/s), MR is the moisture ratio, x is the spatial dimension (m), and t is the drying time (s). For the one-dimensional mass transport in infinite slab geometry, the solution was proposed by Crank (1975) and is represented by Eq. 6 with the following assumptions: constant diffusion coefficients, constant temperature, and negligible shrinkage.

$$MR = \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\exp\left(-\frac{(2n+1)^2\pi^2 D_{eff}t}{L^2}\right) \quad (6)$$

The general solution is a complex formula with infinite terms; however, the vast majority of researchers take into account only the first term of the general expression considering that for sufficiently long drying times only the first term provides a good estimation of the solution (Di Scala and Crapiste 2008). Equation 6 can therefore be simplified into Eq. 7,

$$MR = \frac{8}{\pi^2}\exp\left(-\frac{\pi^2 D_{eff}t}{L^2}\right) \quad (7)$$

where L is the sample thickness (m). The effective moisture diffusivity is typically calculated by plotting the experimental drying data like *ln XR* (moisture ratio) versus *t* (time). Its plot is fitted from a linear function whose slope allows its value to be calculated as Eq. 8:

$$D_{eff} = -\frac{Slope\cdot L^2}{\pi^2} \quad (8)$$

Since effective moisture diffusivity depends on temperature, activation energy values have been obtained by researchers through an Arrhenius-type relationship Eq. 9,

$$D_{eff} = D_0 exp\left(-\frac{E_a}{RT}\right) \quad (9)$$

where D_0 is the pre-exponential factor (m^2/s), R is universal gas constant ($kJ\cdot mol^{-1}\cdot K^{-1}$), T is the absolute temperature (K), and E_a is the activation energy ($kJ\cdot mol^{-1}$).

Table 4 shows the values of effective moisture diffusivity and activation energy calculated by researchers for olive oil mill by-products. The results indicate that both effective moisture diffusivity and activation energy depend on sample thickness. The values of effective moisture diffusivity increase as the drying temperature and sample thickness increase. Activation energy values are augmented when the sample thickness is increased. It is logical to think that for high sample thicknesses, the energy required to produce the molecular movement should be higher.

It has been impossible to find a function that relates the effective moisture diffusivity of by-products with the drying temperature and sample thickness. Finding a relationship between activation energy and sample thickness was an unrealizable task as well; however, for studies of olive oil mill wastes of the same nature, the effective moisture diffusivity dependence with respect to drying temperature and sample thickness was manifested. Figures 11 and 12 show this relationship for studies carried out by Gögüs and Maskan (2006) and Meziane (2011) for “orujo”. A similar relationship was found in the drying of spent coffee grounds (Gómez-de la Cruz *et al*. 2015a). This indicates that

Table 4. Effective Moisture Diffusivity and Activation Energy Values for Different Experiments Conducted by Various Works

Type of Dryer and By-product	Velocity (m/s)	Sample Thickness (mm)	Temp (ºC)	Effective Moisture Diffusivity $(m^2/s)\cdot 10^{-9}$	Activation Energy (kJ/mol)	Reference
Solar Dryers *Alpeorujo*	1	6.2	20 40 80	0.22 0.45 0.7	15.77	(Celma *et al.* 2007)
Solar Dryers *Orujo*	-	30	20 30 40	0.54 0.76 1.41	38.64	(Montero *et al.* 2011)
Fluidized Bed Dryers *Orujo*	1	41	50 60 70 80	68 97 153 193	34.05	(Meziane 2011)
Fluidized Bed Dryers *Orujo*	1	52	50 60 70	82 133 183	36.84	(Meziane 2011)
Fluidized Bed Dryers *Orujo*	1	63	50, 60 70	94 148 215	38.10	(Meziane 2011)
Convective Dryers *Orujo*	1.2	8	50 60 70 80 90 100 110	3.38 4.85 6.25 7.15 7.89 8.82 11.3	17.97	(Akgun and Doymaz 2005)
Convective Dryers *Orujo*	1.2	12	80 90 100 110	0.49 0.62 0.79 0.99	26.71	(Doymaz *et al.* 2004)
Convective Dryers *Orujo*	1.5	6	60 70 80	184 303 342	25.4	(Gögüs and Maskan 2006)
Convective Dryers *Orujo*	1.5	9	60 70 80	217 298 367	25.7	(Gögüs and Maskan 2006)
Convective Dryers *Orujo*	1.5	12	60 70 80	218 322 394	29.2	(Gögüs and Maskan 2006)
Convective Dryers *Alpeorujo*	2	13	50 60 70 80 90	1.03 1.2 1.37 1.54 1.71	12.43	(Vega-Gálvez *et al.* 2010)
Microwave-Convective Dryers *Alpeorujo*	4	7	40 50 60 70	0.89 1.24 1.56 1.95	22.6	(Milczarek *et al.* 2011)
Infrared Dryers *Orujo*	-	7	80 100 120 140	5.96 7.94 13.9 15.9	21.3	(Ruiz Celma *et al.* 2008)

the values of these coefficients depend on several factors that are very difficult to investigate, mainly the variable nature and composition between the same types of wastes (particle size, the amount of oil contained in them, and the percentages of pulp, skin, olive stone, and organics compounds).

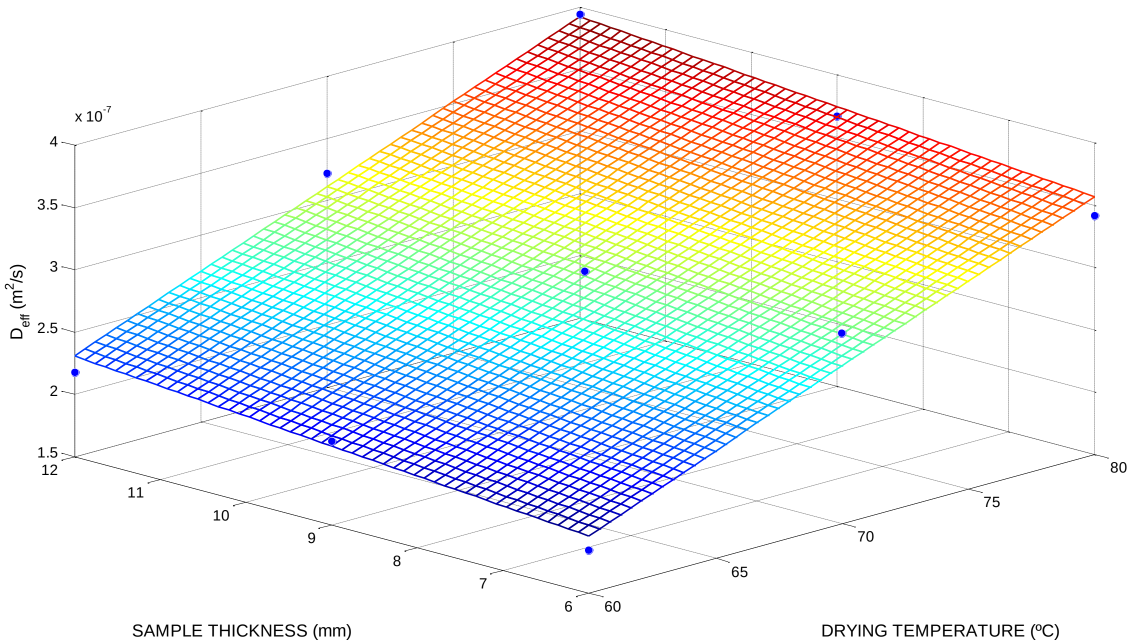

Fig. 11. Effective moisture diffusivity as a function of drying temperature and sample thickness. Experiments performed by Gogus and Maskan (2006)

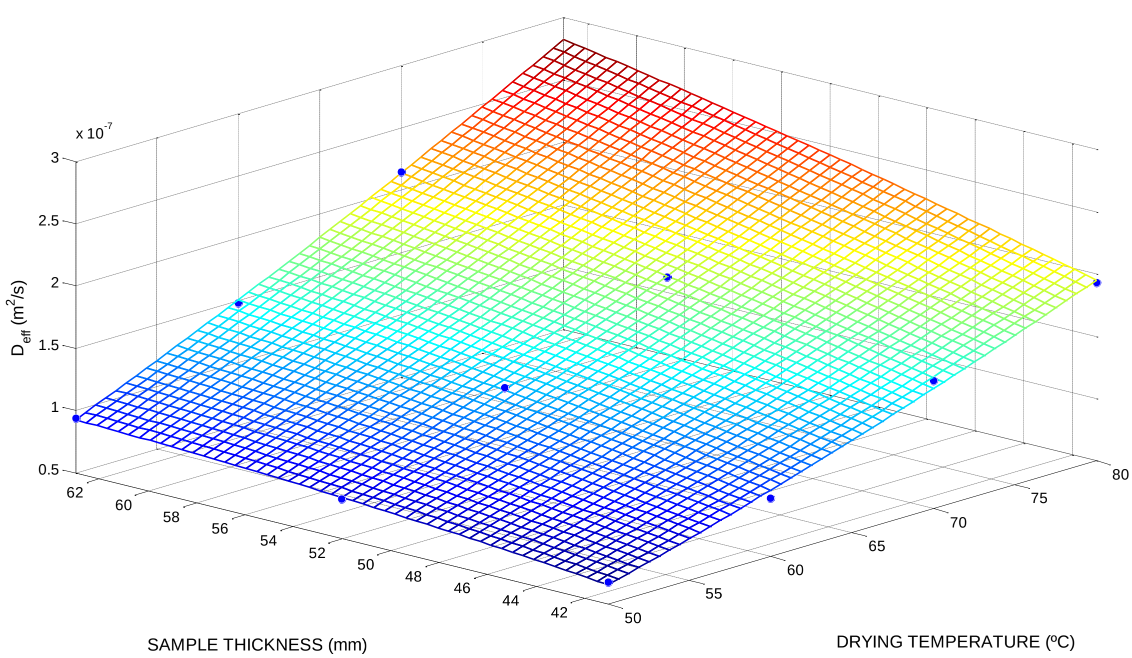

Fig. 12. Effective moisture diffusivity as a function of drying temperature and sample thickness. Experiments performed by Meziane (2011)

CONCLUSIONS

1. The drying kinetics of olive oil mill wastes has been reviewed in different drying systems for different drying conditions. It has been shown that drying depends mainly on three variables: drying temperature, air velocity, and sample thickness, with drying temperature being the main parameter to produce better results.
2. Drying curves of 30 experiments were fitted to the two-term Gaussian model, which presented the best results of fit in all drying tests proposed by various researchers. Its excellent quality of fit allowed for analytically obtaining the drying rate from its derivative function.
3. Drying rate curves permitted the analysis of different drying stages: the warm-up period, first falling rate period, and second falling rate period.
4. Microwave-convective dryers presented the highest average drying rates due to the combination of microwave energy and hot air convection. The lowest average drying rates were found in the solar dryers and were due to low temperatures. However, although solar drying is the slowest process, it is more sustainable and environmental friendly.
5. The drying features of olive oil mill wastes considered in this work showed that the vast majority of the drying process occurred during the falling rate period.
6. Effective moisture diffusivity and activation energy values were analyzed. There was no relationship between these variables and their independent variables. This evidenced that drying of olive oil mill wastes is a very complex physical process that depends heavily on aspects such as particle size, the amount of oil contained in them, and the percentages of pulp, skin, olive stone, and organics compounds.

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