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Obtaining of the drying rate of Alpeorujo for application on rotary dryers.

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***Abstract*— Alpeorujo is a by-product obtained in the process of olive oil extraction. This by-product is a thick sludge formed by vegetation water with organic compounds, pieces of pit, pulp, skin and about 5% olive oil content. Alpeorujo is dried from 60% to 8% moisture content (wet basis) for three main reasons: the elimination of highly polluting product due to its high biochemical oxygen demand (BOD), the extraction of olive oil contained in it (olive pomace oil) by the use of solvents and the obtaining of a biomass fuel called "orujillo" which has a net calorific value of 17500 kJ/kg. Drying kinetics is studied from three basic parameters: drying air temperature, drying air velocity and sample size. A design of experiments, central composite design, based on 15 tests was proposed. Isothermal drying tests were carried out in a drying tunnel and the moisture ratio-time curves were obtained. Drying curves have been fitted by the main mathematical models proposed by the researchers to date. A new mathematical model which presents the results of fits is proposed in this work, two terms Gaussian. Drying rate is one of the fundamental parameters to control the drying process in rotary dryers. It is calculated from the derivate of the moisture ratio-time curves. Effective diffusivity values were obtained in each test. Activation energy was 10230 J/mol for 30 mm height of the sample and 4 m/s velocity of the drying air.**

Keywords—Alpeorujo; drying rate; rotary dryers; drying kinetics; modeling.

I. INTRODUCTION

Alpeorujo is a waste by-product obtained in the olive oil mill industry. The "Two-phase system" separates the virgin olive oil from alpeorujo. This system generates a thick sludge with moisture content ranging from 60% to 70% (wet basis). Nearly 80% of olives is alpeorujo. This implies that the average annual of alpeorujo to treat in the European Union is 4.5 million tons. At present, practically all is dried in rotary dryers. These systems are crucial for ecological management of the olive oil in the world, since this by-product is a serious environmental problem due to its high biochemical oxygen demand (BOD).

The features of alpeorujo are very different from those of olive cake. Olive cake had around 40 % moisture content which was obtained in the old "Three-phase system". This system separated virgin olive oil, olive cake and alpechín. Olive cake was a by-product formed by a small quantity of pieces of pit and pulp of olives and a certain amount of olive oil. Alpechín, formed by vegetation water, sugars and organic compounds, constituted a hazardous pollutant which was stored in reservoirs constructed for this purpose. Furthermore, the "three-phase system" implied large amount of water in the process of olive oil extraction. For these reasons, the change of olive oil extraction system was carried out.

Rotary dryers were designed to dry olive cake. However, in the current process, the by-product obtained is alpeorujo which is formed by a mixture of alpechín and olive cake. This entails a high energy cost due to the elimination of high water content. Another significant drawback is the contribution of sugars which promote agglomerations during the drying process (increasing the particle size to dry). These agglomerations increase product adhesion to the trommel walls.

In addition to deleting an environmental problem, biomass product, "orujillo", and olive pomace oil are obtained in the drying of alpeorujo. To extract the olive oil contained in it, this sludge should be dried to values close to 8% moisture content. Then, it is mixed with a solvent, usually hexane, which facilitates its extraction. Initially, the extracted oil is refined, and later, it is combined with virgin oils. The production of olive pomace oil represents 10% of all olive oils and its price is slightly lower than virgin olive oil. When the olive pomace oil is extracted, a new biomass product is obtained, the "orujillo". It is a green energy source with a high calorific value to consider in Mediterranean countries where the olive oil production is important. This biofuel has special interest as fuel in boilers for residential heating [1], fuel consumption in the drying furnaces and fuel for cogeneration [2] and generation [3] plants (fig.1). Moreover, the cost per kg is low compared with other energy sources such as diesel and natural gas, being a tough competitor with respect to other biomass fuels. The energy released can be regarded as clean. The net calorific value is around 17500 kJ / kg and after drying, the moisture content ranges from 0.08 to 0.12 kg water/kg product.

Drying kinetics is analyzed from three fundamental parameters in rotary dryers: drying air temperature, drying air velocity and alpeorujo sample size inside of the blades of the trommel. The outlet temperature of the Alpeorujo rotary dryers ranges between 80 ºC and 120 ºC, while the inlet temperature may fluctuate between 400 ºC and 500 ºC. The maximum value of the drying gas velocity does not exceed 7 m/s, taking into account that changes along the trommel. Drying samples were carried out in a basket of 50 mm of diameter with different heights, from 10 mm to 50 mm. With these extreme values a design of experiments, central composite design, based on 15 tests was proposed. Isothermal drying tests were carried out in a drying tunnel and the moisture ratio-time curves were obtained. A new mathematical model is proposed in this work, two terms Gaussian. Drying curves have been fitted by the main mathematical models proposed by the researchers to date. Drying rate is calculated from the derivate of the moisture ratio-time curves. Finally, effective diffusivity values were obtained in each test. Activation energy was calculated for 30 mm height of the sample and 4 m/s velocity of the drying air as well.

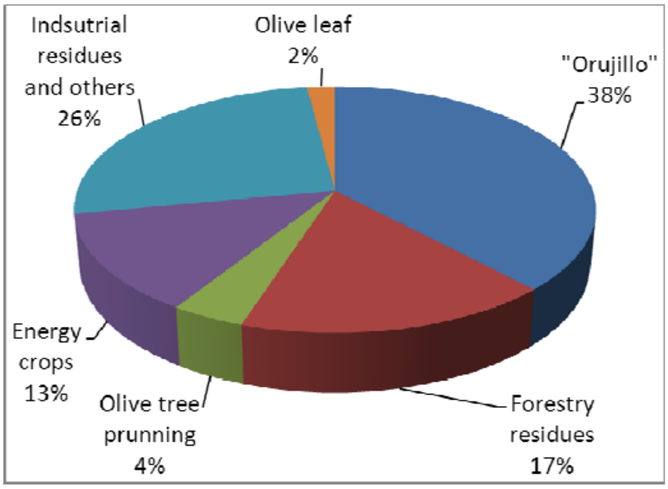

Fig. 1. Biofuels utilized in the generation of electrical power in Andalusia (Spain) [4].

II. Materials and Methods

A. Materials

Alpeorujo samples were kindly donated by several olive oil mills in the province of Jaén (Spain). The samples presented macroscopic heterogeneity due to different particle sizes. To find out the initial moisture content, the samples were dried in an oven (Memmert GmbH + Co.KG, SNB 167 Model 100, Germany) at 105 ºC for 24 hours. Drying samples were performed in triplicate. An average moisture content of 60 ± 5% (wet basis) was found. The same procedure was applied to obtain the equilibrium moisture content which was estimated at 8 ± 1% (wet basis).

B. Drying equipment

Drying tests were carried out in a drying tunnel (Fig. 2). The drying equipment is formed by: a blower, electric resistances (up to 45 kW of power) and a tunnel of 2 m of length with thermal insulation and 0.15 m of square section. To control the constant temperature in each test, a PID (Proportional-Integral-Differential) controller acted over the resistances, measuring the temperature using a PT 100 sensor. The sensor was positioned just before the point of drying of samples. The air velocity of the blower was controlled by a Variable Frequency Drive (VFD) connected to an electric AC motor. The samples were dried in a steel basket of 5 cm of diameter. The variation of mass was measured (every second) by a precision balance (Blauscal AH1200) with an error of ± 0.01 g. The precision balance was connected to personal computer by USB port. The information was stored in files and finally, was analyzed.

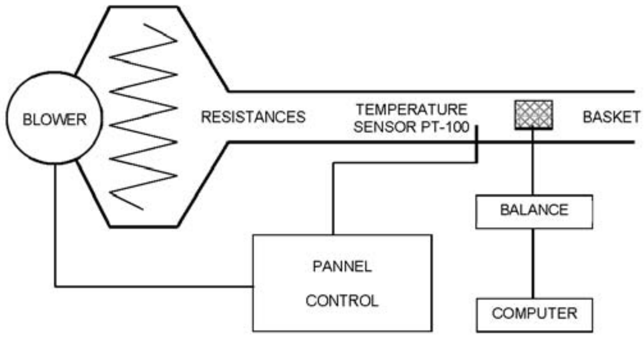

Fig. 2. Drying tunnel scheme.

C. Drying experiments

A central composite design for three variables [5]: drying air velocity, drying air temperature and sample size was carried out. This methodology is based on the extreme values, cubic values and central value for those variables (fig. 3). The extreme values considered in rotary dryers were:

- Drying air temperature: between 100ºC and 425ºC.
- Drying air velocity: between 1 m/s and 7 m / s.
- Sample thickness: between 10 mm and 50 mm.

In total, 15 drying experiment were proposed. Some works taking into account the granulometry of the particles. However, the particle size in rotary dryer is impossible to control. For this reason, only three parameters are studied in this work. Table I shows the parameters of drying for each test.

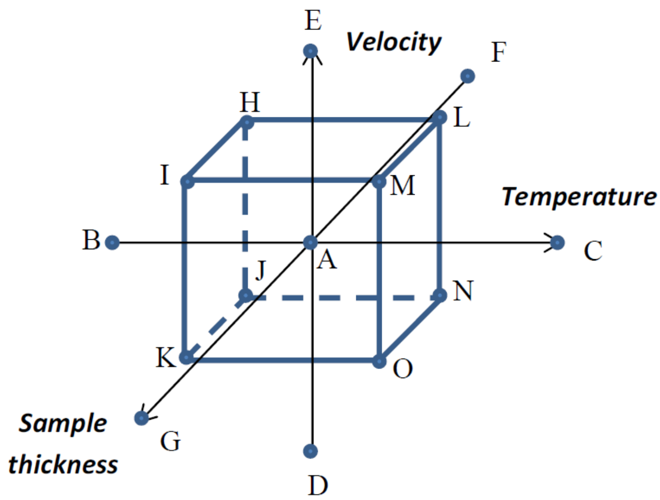

Fig. 3. Central composite design for three variables.

TABLE I. DRYING TEST CARRIED OUT IN THE DRYING TUNNEL.

TEST	T (ºC)	v (m/s)	L (mm)
A	263	4	30
B	100	4	30
C	425	4	30
D	263	1	30
E	263	7	30
F	263	4	10
G	263	4	50
H	181	5.5	20
I	181	5.5	40
J	181	2.5	20
K	181	2.5	40
L	344	5.5	20
M	344	5.5	40
N	344	2.5	20
O	344	2.5	40

III. RESULTS AND DISCUSSION

A. Analysis of drying curves

The variation of moisture ratio with respect to drying time during drying process for the alpeorujo samples in each test is shown in Fig. 4. As can be seen, the moisture ratio decreases when the drying process moves forward in time. The moisture ratio can be expressed as (1):

$$XR = \frac{X_t - X_e}{X_0 - X_e} \qquad (1)$$

where X_t is the moisture content at time t, X_0 is the initial moisture content and X_e is the equilibrium moisture content. Moisture ratio can be expressed like $XR = X_t / X_0$ when the equilibrium moisture content value, X_e, is small with respect to others variables.

Drying curves are usually fitted with empirical, semi-empirical or semi-theoretical mathematical functions. To estimate a mathematical function that represents faithfully the change in the moisture ratio with respect to drying time, drying curves were fitted with the main mathematical models in the drying of agricultural products. A good fit is essential to calculate the drying rate with excellent results, since the errors made in the approximation of drying curves become very highs when the derivate function is obtained. Ten mathematical models were used to approximate the drying curves by non-linear regression analysis. Table II indicates the names of the fit mathematical models and their equations. A new mathematical model is presented in this work, Two Term Gaussian.

The coefficient of determination, R^2, and the root mean square error, RMSE, were utilized to verify the quality of fit. Table III shows the values of R^2 and RMSE for the mathematical models presented in table II, applied to each of the fifteen drying curves. The mathematical models Approach of Diffusion and Midilli et al. obtained an average value of R^2 and RMSE of 0.9992 and 0.0067 and 0.9989 and 0.0082, respectively. Two Term Gaussian model is presented as the best fit model for drying curves of the alpeorujo with an average value of R^2 and RMSE of 0.9999 and 0.0019, respectively.

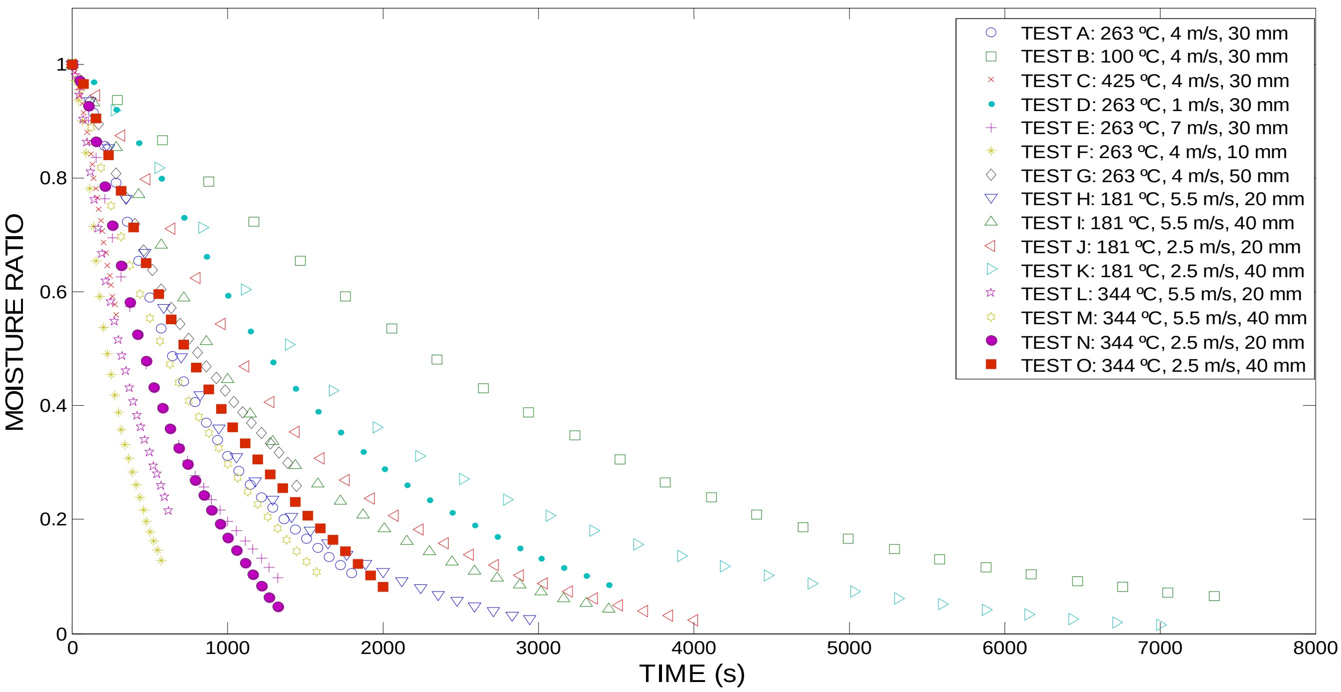

Fig. 4. Drying curves of the alpeorujo for each test.

TABLE II. MATHEMATICAL MODELS IN THE DRYING OF ALPEORUJO

Mathematical model	Equation	References
Lewis	$XR = \exp(-\mathrm{kt})$	[6]
Page	$XR = \exp(-\mathrm{kt}^{n})$	[7]
Modified Page	$XR = \exp(-(\mathrm{kt})^{n})$	[8]
Henderson and Pabis	$XR = a\cdot\exp(-\mathrm{kt})$	[9]
Logarithmic	$XR = a\cdot\exp(-\mathrm{kt})+\mathrm{c}$	[10]
Wang and Singh	$XR = 1+at+bt^{2}$	[11]
Two term	$XR = a\cdot\exp(-\mathrm{k_0 t})+\mathrm{c}\cdot\exp(-\mathrm{k_1 t})$	[12]
Approach of Diffusion	$XR = a\cdot\exp(-\mathrm{kt})+(1-\mathrm{a})\cdot\exp(-\mathrm{kbt})$	[13]
Midilli et al.	$XR = \exp(-\mathrm{kt}^{n})+\mathrm{bt}$	[14]
Two term Gaussian	$XR = a\cdot\exp\left(-\left((t-b)/c\right)^{2}\right)+d\cdot\exp\left(-\left((t-e)/\mathrm{f}\right)^{2}\right)$	Present work

TABLE III. STATISTICAL RESULTS OBTAINED, R^2 Y RMSE, FROM DIFFERENT THIN-LAYER DRYING MODELS

	MATHEMATICAL MODEL									
	Lewis		**Page**		**Modified Page**		**Henderson and Pabis**		**Logarithmic**	
TEST	**R^2**	**RMSE**	**R^2**	**RMSE**	**R^2**	**RMSE**	**R^2**	**RMSE**	**R^2**	**RMSE**
A	0,9866	0,0313	0,9977	0,0131	0,9977	0,0131	0,9966	0,0158	0,9974	0,0139
B	0,9901	0,0275	0,9999	0,0028	0,9999	0,0028	0,9961	0,0173	0,9987	0,0101
C	0,9547	0,0292	0,9998	0,0018	0,9998	0,0018	0,9832	0,0178	0,9961	0,0086
D	0,9762	0,0440	0,9985	0,0109	0,9985	0,0109	0,9923	0,0250	0,9965	0,0168
E	0,9923	0,0233	0,9984	0,0108	0,9984	0,0108	0,9981	0,0116	0,9984	0,0105
F	0,9766	0,0418	0,9965	0,0162	0,9965	0,0162	0,9928	0,0232	0,9953	0,0188
G	0,9947	0,0157	0,9974	0,0110	0,9974	0,0110	0,9982	0,0090	0,9983	0,0090
H	0,9883	0,0308	0,9979	0,0129	0,9979	0,0129	0,9964	0,0171	0,9970	0,0155
I	0,9908	0,0268	0,9980	0,0124	0,9980	0,0124	0,9972	0,0147	0,9976	0,0138
J	0,9820	0,0391	0,9989	0,0099	0,9989	0,0099	0,9937	0,0232	0,9967	0,0168
K	0,9934	0,0224	0,9979	0,0125	0,9979	0,0125	0,9980	0,0124	0,9981	0,0119
L	0,9856	0,0288	0,9978	0,0114	0,9978	0,0114	0,9964	0,0145	0,9977	0,0116
M	0,9951	0,0178	0,9987	0,0092	0,9987	0,0092	0,9978	0,0120	0,9994	0,0061
N	0,9755	0,0450	0,9978	0,0135	0,9978	0,0135	0,9914	0,0267	0,9977	0,0139
O	0,9880	0,0293	0,9988	0,0093	0,9988	0,0093	0,9958	0,0173	0,9993	0,0072
	MATHEMATICAL MODEL									
	Wang and Singh		**Two Term**		**Approach of Diffusion**		**Midilli et al.**		**Two Term Gaussian**	
	R^2	**RMSE**	**R^2**	**RMSE**	**R^2**	**RMSE**	**R^2**	**RMSE**	**R^2**	**RMSE**
A	0,9975	0,0135	0,9975	0,0135	0,9996	0,0054	0,9991	0,0083	**1,0000**	**0,0011**
B	0,9993	0,0073	0,9999	0,0024	0,9999	0,0025	0,9999	0,0023	**0,9999**	**0,0022**
C	0,9983	0,0056	0,9999	0,0014	0,9999	0,0014	0,9999	0,0014	**0,9999**	**0,0015**
D	0,9965	0,0168	0,9968	0,0161	0,9996	0,0054	0,9992	0,0082	**1,0000**	**0,0012**
E	0,9972	0,0142	0,9985	0,0103	0,9996	0,0054	0,9991	0,0078	**0,9999**	**0,0024**
F	0,9963	0,0167	0,9955	0,0184	0,9991	0,0084	0,9981	0,0119	**0,9999**	**0,0026**
G	0,9982	0,0092	0,9981	0,0094	0,9988	0,0075	0,9988	0,0076	**0,9998**	**0,0032**
H	0,9904	0,0278	0,9973	0,0148	0,9995	0,0064	0,9989	0,0096	**1,0000**	**0,0017**
I	0,9925	0,0242	0,9977	0,0133	0,9995	0,0064	0,9990	0,0090	**1,0000**	**0,0014**
J	0,9951	0,0205	0,9971	0,0157	0,9996	0,0057	0,9992	0,0084	**1,0000**	**0,0010**
K	0,9837	0,0352	0,9982	0,0118	0,9993	0,0075	0,9986	0,0103	**1,0000**	**0,0012**
L	0,9979	0,0109	0,9809	0,0333	0,9993	0,0062	0,9988	0,0084	**0,9999**	**0,0027**
M	0,9975	0,0128	0,9998	0,0038	0,9987	0,0094	0,9987	0,0091	**0,9999**	**0,0023**
N	0,9969	0,0160	0,9977	0,0137	0,9979	0,0132	0,9982	0,0123	**0,9999**	**0,0026**
O	0,9981	0,0116	0,9993	0,0070	0,9985	0,0104	0,9989	0,0088	**0,9999**	**0,0022**

B. *Analysis of drying rates*

This value indicates the moisture content variation with respect to time or the amount of evaporated water per time unit. The drying rate is a variable very important to optimize and control the drying process in rotary dryers. The drying rate of alpeorujo can be expressed as (2):

$$x_v = -\frac{d(\mathrm{XR})}{dt} \approx -\frac{XR_{t+\Delta t} - XR_t}{\Delta t} \qquad (2)$$

where $XR_{t+\Delta t}$ and XR_t represent the moisture content at time $t+\Delta t$ and the moisture content at time t, respectively, and t is the drying time (s). The negative sign was included to indicate the drying rate with positive values.

Drying rates were experimentally obtained from the drying curves. Fig. 5 shows the experimental drying rate with respect to moisture ratio. As can be seen, the drying rates exhibit the drying stages proposed by some researchers [15-16]. These stages are: warming-up period, first falling rate period and second falling rate period. There is no constant rate period. The drying of alpeorujo is a complex physic process which depends on diffusion and convection phenomena. The drying rate curves can be fitted from the derivate with respect to time of the models of the table II. Although all mathetical models of drying curves presented good results of fit (table III), in the vast majority of models, when their derivate functions were obtained, the errors made were considerable.

Drying rate can be calculated from the derivate of Two term Gaussian model with an error made very small. This analytical expression can calculate the drying rate for any instant of time or moisture ratio. Furthermore, this mathematical model faithfully reflects the different stages in the drying of alpeorujo.

As expected from fig. 5, the drying rate increases with increase in drying air temperature and drying air velocity. However, the drying rate decreases as the sample thickness increases. This implies that these variables should be especially taking into account in the optimization and control in rotary dryers.

C. *Effective Diffusivity and Activation energy*

Although the warming-up period is governed by convection phenomenon, almost all the drying process occurs in the falling rate period. This period is mainly governed by diffusion phenomenon. In this sense, effective diffusivity values can be obtained for each test.

For that, the partial differential equation, Fick's second law of diffusion (3), should be solved. The solution proposed by Crank [17] for the one-dimensional mass transport in infinite slab geometry, in spherical coordinates, is considered (4). However, some suppositions should be done: negligible shrinkage, migration by diffusion and constant temperature and diffusion coefficients.

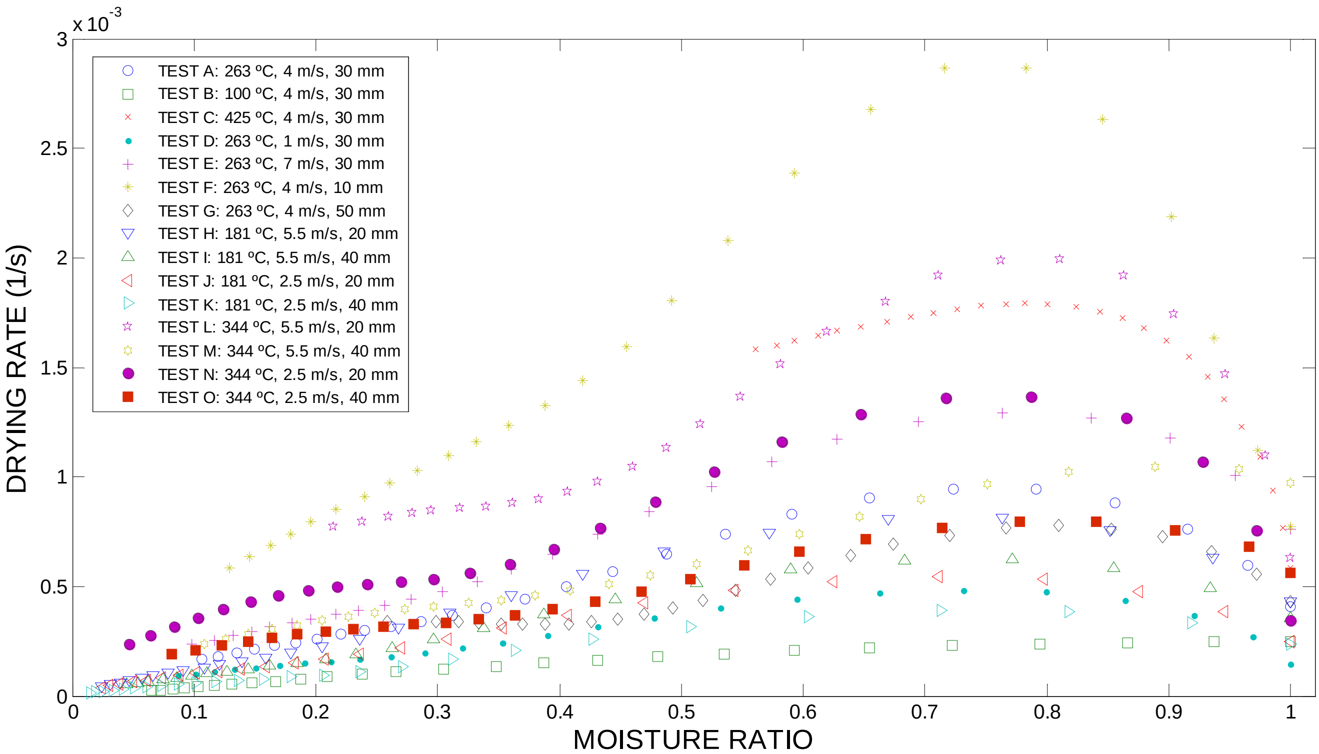

Fig. 5. Drying rate versus moisture ratio of the alperoujo for each test.

$$\frac{\partial(\mathrm{XR})}{\partial t} = D_{eff}\frac{\partial^2(\mathrm{XR})}{\partial x^2} \tag{3}$$

$$XR = \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\exp\left(-\frac{(2n+1)^2\pi^2 D_{eff}t}{L^2}\right) \tag{4}$$

where D_{eff} is the effective diffusivity (m^2/s), L is thickness of slab (m) and t is the drying time (s). Equation (4) can be simplified to only the first term of the series when the drying times are long (5):

$$XR = \frac{8}{\pi^2}\exp\left(-\frac{\pi^2 D_{eff}t}{L^2}\right) \tag{5}$$

This equation can be represented graphically applying the logarithm on both sides. From the slope of the curves *ln (XR)* versus time can be calculated the effective diffusivity as (6):

$$D_{eff} = \frac{slope \cdot L^2}{\pi^2} \tag{6}$$

The values of the effective diffusivity in the alpeorujo for each test are shown in table 4. The coefficient of determination in the approximation of the linear function of the curves *ln (XR)* versus time is shown as well.

TABLE IV. EFFECTIVE DIFFUSIVITY VALUES FOR EACH TEST

TEST	Effective Diffusivity (m^2/s)	R^2
A	$1.29\cdot10^{-7}$	0.9956
B	$4.47\cdot10^{-8}$	0.9823
C	$2.05\cdot10^{-7}$	0.9841
D	$7.68\cdot10^{-7}$	0.9828
E	$1.79\cdot10^{-7}$	0.9923
F	$4.09\cdot10^{-8}$	0.9926
G	$2.44\cdot10^{-7}$	0.9974
H	$6.91\cdot10^{-8}$	0.9368
I	$1.97\cdot10^{-7}$	0.9574
J	$4.83\cdot10^{-8}$	0.9271
K	$1.23\cdot10^{-7}$	0.9476
L	$1.10\cdot10^{-7}$	0.9949
M	$2.44\cdot10^{-7}$	0.9844
N	$1.06\cdot10^{-7}$	0.9178
O	$2.15\cdot10^{-7}$	0.9737

Effective diffusivity depends on the temperature and can be calculated from an Arrhenius type relationship (7):

$$D_{eff} = D_0\exp\left(-\frac{E_a}{RT}\right) \tag{7}$$

where D_0 is the pre-exponential factor (m^2/s), R is the universal gas constant ($J\cdot mol^{-1}\cdot K^{-1}$), T is the absolute temperature (K) and E_a is the activation energy ($J\cdot mol^{-1}$). The activation energy was found out for a velocity of 4 m/s and a sample thickness of 30 mm. Fig. 6 shows the function *ln* D_{eff} versus 1/*T*. As can be seen, the ln D_{eff} decreases linearly with the reciprocal of the temperature. The activation energy was found out in 10.23 kJ / mol.

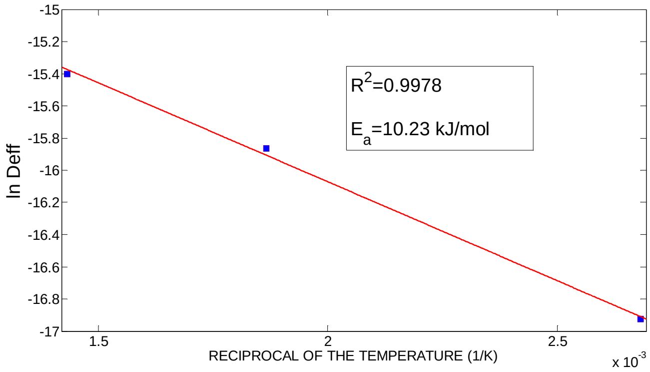

Fig. 6. Arrhenius-type relationship between effective diffusivity logarithmic and the reciprocal of absolute temperature for tests A, B and C.

Similar results can be found in the literature for olive cake. Meziane [18], with a drying air velocity in his experiments of 1 m/s, obtained values of effective diffusivity between $0.68\cdot10^{-7}$ to $2.15\cdot10^{-7}$ m^2/s for tests carried out at temperatures and sample thicknesses between 50 ºC to 80 ºC and 41, 52 and 63 mm, respectively, and activation energy values of 34.05, 36.84, 38.1 kJ/mol for each sample thickness. Gögüs and Maskan [19] calculated, for temperature between 60 ºC to 80 ºC, sample thicknesses of 6, 9 and 12 mm and velocity of 1.5 m/s, effective diffusivity values between $1.84\cdot10^{-7}$ to $3.94\cdot10^{-7}$ m^2/s and activation energy values of 25.4, 25.7, 29.2 kJ/mol for each sample thickness, respectively. Akgun and Doymaz [10] found out values between $4.95\cdot10^{-10}$ to $1.42\cdot10^{-9}$ m^2/s for temperatures ranging from 50-110 ºC, a sample thickness of 8 mm and a velocity of 1.2 m/s. The activation energy in this research was 17.97 kJ / mol.

IV. CONCLUSION

A design of experiments was carried out taking into account the most important conditions in the drying of alpeorujo in rotary dryers. The drying kinetics was analyzed. Drying curves were fitted with the main mathematical model in the drying of agricultural products. Drying rates were calculated. A new empirical mathematical model presented the best results of fit, both in the drying curves and the drying rates. Effective diffusivity values were obtained and an activation energy value was found. Results obtained in this work can serve as a starting point for calculating the drying rate in any state in the drying of alpeorujo in rotary dryers from techniques like neural networks.

NOMENCLATURE

a,b,c,d,e,f,n	Coefficients of the mathematical models
k, k_0, k_1	Constants of the mathematical models (s^{-1})
D_{eff}	Effective diffusivity (m^2/s)
D_0	Pre-exponential factor of the Arrhenius equation (m^2/s)
E_a	Activation energy (kJ/mol)
L	Thickness of the slab (m)
R	Universal gas constant ($kJ \cdot mol^{-1} \cdot K^{-1}$)
R^2	Coefficient of determination
RMSE	Root mean square error
t	Time (s)
T	Temperature (ºC, K)
v	Velocity ($m \cdot s^{-1}$)
X_e	Equilibrium moisture content (kg moisture/kg dry matter)
X_0	Initial moisture content (kg moisture/kg dry matter)
X_t	Moisture content at time t (kg moisture/kg dry matter)
XR	Dimensionless moisture ratio
x_v	Drying rate (kg moisture/(kg dry matter · s))

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