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**ESTUDIO Y ANÁLISIS DE LA CINÉTICA DE**  
**SECANO DE SUBPRODUCTOS DE**  
**ALMAZARA PARA SU APLICACIÓN A**  
**SECADEROS ROTATIVOS**

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**TESIS DOCTORAL**

***Estudio y Análisis de la cinética de secado de subproductos de almazara  
para su aplicación a secaderos rotativos***

Por

**D. Francisco Javier Gómez de la Cruz**

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para la obtención del

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Directores de Tesis

**Dr. D. Fernando A. Cruz Peragón**

**Dr. D. José M. Palomar Carnicero**

**Jaén, Mayo de 2015**



D. Fernando A. Cruz Peragón, Doctor Ingeniero Industrial y Profesor Titular de Universidad del Departamento de Ingeniería Mecánica y Minera de la Escuela Politécnica Superior de la Universidad de Jaén, y D. José Manuel Palomar Carnicero, Doctor Ingeniero Industrial y Profesor Titular de Universidad del Departamento de Ingeniería Mecánica y Minera de la Escuela Politécnica Superior de la Universidad de Jaén.

MANIFIESTAN:

Que la presente Memoria titulada “ESTUDIO Y ANÁLISIS DE LA CINÉTICA DE SECADO DE SUBPRODUCTOS DE ALMAZARA PARA SU APLICACIÓN A SECADEROS ROTATIVOS”, presentada por D. Francisco Javier Gómez de la Cruz para optar al grado de Doctor por la Universidad de Jaén, ha sido realizada bajo nuestra dirección en el Departamento de Ingeniería Mecánica y Minera de la Universidad de Jaén y, por ello, autorizamos la presentación de la misma.

Jaén, 6 de Mayo de 2015

Fdo. Fernando A. Cruz Peragón

Fdo. José Manuel Palomar Carnicero

Memoria presentada por D. Francisco Javier Gómez de la Cruz para optar al Grado de Doctor por la Universidad de Jaén.

Fdo. Francisco Javier Gómez de la Cruz.

## TESIS COMO COMPENDIO DE PUBLICACIONES

La presente Tesis Doctoral se presenta como agrupación de los trabajos de investigación publicados por el doctorando en medios científicos relevantes en su ámbito de conocimiento. Se han cumplido los siguientes requisitos:

- (I) La Tesis Doctoral pertenece al Programa Oficial de Doctorado en Ingeniería y Arquitectura (R.D. 1993/2007) “Modelado de Sistemas Térmicos” del departamento de Ingeniería Mecánica y Minera.
- (II) La presentación de la Memoria ha sido realizada según indica el “Artículo 23.3 Tesis Doctoral” de la NORMATIVA DE LA UNIVERSIDAD DE JAÉN PARA LA DEFENSA DE TESIS DOCTORALES.
- (III) Las publicaciones presentadas son conformes a los criterios de calidad mínimos para que una tesis doctoral pueda iniciar el trámite de evaluación y defensa en los programas de doctorado vigentes. La presente Memoria se enmarca dentro de los Programas de Doctorado del ámbito de la Ingeniería y Arquitectura según la CNEAI (Campo 6 Ingeniería y Arquitectura), y la cual indica en su apartado a) que:  
“Los trabajos deben ser publicados en revistas de reconocida valía, aceptándose como tales las incluidas en los listados por ámbitos científicos en el Subject Category Listing del Journal Citation Reports (Science Citation Index) del Web of Knowledge (WoK)”.

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## DIFUSIÓN DE RESULTADOS

La presente Tesis Doctoral forma parte de los estudios realizados bajo el soporte económico del proyecto “Modelado y Control de Secadero Rotativo de Orujo (AGR-6131)” de la Junta de Andalucía (Proyectos de excelencia 2010-2014).

Los trabajos descritos en esta Memoria se encuentran recogidos en las siguientes publicaciones y comunicaciones a congresos:

### PUBLICACIONES INTERNACIONALES INCLUIDAS EN EL JOURNAL CITATION REPORTS.

F.J. Gómez-de la Cruz, P.J. Casanova-Peláez, J.M Palomar-Carnicero, F. Cruz-Peragón, 2014. *Drying kinetics of olive stone: a valuable source of biomass obtained in the olive oil extraction*. Energy; 75: 146-152. ISSN: 0360-5442. Impact Factor: 4.159

DOI:10.1016/j.energy.2014.06.085

#### Journal Ranking

For 2013, the journal ENERGY has an Impact Factor of 4.159.

This table shows the ranking of this journal in its subject categories based on Impact Factor.

Category Name	Total Journals in Category	Journal Rank in Category	Quartile in Category
ENERGY & FUELS	83	13	Q1
THERMODYNAMICS	55	2	Q1

F.J. Gómez-de la Cruz, F. Cruz-Peragón, P.J. Casanova-Peláez, J.M Palomar-Carnicero, 2015. *A vital stage in the large-scale production of biofuels from spent coffee grounds: The drying kinetics*. Fuel Processing Technology; 130: 188-196. ISSN: 0378-3820. Impact Factor: 3.019.

DOI:10.1016/j.fuproc.2014.10.012

#### Journal Ranking

For 2013, the journal FUEL PROCESSING TECHNOLOGY has an Impact Factor of 3.019.

This table shows the ranking of this journal in its subject categories based on Impact Factor.

Category Name	Total Journals in Category	Journal Rank in Category	Quartile in Category
CHEMISTRY, APPLIED	71	12	Q1
ENERGY & FUELS	83	27	Q2
ENGINEERING, CHEMICAL	133	18	Q1

F.J. Gómez-de la Cruz, P.J. Casanova-Peláez, J.M Palomar-Carnicero, F. Cruz-Peragón, 2015. *Modeling of olive-oil mill waste rotary dryers: Green energy recovery systems*. Applied Thermal Engineering; 80: 362-373. ISSN: 1359-4311. Impact Factor: 2.624.

DOI: [10.1016/j.applthermaleng.2015.01.035](https://doi.org/10.1016/j.applthermaleng.2015.01.035).

**Journal Ranking** ⓘ

For 2013, the journal **APPLIED THERMAL ENGINEERING** has an Impact Factor of **2.624**.

This table shows the ranking of this journal in its subject categories based on Impact Factor.

Category Name	Total Journals in Category	Journal Rank in Category	Quartile in Category
ENERGY & FUELS	83	34	Q2
ENGINEERING, MECHANICAL	128	10	Q1
MECHANICS	139	8	Q1
THERMODYNAMICS	55	6	Q1

F.J. Gómez-de la Cruz, J.M Palomar-Carnicero, P.J. Casanova-Peláez, F. Cruz-Peragón, 2015. *Experimental determination of effective moisture diffusivity during the drying of clean olive stone: dependence of temperature, moisture content and sample thickness*. Fuel Processing Technology; In Press. ISSN: 0378-3820. Impact Factor: 3.019.

DOI: [10.1016/j.fuproc.2015.03.018](https://doi.org/10.1016/j.fuproc.2015.03.018)

**Journal Ranking** ⓘ

For 2013, the journal **FUEL PROCESSING TECHNOLOGY** has an Impact Factor of **3.019**.

This table shows the ranking of this journal in its subject categories based on Impact Factor.

Category Name	Total Journals in Category	Journal Rank in Category	Quartile in Category
CHEMISTRY, APPLIED	71	12	Q1
ENERGY & FUELS	83	27	Q2
ENGINEERING, CHEMICAL	133	18	Q1

F.J. Gómez-de la Cruz, P.J. Casanova-Peláez, R. López-García, F. Cruz-Peragón. *Review of the Drying Kinetics of Olive Oil Mill Wastes: Biomass Recovery*. Bioresources. (Under Review). ISSN: 1930-2126. Impact Factor: 1.549.

**Journal Ranking** ⓘ

For 2013, the journal **BioResources** has an Impact Factor of **1.549**.

This table shows the ranking of this journal in its subject categories based on Impact Factor.

Category Name	Total Journals in Category	Journal Rank in Category	Quartile in Category
MATERIALS SCIENCE, PAPER & WOOD	21	4	Q1

## CONGRESOS INTERNACIONALES.

F.J. Gómez-de la Cruz, P.J. Casanova-Peláez, J.M. Palomar-Carnicero, S. Sánchez, R. Pacheco, F. Cruz-Peragón, 2014. *Obtaining of the drying rate of alpeorujó for application on rotary dryers*. ICERE 2014 International Conference on Environment and Renewable Energy, (París, Francia).

F.J. Gómez-de la Cruz, P.J. Casanova-Peláez, J.M. Palomar-Carnicero, S. Sánchez, R. Pacheco, F. Cruz-Peragón, 2014. *Average and variable effective moisture diffusivity of thin layer drying of olive stone*. ICERE 2014 International Conference on Environment and Renewable Energy, (París, Francia).

F.J. Gómez-de la Cruz, F. Cruz-Peragón, P.J. Casanova-Peláez, S. Sánchez, R. Pacheco, J.M. Palomar-Carnicero, 2014. *Hot-air drying characteristics of olive stone*. GREENCHEMSE 2014 International Conference on Green Chemistry and Sustainable Engineering, (Barcelona, España).

F.J. Gómez-de la Cruz, F. Cruz-Peragón, P.J. Casanova-Peláez, S. Sánchez, R. Pacheco, J.M. Palomar-Carnicero, 2014. *Variable effective moisture diffusivity determination in the drying of spent coffee grounds*. GREENCHEMSE 2014 International Conference on Green Chemistry and Sustainable Engineering, (Barcelona, España).

F.J. Gómez-de la Cruz, F. Cruz-Peragón, P.J. Casanova-Peláez, S. Sánchez, R. Pacheco, J.M. Palomar-Carnicero, 2015. *Water diffusion coefficients during the drying of two-phase olive mill waste from an experimental design of two variables*. CIEM 2015 International Congress of Energy and Environment Engineering and Management, (París, Francia).

F.J. Gómez-de la Cruz, F. Cruz-Peragón, P.J. Casanova-Peláez, S. Sánchez, R. Pacheco, J.M. Palomar-Carnicero, 2015. *New experimental equipment based on a rotary dryer for drying of olive mill by-products*. CIEM 2015 International Congress of Energy and Environment Engineering and Management, (París, Francia).



*A mis padres,  
esposa,  
abuelos,  
y a toda mi gran familia.*

***“La raza humana necesita un desafío intelectual. Debe ser aburrido ser Dios, y no tener nada que descubrir”***

**(Stephen W. Hawking)**



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### III. NOMENCLATURA.

#### LETRAS EN LATÍN.

$a, b, c, d, e, f, n$	Coefficientes de los modelos matemáticos de capa delgada
$a_0, a_1, a_2, a_3, a_4, a_5$	Coefficientes del modelo polinómico multivariable de 2º orden
$A$	Constante empírica de la correlación de Saeman y Mitchell
$c_p$	Calor específico a presión constante ( $J \cdot kg^{-1} \cdot K^{-1}$ )
$D$	Diámetro del trómel (m)
$d_p$	Diámetro de partícula ( $\mu m$ )
$D_{eff}$	Difusividad de humedad efectiva ( $m^2/s$ )
$D_0$	Factor pre-exponencial de la ecuación de Arrhenius ( $m^2/s$ )
$DR$	Tasa de secado ( $kg_w/kg_{dp} \cdot s$ )
$E_a$	Energía de activación ( $J \cdot mol^{-1}$ )
$Fo$	Número de Fourier de difusión
$G$	Flujo de masa, por unidad de área. ( $kg \cdot s^{-1} \cdot m^{-2}$ )
$H$	Contenido de humedad en el subproducto, base húmeda
$k, k_0, k_1, k_2, k_3$	Constantes de los modelos matemáticos de capa delgada ( $s^{-1}$ )
$K'$	Constante empírica de la correlación de Kelly
$K$	Constante empírica para calcular el coeficiente volumétrico de transferencia de calor
$L$	Longitud del trómel (m)
$L_c$	Espesor de la muestra (m)
$LMTD$	Diferencia de temperaturas media logarítmica
$m$	Masa (kg)
$\dot{m}$	Flujo de masa ( $kg \cdot s^{-1}$ )
$N$	Velocidad angular del trómel (rpm)
$p_1, p_2$	Coefficientes de ajuste de la derivada de la función parabólica
$Q_G$	Calor del gas de la combustión (J)
$Q_H$	Calor que eleva la temperatura en el gas (J)
$Q_L$	Calor que eleva la temperatura de la fase líquida en el subproducto (J)
$Q_P$	Calor que eleva la temperatura del subproducto (J)

$Q_v$	Calor que evapora una porción líquida en el subproducto (J)
$R$	Constante universal de los gases ( $J \cdot kg^{-1} \cdot K^{-1}$ )
$R^2$	Coefficiente de determinación
$RMSE$	Raíz cuadrada del error cuadrático medio
$s$	Pendiente matemática
$S$	Pendiente del trómel
$t$	Tiempo (s)
$T$	Temperatura ( $^{\circ}C$ , K)
$U$	Coefficiente volumétrico de transferencia de calor ( $W \cdot m^{-3} \cdot K^{-1}$ )
$v$	Velocidad (m/s)
$V$	Volumen ( $m^3$ )
$x$	Dimensión espacial de transporte de masa (m)
$x_v$	Tasa de secado ( $s^{-1}$ )
$X$	Contenido de humedad del subproducto, base seca
$XR$	Ratio de humedad
$Y$	Contenido de humedad en el aire, base seca

#### LETRAS EN GRIEGO.

$\lambda$	Calor latente de vaporización del agua
$\nu$	Constante empírica para calcular el coeficiente volumétrico de transferencia de calor
$\bar{\tau}$	Tiempo de residencia medio

#### SUBÍNDICES.

$a$	Aire
$cal$	Calculada
$da$	Aire seco
$dp$	Subproducto seco
$e$	Equilibrio

<i>ev</i>	Evaporada
<i>exp</i>	Experimental
<i>in</i>	Entrada
<i>out</i>	Salida
<i>p</i>	Subproducto
<i>t</i>	En el instante <i>t</i>
<i>v</i>	Vapor
<i>w</i>	Agua
0	Inicial



## RESUMEN.

Debido a la importancia del sector oleícola en la provincia de Jaén y, por consiguiente, a la actividad agroindustrial desarrollada, es necesaria la inversión en técnicas de mejora en este campo. En concreto, esta investigación está centrada en el tratamiento de secado de los subproductos obtenidos tras la molturación de la aceituna, *el alpeorujo* y *el hueso de aceituna*.

La transición del sistema de extracción de aceite de oliva de tres fases al de dos fases, ha propiciado que el secado del *alpeorujo* se haya complicado enormemente debido a su alto contenido de humedad (60-70 %, base húmeda), su naturaleza y composición. En estas condiciones, los principales sistemas de secado de este subproducto, los secaderos rotativos, necesitan mejorar su rendimiento que se vio afectado por el cambio de un subproducto de tres fases (*orujo*) a uno de dos fases (*alpeorujo*). Por este motivo, es necesario analizar los principales modelos matemáticos que estudian los fenómenos de transferencia de calor y masa en secaderos rotativos y mejorar las condiciones durante el secado, así como su eficiencia.

Por otro lado, la separación del *hueso de aceituna* del *alpeorujo* ha propiciado su revalorización como biomasa. Sin embargo, para obtener un biocombustible eficiente, una de las principales medidas pasa por un secado efectivo.

Esta Tesis Doctoral, revisa y analiza en una primera instancia, los principales modelos matemáticos de transferencia de calor y masa en secaderos rotativos de subproductos de almazara, así como los trabajos llevados a cabo por los investigadores hasta la fecha sobre el estudio de la cinética de secado.

En segundo lugar, y como parte experimental y fundamental de este trabajo, se realizan nuevos estudios sobre la cinética de secado en el *alpeorujo* y *hueso de aceituna*. Adicionalmente, se han realizado experimentos de secado con otros biocombustibles emergentes como los *posos de café*, los cuales han apoyado estas investigaciones.

Los estudios de la cinética de secado en estos subproductos han sido realizados en un secadero convectivo experimental y los resultados obtenidos se aplicarán principalmente a mejorar los procesos de secado en secaderos rotativos.



## 1. INTRODUCCIÓN.

El aceite de oliva es uno de los alimentos más importantes en la dieta Mediterránea, particularmente el aceite de oliva virgen extra, el cual presenta excelentes beneficios nutricionales y para la salud. La producción anual media de aceite de oliva está alrededor de las 2.8 millones de toneladas ([International Olive Council \(IOC\), 2014b](#)). Aproximadamente, dos millones de toneladas son producidas en la Unión Europea, que acapara el 70 % de la producción mundial. España, Italia, y Grecia son los mayores productores de aceite de oliva con unas producciones anuales medias de 1.215, 0.456, y 0.318 millones de toneladas, respectivamente ([International Olive Council \(IOC\), 2014a](#)).

A lo largo del siglo XX, los métodos de presión tradicionales dedicados a la extracción de aceite de oliva han sido reemplazados gradualmente por nuevas tecnologías basadas en diferentes procesos como la molturación y trituración a través del uso de martillos de acero inoxidable, batido termo-mecánico, y centrifugación horizontal en sistemas continuos de decantado ([Altieri et al., 2013](#); [Boncinelli et al., 2009](#)). Actualmente, son usados dos sistemas de decantación, o sistemas de centrifugación en continuo, en la extracción de aceite de oliva: el sistema de tres fases y el sistema de dos fases.

Desde la década de los setenta hasta la década de los noventa, el proceso de extracción de aceite de oliva era llevado a cabo en decantadores de tres fases: aceite de oliva virgen, *orujo* y *alpechín*. El *orujo* es un subproducto formado a partir de piel, fragmentos de hueso y pulpa de la aceituna, junto con un cierto contenido de aceite de oliva, entre el 5 % y el 8 %. Sin embargo, este sistema presentaba varios problemas medioambientales. En primer lugar, grandes cantidades de agua eran necesarias antes del proceso de decantación para separar correctamente las tres fases y, segundo, el *alpechín*, que era almacenado y controlado en balsas ubicadas generalmente cerca de las almazaras, presentaba un serio problema medioambiental debido a su alta demanda bioquímica de oxígeno (DBO) y a los fuertes olores emanados.

El sistema de decantación de dos fases, o sistema ecológico, fue implantado a principios de la década de los noventa con el objetivo de eliminar los problemas medioambientales causados por el sistema de decantación de tres fases, así como mejorar el uso de los recursos hídricos más eficientemente ([Bagatin et al., 2014](#); [Klemeš](#)

et al., 2013; Wan Alwi et al., 2014). En este sistema solo dos componentes son obtenidos: el aceite de oliva virgen y un subproducto formado por una mezcla de *orujo* y aguas de vegetación, el cual es llamado *alpeorujo*. Sin embargo, este sistema produce un subproducto con un alto contenido de humedad, entre el 60 % y el 70 % (base húmeda), en comparación con el contenido de humedad del *orujo* obtenido en el sistema de tres fases, el cual presentaba contenidos de humedad entre el 35 % y el 45 %.

Por tanto, estos subproductos necesitan ser secados por tres motivos principales. Primero para eliminar un problema medioambiental peligroso, segundo para extraer el aceite de oliva contenido en ellos y tercero para obtener un biocombustible sólido apto para la generación de energía, el *orujillo*. Para la extracción del aceite de oliva en estos subproductos, tanto el *orujo* como el *alpeorujo* necesitan ser secados hasta valores cercanos a su humedad de equilibrio, en torno al 7.5 % (base húmeda) (Moral and Méndez, 2006). Con estas condiciones, el disolvente (generalmente hexano) usado en el proceso de extracción del aceite de *orujo* es más efectivo. Inicialmente, el aceite extractado es refinado, y posteriormente es combinado con aceite de oliva virgen. La producción de aceite de *orujo* representa el 10 % de todos los aceites de oliva, el cual está siendo considerado incluso en la producción de biodiesel (Lama-Muñoz et al., 2014; Lopez et al., 2014; Ouachab and Tsoutsos, 2013). Tras este proceso, un subproducto biomásico con un considerable poder calorífico inferior, en torno a 17.5 MJ/kg, es obtenido, el *orujillo*. Esta abundante fuente de energía verde tiene especial interés como biocombustible para generación de energía eléctrica en plantas de generación y cogeneración (tabla 1.1) (Cruz-Peragón et al., 2006; Jurado et al., 2003), y para la generación de energía térmica en procesos industriales y calefacción (García-Maraver et al., 2012). El coste por kg es bajo comparado con otros combustibles fósiles como el gasoil o el gas natural, siendo un duro competidor con respecto a otros combustibles biomásicos. La tabla 1.2 indica el consumo anual por tipo de biomasa en Andalucía y sus características más importantes (Agencia Andaluza de la Energía (AAE), 2013).

En la actualidad, el proceso físico del secado es el único método para tratar los subproductos de almazara, y la gran mayoría son secados en secaderos rotativos en isocorriente (Gómez-de la Cruz et al., 2015c). Grandes cantidades de estos subproductos son tratados anualmente en las industrias de extracción secundarias, las *orujeras*.

**Tabla 1.1** Plantas de generación de energía eléctrica con biomasa en Andalucía (Agencia Andaluza de la Energía (AAE), 2013).

PLANTA	Potencia (MW)	Tipo de biomasa	Localidad	Provincia
Albaida recursos naturales	1.70	Restos de invernaderos	Níjar	Almería
Agroenergética Baena	25.00	Alpeorujos	Baena	Córdoba
Bioenergética Egabrense	8.00	<i>Orujillo</i>	Cabra	Córdoba
Severaes	0.10	Poda de olivo	Cañete de las Torres	Córdoba
Bioenergía Santamaría	14.30	<i>Orujillo</i>	Lucena	Córdoba
Hermanos Santa María Muñoz e hijos	1.72	<i>Orujillo</i>	Lucena	Córdoba
Agroenergética de Palenciana	5.37	Alpeorujos	Palenciana	Córdoba
El tejedor autogeneración	5.65	Alpeorujos	Palenciana	Córdoba
Vetejar	12.90	Alpeorujos	Palenciana	Córdoba
Biomasa Puente Genil	9.82	<i>Orujillo</i>	Puente Genil	Córdoba
Ence I	40.95	Madera	San Juan del Puerto	Huelva
Ence II	27.00	Madera	San Juan del Puerto	Huelva
Tradema	2.00	Madera	Linares	Jaén
Bioenergética de Linares	15.00	<i>Orujillo</i> , residuos agrícolas y forestales cultivos energéticos	Linares	Jaén
La Loma	16.00	<i>Orujillo</i>	Villanueva del Arzobispo	Jaén
Aldebarán Energía del Guadalquivir	6.00	Poda de olivo, residuos forestales, paja de cereal	Andújar	Jaén
Fuente de Piedra	8.04	<i>Orujillo</i>	Fuente de Piedra	Málaga
Extragol	9.15	<i>Orujillo</i> , poda, cultivos energéticos	Villanueva de Algaidas	Málaga
Total	208.7			

**Tabla 1.2** Características de la biomasa y consumo anual por tipo de biomasa en Andalucía, (Agencia Andaluza de la Energía (AAE), 2013).

Biomasa	Energía (KTEP)	PCI (kJ/kg base seca)	Contenido de humedad (% base húmeda)
<i>Orujillo</i>	216.40	17500	(8-12)
<i>Hueso de aceituna</i>	173.83	18800	(10-15)
Madera forestal	54.35	18000	(40-60)
Pellets, astillas de madera	16.22	17500	<12
Biogás	6	(14200- 30000)	---

Los secaderos rotativos de residuos de almazara fueron diseñados para secar *orujo* (sistema de tres fases). Sin embargo, debido a la transición hacia el sistema de dos fases, el secado del *alpeorujo* presenta serios problemas como consecuencia de su alto contenido de humedad, naturaleza y composición. Esto implica un elevado coste energético para eliminar grandes cantidades de agua. Así mismo, compuestos contenidos en el *alpeorujo*, como los azúcares, contribuyen a la formación de aglomeraciones durante el proceso de secado y a su adhesión a las paredes del trómel.

La mejora del rendimiento de estas instalaciones es, actualmente, uno de los principales objetivos, siendo necesario lograr una optimización entre el coste energético y los productos obtenidos con una considerable calidad. Para resolver el problema, los investigadores se centran en la automatización y control de los secaderos: trómel y horno (Arjona, 2002; Arjona et al., 2005; Casanova-Peláez, 2009; Casanova-Peláez et al., 2012; Castaño et al., 2001; Castaño et al., 2012), y en el control de los parámetros más importantes durante el proceso de secado: temperaturas del gas de secado a la entrada y salida del trómel, y temperaturas y humedades de los residuos a la entrada y salida del trómel.

La presente Tesis Doctoral analiza y revisa en una primera instancia las principales contribuciones de la cinética de secado y los principales modelos matemáticos de transferencia de calor y masa en secaderos rotativos de los principales subproductos de almazara (*orujo* y *alpeorujo*). Sin embargo, el principal objetivo de esta Memoria es presentar nuevos estudios sobre la cinética de secado en el *alpeorujo* y en el *hueso de aceituna* que ayuden a mejorar y completar el proceso de secado a gran escala en secaderos rotativos. Además, se reflejan estudios realizados sobre la cinética de secado de los *posos de café*, un biocombustible emergente, para contribuir al estudio de los subproductos de almazara.

## 1.1 REVISIÓN SOBRE LA CINÉTICA DE SECADO DE LOS SUBPRODUCTOS DE ALMAZARA.

El proceso de secado de residuos de almazara es normalmente un fenómeno complejo donde intervienen fenómenos de transferencia de calor y masa por convección y difusión. Esto es debido principalmente a la diversa composición del subproducto: partículas formadas por huesos y pulpa de diferentes tamaños, tamaños de muestra en las aspas del trómel, agua vegetal, azúcares y a las diferentes condiciones de temperatura y velocidad del aire de secado. Durante su recorrido en el trómel se producen intercambios de energía (calor cedido al sólido desde el medio gaseoso caliente circundante) y de masa (la fracción líquida que al evaporarse abandona el sólido y pasa a formar parte de la corriente gaseosa). Así pues, el sólido se calienta y se seca, y el gas de secado se humedece y se enfría.

El contenido de humedad puede alojarse en los residuos de almazara de tres formas distintas (Perry and Green, 2007):

-En la superficie (no llegando a mezclarse con el sólido), por lo que es la parte más fácil de evaporar.

-En los poros del sólido, por lo que necesita ser llevada a la superficie para poder evaporarse.

-Por capilaridad, íntimamente unida al sólido y por tanto, que requiere más esfuerzo para su extracción.

Diferentes investigadores han analizado la cinética de secado del *orujo* y del *alpeorujo* en otros sistemas de secado como el secado en lecho fluido (Liébanes et al., 2006; Meziane, 2011), secaderos convectivos (Akgun and Doymaz, 2005; Arjona et al., 1999; Casanova-Peláez et al., 2015; Doymaz et al., 2004a; Freire et al., 2001; Gögüs and Maskan, 2006; Krokida et al., 2002; Vega-Gálvez et al., 2010), secaderos por microondas con convección asistida (Gögüs and Maskan, 2001; Milczarek et al., 2011), secaderos por infrarrojos (Ruiz Celma et al., 2008) y secaderos solares (Celma et al., 2007a; Montero et al., 2010; Montero et al., 2011a). El estudio experimental de la cinética de secado en los subproductos de almazara es fundamental para entender y mejorar los procesos de secado a gran escala en secaderos rotativos. La cinética de

secado permite obtener las condiciones de secado, contenido de humedad de la muestra y temperatura del aire de secado, en las cuales aparecen la liberación de materia volátil e incluso la combustión. Además, estos fenómenos están íntimamente relacionados con la aparición de hidrocarburos aromáticos policíclicos (HAP), principalmente el benzopireno, en el aceite de *orujo* (Ergönül and Sánchez, 2013; Moreda et al., 2004). Actualmente, en la Unión Europea el máximo límite tolerable para el benzopireno en el grupo de los aceites y grasas destinados para el consumo humano o uso como ingrediente en alimentos ha sido establecido en un nivel de 2 µg/kg por la Commission Regulation (EU) nº 835/2011 de 19 de agosto de 2011, desde el 1 de septiembre de 2012 (Commission of the European Communities, 2011). Arjona et al. (1999) han demostrado que la liberación de volátiles y el inicio de la combustión ocurrían a temperaturas por encima de 200°C y 250°C, respectivamente. Por otro lado, la cinética de secado es capaz de identificar los mecanismos de transferencia de calor y masa, los tiempos de secado y las tasas de secado en cada instante de tiempo. Con respecto al secado del *orujo* y *alpeorujo*, la gran mayoría de los investigadores acuerdan que es producido principalmente por el fenómeno de difusión en *el periodo de caída de la tasa de secado*. En estos estudios, establecer las curvas de secado es el primer paso en analizar la cinética de secado. Los experimentos analizan la variación del contenido de agua en la muestra con respecto al tiempo bajo condiciones isotérmicas, a una velocidad constante de aire de secado determinada y un espesor de muestra específico. Las curvas de secado, ratio de humedad frente al tiempo, son aproximadas con los principales modelos matemáticos de capa delgada en el secado de productos agrícolas (Tabla 1.3).

**Tabla 1.3** Modelos matemáticos de capa delgada en el secado de subproductos de almazara.

Nombre del modelo	Ecuación	Referencia
Lewis	$XR = \exp(-kt)$	(Lewis, 1921)
Page	$XR = \exp(-kt^n)$	(Page, 1949)
Modified Page	$XR = \exp(-(kt)^n)$	(Overhults et al., 1973)
Henderson and Pabis	$XR = a \cdot \exp(-kt)$	(Henderson and Pabis, 1961)
Logarithmic	$XR = a \cdot \exp(-kt) + c$	(Akgun and Doymaz, 2005)
Wang and Singh	$XR = 1 + at + bt^2$	(Wang and Singh, 1978)
Two term	$XR = a \cdot \exp(-k_0t) + c \cdot \exp(-k_1t)$	(Noomhorm and Verma, 1986)
Approach of Diffusion	$XR = a \cdot \exp(-kt) + (1 - a) \cdot \exp(-kbt)$	(Yaldiz et al., 2001)
Midilli et al.	$XR = a \cdot \exp(-kt^n) + bt$	(Midilli et al., 2002)
Two term Gaussian	$XR = a \cdot \exp\left[-\left(\frac{t-b}{c}\right)^2\right] + d \cdot \exp\left[-\left(\frac{t-e}{f}\right)^2\right]$	(Gómez-de la Cruz et al., 2014b)

El ratio de humedad puede ser expresado como:

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

donde  $X_t$  es el contenido de humedad en el instante  $t$ ,  $X_0$  es el contenido de humedad inicial y  $X_e$  es el contenido de humedad de equilibrio, expresándose las unidades en base seca. Sin embargo, el ratio de humedad puede ser expresado como  $XR = X_t/X_0$ , ya que la humedad de equilibrio suele ser pequeña con respecto a la humedad inicial. La tasa de secado es calculada experimentalmente a partir de la variación del contenido de humedad con respecto al tiempo como:

$$DR \approx -\frac{X_{t+\Delta t} - X_t}{\Delta t} \quad (2)$$

donde  $DR$  es la tasa de secado ( $kg_w/kg_{ds}\cdot s$ ),  $X_{t+\Delta t}$  y  $X_t$  representan el contenido de humedad en el instante  $t + \Delta t$  y el contenido de humedad en el instante  $t$  ( $kg_w/kg_{ds}$ ), respectivamente, y  $t$  es el tiempo de secado ( $s$ ). El signo negativo es incluido para indicar la tasa de secado con valores positivos. La tasa de secado puede ser calculada a partir de la derivada con respecto al tiempo de los modelos matemáticos de capa delgada propuestos en la [tabla 1.3](#) usando la [Ec. \(3\)](#):

$$x_v = -\frac{d(XR)}{dt} \quad (3)$$

En esta ecuación  $x_v$  indica que la tasa de secado es expresada como la inversa del tiempo ( $s^{-1}$ ).

Tanto el tiempo de secado como la tasa de secado dependen especialmente de la temperatura del aire de secado, del contenido de humedad de la muestra, de la velocidad del aire de secado, del espesor de la muestra a secar y del tamaño de partícula.

### 1.1.1 Dependencia de la temperatura de secado y del contenido de humedad de la muestra.

La temperatura de aire de secado es el factor más decisivo en el proceso de secado, independientemente de los otros factores (Freire et al., 2001). La tasa de secado se incrementa linealmente independientemente del contenido de humedad, siendo incluso más sensible para altos valores de humedad. Esto se debe a que la resistencia de la transferencia de calor y masa es mayor a bajos contenidos de humedad. Por otro lado, el tiempo de secado también decrece con la temperatura. De hecho, cuanto mayor es la temperatura, menor es el tiempo de secado y mayores tasas de secado son obtenidas.

Otro factor importante que explica adecuadamente el proceso de transferencia de masa por difusión durante el secado de los subproductos de almazara es la difusividad de humedad efectiva. Esta variable está asociada con la difusión de masa dentro del medio durante los cambios de contenido de humedad con respecto al tiempo y puede ser obtenida experimentalmente a partir de la segunda ley de la difusión de Fick (Ec. (4)). La solución de esta ecuación en derivadas parciales (Ec. (5)) fue propuesta por Crank (Crank, 1975) para el transporte de masa unidimensional en geometría de losa infinita.

$$\frac{\partial(XR)}{\partial t} = D_{eff} \frac{\partial^2(XR)}{\partial x^2} \quad (4)$$

$$XR = \frac{8}{\pi^2} \sum_{j=0}^{\infty} \frac{1}{(2j+1)^2} \exp\left(-\frac{(2j+1)^2 \pi^2 t D_{eff}}{L_c^2}\right) \quad (5)$$

donde  $D_{eff}$  es la difusividad de humedad efectiva ( $m^2/s$ ),  $t$  es el tiempo de secado ( $s$ ),  $x$  es la dimensión espacial por donde se transfiere la humedad ( $m$ ) y  $L_c$  es el espesor de la muestra ( $m$ ). Para grandes periodos de tiempo, la ecuación puede ser aproximada al primer término de la serie y tomando logaritmos en ambos lados se tiene:

$$\ln XR = \ln \frac{8}{\pi^2} - \frac{\pi^2 t D_{eff}}{L_c^2} \quad (6)$$

Esta ecuación representa la dependencia lineal entre  $\ln XR$  y el tiempo de secado. En este caso, la difusividad de humedad efectiva puede ser calculada a partir de la pendiente de la función lineal que aproxima los datos experimentales obtenidos.

A partir de los datos experimentales, otra variable puede ser obtenida, la energía de activación. Esta variable indica la energía mínima necesaria para producir el proceso de difusión en un medio, en otras palabras, la energía mínima para comenzar el secado. La dependencia de la temperatura de la difusividad de humedad efectiva puede ser representada por una relación tipo Arrhenius como:

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

donde  $D_0$  es el factor pre-exponencial de la ecuación de Arrhenius ( $m^2/s$ ),  $E_a$  es la energía de activación ( $kJ/mol$ ),  $R$  es la constante de los gases ideales ( $kJ/mol K$ ) y  $T$  es la temperatura absoluta ( $K$ ).

### 1.1.2 Dependencia de la velocidad del aire de secado.

Experimentos llevados a cabo hasta la fecha han demostrado que la velocidad del aire de secado interviene considerablemente en la tasa de secado y en el tiempo de secado. De hecho, el efecto de la velocidad llega a ser más decisivo en la primera etapa del proceso de secado (incrementando la tasa de secado) tanto para altas como bajas temperaturas (Casanova-Peláez et al., 2015). Sin embargo, para bajos contenidos de humedad, el efecto de la velocidad llega a ser despreciable (la migración de agua hacia la superficie es insignificante cuando es dependiente de la velocidad). Freire et al. (2001) identificaron la existencia de una velocidad crítica. Para valores por encima de esta velocidad crítica, el incremento en la tasa de secado llega a ser muy pequeño y el secado es controlado por mecanismos de transferencia de masa internos (migración de la humedad a través de la estructura interna del subproducto). Por otro lado, para valores por debajo de la velocidad crítica, las variaciones en la tasa de secado no deben ser despreciadas. Esto indica que la temperatura del aire de secado prevalece sobre la velocidad del aire de secado.

### 1.1.3 Dependencia del espesor de la muestra.

Otra vía para analizar la tasa de secado y el tiempo de secado es a partir del espesor de la muestra a secar. Los investigadores han demostrado que la tasa de secado aumenta conforme el espesor de la muestra disminuye. El cálculo de la difusividad de humedad efectiva permite obtener similares conclusiones. Estos valores fueron obtenidos para diferentes espesores considerando muestras con geometría de losa infinita, en particular, 6 mm, 9 mm y 12 mm con una velocidad de aire constante de 1.5 m/s entre 60°C y 80°C (Gögüs and Maskan, 2006) y para los espesores de 41 mm, 52 mm y 63 mm con una velocidad de aire constante de 1 m/s entre 50 °C y 80°C (Meziane, 2011). El logaritmo neperiano de  $D_{eff}$  puede ser expresado como una función de la inversa de la temperatura absoluta. Los valores de la energía de activación obtenidos por estos investigadores mostraron que son más grandes cuando los espesores de muestra son mayores. Para los espesores 6 mm, 9 mm y 12 mm, los valores de la energía de activación fueron 25.4 kJ/mol, 25.7 kJ/mol y 29.2 kJ/mol respectivamente, y para los espesores de 41 mm, 52 mm y 63 mm, los valores de la energía de activación fueron 34.05 kJ/mol, 36.84 kJ/mol y 38.10 kJ/mol respectivamente.

### 1.1.4 Dependencia del tamaño de partícula.

Por último, se analiza la influencia que tiene el tamaño de la partícula en el proceso de secado. Los análisis realizados muestran una heterogeneidad en el tamaño de partículas en una muestra concreta. Los resultados obtenidos (Arjona et al., 1999) revelan que la tasa de secado se incrementa cuando el tamaño medio de la partícula,  $d_p$ , disminuye. Esto es evidente ya que la superficie específica (superficie en contacto con el gas por unidad de volumen) de las partículas aumenta cuando el tamaño de partícula disminuye, y por tanto, mayor intercambio de transferencia de calor existe. Sin embargo, hay un fenómeno importante a tener en cuenta cuando el tamaño de partícula presenta valores inferiores a 2.5 mm (Freire et al., 2001). En este rango el tiempo de secado se incrementa cuando el tamaño de partícula decrece. Esto puede ser debido a que la composición de partículas es bastante heterogénea, formada por un alto contenido de humedad y un cierto contenido de aceite. El aceite puede establecer un efecto barrera

que evite la transferencia de humedad en los residuos de almazara. Las investigaciones llevadas a cabo por [Gögüs y Maskan \(2006\)](#) mostraron que partículas de un tamaño mayor a 2 mm tenían una estructura porosa que permitía la salida del agua. Lo contrario ocurría cuando el tamaño de partícula era más pequeño, inferior a 1 mm, provocando la aglomeración de partículas construyendo estructuras no porosas que impiden el transporte de humedad hacia la superficie.

Como se indicó anteriormente, el estudio de la cinética de secado a partir del tamaño de partícula es excesivamente complicado, sobre todo cuando se aplica a secaderos a gran escala donde se hace imposible su control. Por este motivo, los principales parámetros a considerar en la programación de estudios de la cinética de secado serán la temperatura del aire de secado, la velocidad del aire de secado y el espesor de la muestra.

Finalmente, la [tabla 1.4](#) indica un resumen de las variables más importantes en la cinética de secado de los subproductos de almazara a partir de diferentes condiciones de secado en varios tipos de secaderos. Los resultados obtenidos por todos los investigadores indican valores diferentes de difusividad de humedad efectiva y de energía de activación. Los valores de la difusividad de humedad efectiva dependen principalmente de la temperatura del aire de secado y el espesor de la muestra. De hecho, cuando la temperatura del aire de secado y el espesor de la muestra se incrementan, los valores de esta variable lo hacen también. Los valores de la energía de activación dependen principalmente del espesor de la muestra. Cuanto mayor es el espesor de la muestra, mayor es la energía de activación necesaria para producir el secado.

**Tabla 1.4** Resumen de las principales contribuciones en la cinética de secado de subproductos de almazara realizados en diferentes tipos de secaderos.

Tipo de secadero y subproducto	Velocidad (m/s)	Espesor muestra (mm)	Temperatura (°C)	Difusividad de humedad efectiva (m <sup>2</sup> /s)	Energía de activación (kJ/mol)	Referencia
<b>Solar</b> <i>Alpeorujo</i>	1	6.2	20 40 80	2.224·10 <sup>-10</sup> 4.544·10 <sup>-10</sup> 6.993·10 <sup>-10</sup>	15.77	(Celma et al., 2007b)
<b>Solar</b> <i>Orujo</i>	-	30	20 30 40	5.364·10 <sup>-10</sup> 7.622·10 <sup>-10</sup> 1.406·10 <sup>-9</sup>	38.64	(Montero et al., 2011b)
<b>Lecho fluido</b> <i>Orujo</i>	1	41	50 60 70 80	0.68·10 <sup>-7</sup> 0.97·10 <sup>-7</sup> 1.53·10 <sup>-7</sup> 1.93·10 <sup>-7</sup>	34.05	(Meziane, 2011)
<b>Lecho fluido</b> <i>Orujo</i>	1	52	50 60 70	0.82·10 <sup>-7</sup> 1.33·10 <sup>-7</sup> 1.83·10 <sup>-7</sup>	36.84	(Meziane, 2011)
<b>Lecho fluido</b> <i>Orujo</i>	1	63	50, 60 70	0.94·10 <sup>-7</sup> 1.48·10 <sup>-7</sup> 2.15·10 <sup>-7</sup>	38.10	(Meziane, 2011)
<b>Convectivo</b> <i>Orujo</i>	1.2	8	50 60 70 80 90 100 110	3.382·10 <sup>-9</sup> 4.846·10 <sup>-9</sup> 6.252·10 <sup>-9</sup> 7.146·10 <sup>-9</sup> 7.887·10 <sup>-9</sup> 8.818·10 <sup>-9</sup> 1.134·10 <sup>-8</sup>	17.97	(Akgun and Doymaz, 2005)
<b>Convectivo</b> <i>Orujo</i>	1.2	12	80 90 100 110	4.89·10 <sup>-10</sup> 6.188·10 <sup>-10</sup> 7.877·10 <sup>-10</sup> 9.98·10 <sup>-10</sup>	26.71	(Doymaz et al., 2004b)
<b>Convectivo</b> <i>Orujo</i>	1.5	6	60 70 80	1.84·10 <sup>-7</sup> 3.03·10 <sup>-7</sup> 3.42·10 <sup>-7</sup>	25.4	(Gögüs and Maskan, 2006)
<b>Convectivo</b> <i>Orujo</i>	1.5	9	60 70 80	2.17·10 <sup>-7</sup> 2.98·10 <sup>-7</sup> 3.67·10 <sup>-7</sup>	25.7	(Gögüs and Maskan, 2006)
<b>Convectivo</b> <i>Orujo</i>	1.5	12	60 70 80	2.18·10 <sup>-7</sup> 3.22·10 <sup>-7</sup> 3.94·10 <sup>-7</sup>	29.2	(Gögüs and Maskan, 2006)
<b>Convectivo</b> <i>Alpeorujo</i>	2	13	50 60 70 80 90	1.03·10 <sup>-9</sup> 1.2·10 <sup>-9</sup> 1.37·10 <sup>-9</sup> 1.54·10 <sup>-9</sup> 1.71·10 <sup>-9</sup>	12.43	(Vega-Gálvez et al., 2010)
<b>Microondas-Convectivo</b> <i>Alpeorujo</i>	4	7	40 50 60 70	8.95·10 <sup>-10</sup> 1.24·10 <sup>-9</sup> 1.56·10 <sup>-9</sup> 1.95·10 <sup>-9</sup>	22.6	(Milczarek et al., 2011)
<b>Infrarrojos</b> <i>Orujo</i>	-	7	80 100 120 140	5.958·10 <sup>-9</sup> 7.94·10 <sup>-9</sup> 13.901·10 <sup>-9</sup> 15.88·10 <sup>-9</sup>	21.3	(Ruiz Celma et al., 2008)

## 1.2 MODELOS DE ESTUDIO DE TRANSFERENCIA DE CALOR Y MASA EN SECADEROS ROTATIVOS DE SUBPRODUCTOS DE ALMAZARA.

Los modelos de transferencia de calor y masa en el trómel de un secadero rotativo han sido estudiados a partir de modelos completos y modelos por secciones. Los primeros están principalmente basados en variables como el tiempo de residencia y el coeficiente de transferencia de calor volumétrico (Friedman and Marshall, 1949a; Friedman and Marshall, 1949b; Kamke and Wilson, 1986a; Kamke and Wilson, 1986b; Kelly and O'Donnell, 1977; McCormick, 1962; Moss and Nonhebel, 1971; Myklestad, 1963; Perry and Green, 2007). Por otro lado, los modelos completos son analizados tanto bajo condiciones en estado estacionario (Iguaz et al., 2003; Peinado et al., 2011; Pérez-Correa et al., 1998) como en estado transitorio (Riquelme and Benavides, 1991; Yliniemi et al., 1999). Los segundos estudian el trómel a partir de modelos discretizados dividiendo la longitud del trómel en secciones (Kamke and Wilson, 1986b; Rastikian et al., 1999; Wang et al., 1993).

Hasta hoy, el diseño y modelado de un secadero rotativo de residuos de almazara es una difícil tarea de realizar. Aunque no existen muchos investigadores en este campo (Arjona et al., 2005; Casanova-Peláez, 2009; Castaño, 2003; Castaño et al., 2012; Krokida et al., 2002), todos realizan un estudio del trómel en secaderos rotativos en isocorriente. Estos modelos están basados en ecuaciones diferenciales que muestran los principios de la conservación de la masa y la energía. El problema es resolver un modelo matemático que simule las condiciones de temperatura, velocidad del aire de secado y contenidos de humedad en el subproducto dentro del trómel similares a la [figura 1.1](#). Para hacer esto, los investigadores proponen el estudio del trómel aplicando ecuaciones diferenciales al modelo completo, o dividiéndolo en secciones finitas. La variable controlada es la temperatura del aire de secado a la salida del trómel, ya que la medida de humedad in situ es muy complicada. La [figura 1.2](#) exhibe una representación de los fenómenos de transferencia de calor y masa dentro del trómel. La transferencia de calor es principalmente llevada a cabo por fenómenos de conducción-convección. Las pérdidas de calor se consideran despreciables con respecto a otras.

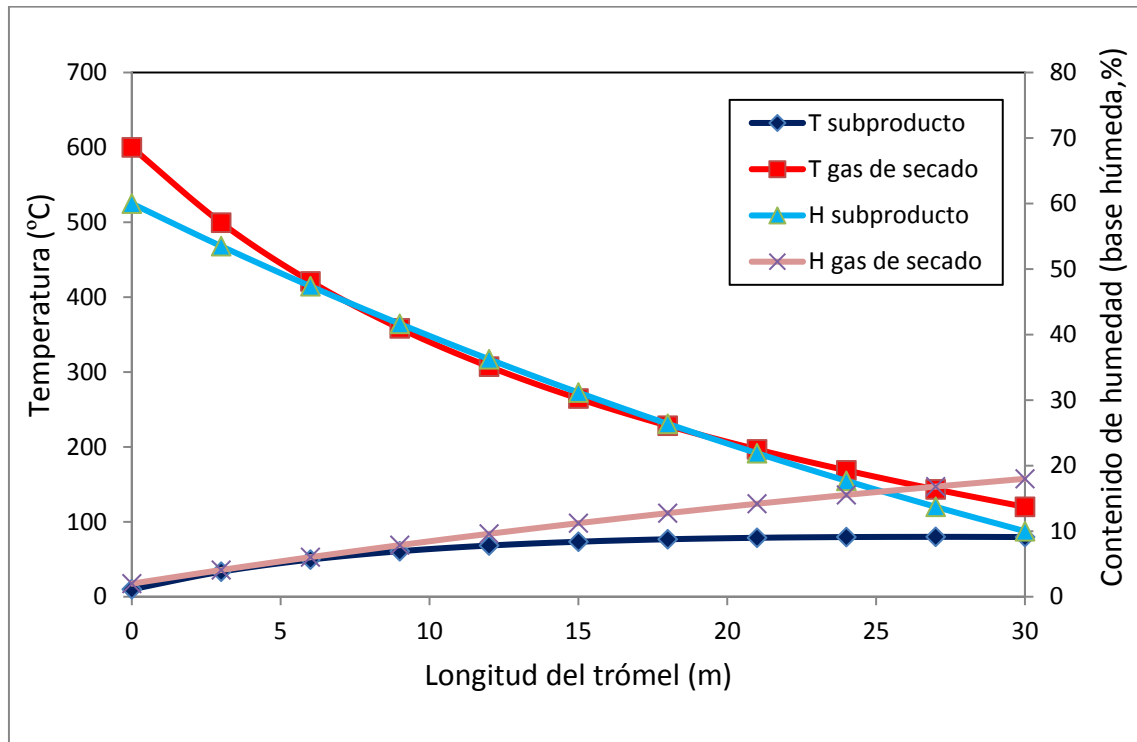


Figura 1.1 Evolución de la temperatura y humedad en un secadero rotativo de subproductos de almazara.

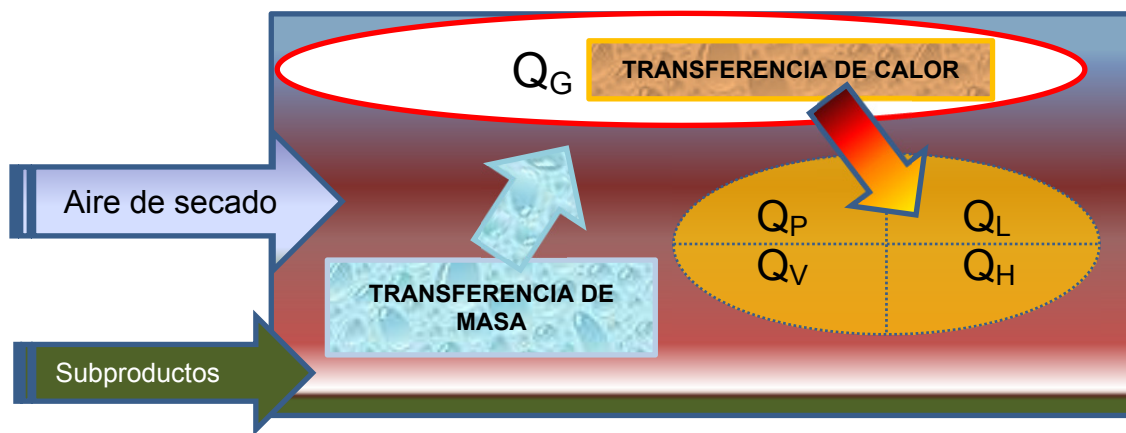


Figura 1.2 Transferencia de calor y masa en el trómel.

El calor que contienen los gases de la combustión ( $Q_G$ ) que entran en el trómel se transforma en:

- Eleva la temperatura de la fase sólida del subproducto ( $Q_P$ ).
- Incrementa la temperatura de la fase líquida del subproducto ( $Q_L$ ).
- Evapora una parte del líquido que contiene el subproducto ( $Q_V$ ).
- Eleva la temperatura del vapor contenido en el gas ( $Q_H$ ).

## 1.2.1 Modelos completos.

### 1.2.1.1 Tiempo de residencia y coeficiente volumétrico de transferencia de calor.

El coeficiente volumétrico de transferencia de calor y el tiempo de residencia son utilizados como submodelos para describir el espacio interno del trómel ocupado por los gases. Estas relaciones son en su mayoría de naturaleza empírica. El tiempo de residencia se define como la masa retenida ( $kg$ ) dividido por el flujo de materia sólida ( $kg/s$ ). Este parámetro puede ser medido conociendo los datos anteriores. La masa podría calcularse extrayéndola del secadero y pesándola. El tiempo medio de residencia puede calcularse como:

$$\bar{\tau} = \frac{m_p}{\dot{m}_p} \quad (8)$$

Sin embargo, para este tipo de sistemas se recurre a funciones matemáticas (Perry and Green, 2007). La ecuación más utilizada en secaderos rotativos fue propuesta por Friedman y Marshall (Friedman and Marshall, 1949a; Friedman and Marshall, 1949b):

$$\bar{\tau} = \frac{0.23 \cdot L}{S \cdot N^{0.9} \cdot D} - 0.6 \frac{5d_p^{-0.5} \cdot L \cdot G_a}{G_p} \quad (9)$$

donde  $L$  es la longitud del secadero ( $m$ ),  $D$  su diámetro interior ( $m$ ),  $N$  es la velocidad de giro del cilindro ( $rpm$ ),  $S$  es su pendiente ( $m/m$ ),  $G_a$  y  $G_p$  ( $kg \cdot s^{-1} \cdot m^{-2}$ ) son las velocidades másicas de alimentación del gas y de la materia sólida seca, respectivamente, y  $d_p$  ( $\mu m$ ) es el tamaño de partícula de peso promedio del material a secar.

Otras investigaciones utilizan la correlación de Kelly (Kelly and O'Donnell, 1977):

$$\bar{\tau} = \frac{K' \cdot L}{S \cdot N \cdot D} \quad (10)$$

siendo  $K'$  una constante empírica. Otra correlación la proporciona Saeman y Mitchell (Moss and Nonhebel, 1971):

$$\bar{\tau} = \frac{L}{A \cdot S \cdot N \cdot D} \quad (11)$$

donde  $A$  es una constante que depende del diseño del trómel.

Las investigaciones realizadas por Casanova (Casanova-Peláez, 2009) en un trómel de un secadero industrial de *alpeorujó* presentan un tiempo de residencia experimental de 21.5 minutos y un tiempo de residencia de diseño de 20 minutos.

El coeficiente volumétrico de transferencia de calor es un parámetro a considerar para la evolución de las variables del tambor del secadero rotativo. Este parámetro es definido como la transferencia de calor transmitida a una unidad de volumen del trómel cuando la diferencia de temperatura es la unidad. Todos los investigadores (Friedman and Marshall, 1949a; Friedman and Marshall, 1949b; McCormick, 1962; Myklestad, 1963) llegan a conclusiones similares: el coeficiente volumétrico de transferencia de calor,  $U$ , depende del flujo del sólido, el flujo del aire y las propiedades físicas del sólido a secar:

$$U = \frac{K \cdot G^v}{D} \quad (12)$$

donde  $G$  es el flujo de gases de secado ( $kg \cdot h^{-1} \cdot m^{-2}$ ),  $D$  es el diámetro del tambor ( $m$ ) y  $K$  y  $v$  son constantes empíricas que dependen del material a secar. Para el *alpeorujó* los investigadores obtienen un coeficiente volumétrico de transferencia de calor que oscila entre  $0.11-0.17 kW \cdot m^{-3} \cdot K^{-1}$  (Casanova-Peláez, 2009). Castaño (2003) obtiene unos valores empíricos de  $v$  y  $K$  de  $0.5595$  y  $336$  respectivamente, con un valor de  $U \approx 0.3 kW \cdot m^{-3} \cdot K^{-1}$ . Para el estudio en régimen estacionario el uso de  $U$ , arroja resultados satisfactorios. Sin embargo, no refleja el comportamiento dinámico cuando varía algún parámetro.

### 1.2.1.2 Modelos en estado estacionario.

Para el estado estacionario se aplican las ecuaciones de conservación a los flujos másicos de la fase sólida y de la gaseosa, obteniendo:

$$\dot{m}_{p,in} = \dot{m}_{p,out} + \dot{m}_{w,ev} \quad (13)$$

$$\dot{m}_{a,in} = \dot{m}_{a,out} - \dot{m}_{w,ev} \quad (14)$$

Para los balances de energía, los calores que se transfieren pueden cuantificarse de un modo básico siguiendo una serie de consideraciones. Suponemos que el aire de secado a la entrada del trómel está completamente seco (en la realidad tendrá un contenido de humedad muy pequeño) y que los calores específicos del aire, agua y subproducto son constantes, así como el calor latente de vaporización del agua. De ese modo podemos calcular el valor de cada uno de ellos:

-Calor que aporta el gas de secado (aire): que corresponde al decremento de temperatura sufrido.

$$\frac{\partial Q_G}{\partial t} = \dot{m}_{da} \cdot c_{P,da} \cdot (T_{a,in} - T_{a,out}) \quad (15)$$

-Calor que recibe el sólido (subproducto a secar): se invierte en elevar su temperatura.

$$\frac{\partial Q_p}{\partial t} = \dot{m}_{dp} \cdot c_{P,dp} \cdot (T_{p,out} - T_{p,in}) \quad (16)$$

-Calor que recibe el líquido (agua): se invierte en elevar su temperatura antes de que se vaporice.

$$\frac{\partial Q_L}{\partial t} = \dot{m}_{dp} \cdot X \cdot c_{P,w} \cdot (T_{p,out} - T_{p,in}) \quad (17)$$

-Calor para la evaporación: calor necesario para cambiar el estado del agua líquida que está contenida en el sólido a vapor, que pasa a añadirse a la corriente de aire.

$$\frac{\partial Q_V}{\partial t} = \dot{m}_{w,ev} \cdot \lambda \quad (18)$$

-Calor de recalentamiento: se invierte en elevar la temperatura del vapor.

$$\frac{\partial Q_H}{\partial t} = \dot{m}_{w,ev} \cdot c_{p,v} \cdot (T_{a,in} - T_{a,out}) \quad (19)$$

En estas ecuaciones  $\dot{m}$  ( $kg \cdot s^{-1}$ ) es el flujo másico,  $c_p$  ( $J \cdot kg^{-1} \cdot K^{-1}$ ) representa el calor específico a presión constante,  $T_a$  y  $T_p$  ( $K$ ) son las temperaturas del aire y subproducto respectivamente,  $X$  es el contenido de humedad del subproducto (base seca) y  $\lambda$  ( $J \cdot kg^{-1}$ ) es el calor latente de vaporización del agua.

Si las pérdidas por transmisión de calor hacia el exterior se desprecian, el balance energético global es:

$$\frac{\partial Q_G}{\partial t} = \frac{\partial Q_p}{\partial t} + \frac{\partial Q_L}{\partial t} + \frac{\partial Q_V}{\partial t} + \frac{\partial Q_H}{\partial t} \quad (20)$$

Por otro lado, si se considera el secadero como un intercambiador de calor, la cantidad de calor transferida por unidad de tiempo en la totalidad del secadero puede expresarse del modo:

$$\frac{\partial Q_G}{\partial t} = U \cdot V \cdot LMTD \quad (21)$$

siendo  $U$  el coeficiente volumétrico de transmisión de calor por unidad de tiempo ( $kW \cdot m^{-3} \cdot K^{-1}$ ) y cuyo valor se obtiene experimentalmente a partir de la Ec.(12),  $V$  es el volumen del secadero ( $m^3$ ), y  $LMTD$  ( $K$ ) es la diferencia de temperatura media logarítmica entre la temperatura del aire de secado y la del sólido. Si no se conocen estas temperaturas, se puede usar la media logarítmica entre las depresiones de bulbo húmedo del aire de secado a la entrada y salida del secadero.

### 1.2.1.3 Modelos en estado transitorio.

Este modelo consiste en aplicar las ecuaciones de conservación de la energía y la masa al secadero completo, para cada instante de tiempo. Los balances de masa de

subproducto y aire húmedo están relacionados por el flujo de agua evaporada. Con respecto a los balances de masa:

-En el subproducto seco, la variación de la masa seca retenida en el trómel es la diferencia entre los flujos de subproducto seco a la entrada y a la salida:

$$\frac{dm_{dp}}{dt} = \dot{m}_{dp,in} - \dot{m}_{dp,out} \quad (22)$$

-En el contenido de humedad del subproducto, la variación de la humedad contenida en el sólido es:

$$\frac{d(m_{dp}X)}{dt} = \dot{m}_{dp,in}X_{in} - \dot{m}_{dp,out}X_{out} - DR \cdot m_{dp} \quad (23)$$

El primer y segundo término en el lado derecho de la igualdad representan la diferencia del flujo de agua de entrada y salida (contenida en el subproducto). El tercer término indica el flujo de agua que es evaporado en cada instante de tiempo.  $X$  y  $DR$ , indican el contenido de humedad en el subproducto ( $kg_w/kg_{ds}$ ) y la tasa de secado ( $kg_w/kg_{ds} \cdot s$ ), respectivamente.

-En el contenido humedad del aire de secado, la variación de la humedad con el tiempo es:

$$\frac{d(m_{da}Y)}{dt} = \dot{m}_{da,in}Y_{in} - \dot{m}_{da,out}Y_{out} + DR \cdot m_{dp} \quad (24)$$

El primer y segundo término en el lado derecho de la igualdad representan la diferencia del flujo de agua de entrada y salida (contenida en el aire). El tercer término indica el flujo de agua que es evaporado en cada instante de tiempo.  $Y$  y  $DR$ , indican el contenido de humedad en el aire ( $kg_w/kg_{da}$ ) y la tasa de secado ( $kg_w/kg_{da} \cdot s$ ), respectivamente.

Estas ecuaciones pueden simplificarse como:

$$\frac{dX}{dt} = \frac{1}{m_{dp}} \left[ \dot{m}_{dp,in} X_{in} - \dot{m}_{dp,out} X_{out} - X \frac{dm_{dp}}{dt} \right] \quad (25)$$

$$\frac{dY}{dt} = \frac{1}{m_{da}} \left[ \dot{m}_{da,in} Y_{in} - \dot{m}_{da,out} Y_{out} - Y \frac{dm_{da}}{dt} \right]$$

Y las ecuaciones de conservación de la energía se expresan:

-En el subproducto, la variación de entalpía en el subproducto húmedo contenido en el trómel con el tiempo es igual a:

$$\frac{d(m_{dp} c_{P,p} T_p)}{dt} = \dot{m}_{dp,in} c_{P,p} T_{p,in} - \dot{m}_{dp,out} c_{P,p} T_{p,out} + U \cdot V (T_a - T_p) - DR \cdot m_p \lambda - DR \cdot m_p c_{P,v} (T_a - T_p) \quad (26)$$

donde el primer y segundo término a la derecha de la igualdad indican la diferencia de entalpía de los flujos de entrada y salida del subproducto húmedo. El tercer término es el calor transferido del aire al subproducto. El cuarto término representa el calor requerido para evaporar la humedad del subproducto y el quinto término es el calor requerido para calentar el vapor de agua hasta la temperatura del aire.

-En el aire de secado, la variación de la entalpía del aire húmedo contenido en el trómel con el tiempo es igual a:

$$\frac{d(m_{da} c_{P,a} T_a)}{dt} = \dot{m}_{da} c_{P,a} T_{a,in} - \dot{m}_{da} c_{P,a} T_{a,out} - U \cdot V \cdot LMTD + DR \cdot m_{dp} \lambda + DR \cdot m_{dp} c_{P,v} (T_a - T_p) \quad (27)$$

donde el primer y segundo término a la derecha de la igualdad indican la diferencia de entalpía de los flujos de entrada y salida del aire húmedo. El tercer término es el calor transferido del aire al subproducto. El cuarto término representa el calor necesario para evaporar el agua y el quinto término es la entalpía del agua evaporada del subproducto hasta llegar a la temperatura del aire.

### 1.2.2 Modelos divididos por secciones.

Debido a que el proceso de evaporación no es idéntico a lo largo de la longitud del trómel, su estudio puede realizarse dividiéndolo en pequeñas secciones. Se supondría así que el proceso de evaporación es idéntico a lo largo de una sección, y distinto entre diferentes secciones. Por tanto, sería necesario modelarlo en función de otros parámetros como: temperaturas del aire de secado, contenido de humedad del subproducto, tiempos de secado, tasa de secado, etc. Los flujos y temperaturas a la salida de una sección constituyen las temperaturas y flujos de entrada de la siguiente sección. En este tipo de modelo son válidas las ecuaciones expuestas anteriormente, considerando que la cantidad de agua que se evapora en una sección pasará a formar parte de la corriente gaseosa de la siguiente, aumentando, por tanto, su contenido de humedad. Supuestas las condiciones de alimentación del secadero, las demás secciones se resuelven consecutivamente.

Para poder realizar por completo los balances de masa y energía, se necesita conocer los tiempos de secado, la tasa de secado y el coeficiente volumétrico de transmisión de calor. Estos valores pueden evaluarse a partir del estudio de la cinética de secado del subproducto, de datos experimentales y de expresiones empíricas. Además, es necesario conocer o estimar el tiempo de residencia del material en el secadero, para determinar el avance del material de una sección a otra. El trómel es dividido en un número finito de elementos volumétricos situados en serie donde se aplican a cada elemento las ecuaciones de conservación. Cada elemento de volumen está limitado longitudinalmente por dos secciones, la de entrada ( $i-1$ ) y la de salida ( $i$ ). Conociendo las condiciones de entrada (alimentación), las demás pueden resolverse en serie. Las consideraciones para este modelo se detallan a continuación:

-Los calores específicos del aire y del sólido van cambiando, ya que varía la humedad de los mismos.

-El agua evaporada en una sección pasa al flujo gaseoso, interviniendo en las siguientes secciones. Igual ocurre con el calor que lleva asociado.

-La velocidad del gas cambia ya que su temperatura disminuye y su humedad aumenta.

-La tasa de secado varía a lo largo del modelo (en función de las humedades y temperaturas de sólido y aire, tamaño de muestra, etc.).

Aplicando el principio de conservación de la masa de aire seco:

$$\frac{\partial(m_{da})^{(i)}}{\partial t} = \dot{m}_{da}^{(i-1)} - \dot{m}_{da}^{(i)} \quad (28)$$

En la masa del subproducto seco:

$$\frac{\partial(m_{dp})^{(i)}}{\partial t} = \dot{m}_{dp}^{(i-1)} - \dot{m}_{dp}^{(i)} \quad (29)$$

En el agua contenida en el subproducto:

$$\frac{\partial(m_w)^{(i)}}{\partial t} = \dot{m}_{w,p}^{(i-1)} - \dot{m}_{w,p}^{(i)} - DR^{(i)} \cdot m_{dp}^{(i)} \quad (30)$$

En el vapor de agua contenido en el aire:

$$\frac{\partial(m_v)^{(i)}}{\partial t} = \dot{m}_v^{(i-1)} - \dot{m}_v^{(i)} + DR^{(i)} \cdot m_{da}^{(i)} \quad (31)$$

Aplicando la ecuación de la conservación de la energía en el subproducto se tiene:

$$\begin{aligned} \frac{\partial(c_{p,dp}^{(i)} \cdot m_{dp}^{(i)} + c_{p,w}^{(i)} \cdot m_w^{(i)}) \cdot T_p^{(i)}}{\partial t} = U^{(i)} \cdot V \cdot (T_a^{(i)} - T_p^{(i)}) + (c_{p,dp}^{(i)} \cdot m_{dp}^{(i)} + c_{p,w}^{(i)} \cdot m_w^{(i)}) \cdot T_p^{(i)} - \\ (c_{p,dp}^{(i-1)} \cdot m_{dp}^{(i-1)} + c_{p,w}^{(i-1)} \cdot m_w^{(i-1)}) \cdot T_p^{(i-1)} - DR^{(i)} \cdot m_{dp}^{(i)} \cdot (c_{p,w}^{(i)} \cdot T_p^{(i)} + \lambda) \end{aligned} \quad (32)$$

Aplicando la ecuación de la conservación de la energía en el gas de secado:

$$\begin{aligned} \frac{\partial(c_{p,v}^{(i)} \cdot m_v^{(i)} + c_{p,da}^{(i)} \cdot m_{da}^{(i)}) \cdot T_p^{(i)}}{\partial t} = -U^{(i)} \cdot V \cdot (T_a^{(i)} - T_p^{(i)}) + (c_{p,da}^{(i)} \cdot m_{da}^{(i)} + c_{p,v}^{(i)} \cdot m_v^{(i)}) \cdot T_a^{(i)} - \\ (c_{p,da}^{(i-1)} \cdot m_{da}^{(i-1)} + c_{p,v}^{(i-1)} \cdot m_v^{(i-1)}) \cdot T_a^{(i-1)} - DR^{(i)} \cdot m_{da}^{(i)} \cdot c_{p,v}^{(i)} (T_p^{(i)} - T_a^{(i)}) \end{aligned} \quad (33)$$

En las ecuaciones (32) y (33), el primer término a la derecha de la igualdad es positivo y negativo respectivamente, porque el aire caliente transfiere calor al subproducto.

### 1.3 NUEVOS ESTUDIOS SOBRE LA CINÉTICA DE SECADO DEL *ALPEORUJO*, *HUESO DE ACEITUNA* Y OTROS RESIDUOS BIOMÁSICOS.

Después de realizar una revisión de la cinética de secado de los subproductos de almazara e identificar los principales modelos matemáticos de transferencia de calor y masa en secaderos rotativos, nuevos estudios de la cinética para su aplicación posterior a secaderos rotativos han sido realizados en esta Memoria.

En primer lugar, se ha realizado un nuevo estudio de la cinética de secado en el *alpeorujo* a partir de tres parámetros fundamentales en secaderos rotativos: la temperatura del aire de secado, la velocidad del aire de secado y el tamaño de muestra en el interior de las aspas del trómel (Gómez-de la Cruz et al., 2014a). Hasta la fecha, los estudios sobre la cinética de secado tanto en el *orujo* como en el *alpeorujo* presentan limitaciones con respecto a la magnitud de las variables utilizadas. La mayoría de trabajos han sido realizados bajo unas condiciones de temperatura y velocidades muy bajas y por tanto, las variables obtenidas no reflejan las condiciones dadas a lo largo del secado en el trómel. Además, los modelos matemáticos de ajuste de la tasa de secado no reflejan fielmente las etapas de secado: *etapa de calentamiento*, *primera etapa de caída de la tasa de secado* y *segunda etapa de caída de la tasa de secado*, las cuales muestran los principales mecanismos de eliminación de humedad. Se pretende realizar un diseño de experimentos que abarque un amplio abanico de estas variables y que sean semejantes a las utilizadas en secaderos rotativos.

En segundo lugar, esta Tesis Doctoral ha abierto nuevas líneas de investigación destinadas a la revalorización del *hueso de aceituna* y otros residuos biomásicos en alza como los *posos de café* mediante el estudio de su cinética de secado, el cual permitirá obtener las mejores condiciones óptimas de su secado sin empeorar sus cualidades.

En los últimos años, la separación del *hueso de aceituna* en la propia almazara ha supuesto un aporte económico auxiliar debido a sus múltiples aplicaciones: carbón activo, cosméticos, abrasivos y principalmente como biocombustible (Pattara et al., 2010; Rodríguez et al., 2008). Con una producción media anual en Andalucía de 360,000 toneladas (Agencia Andaluza de la Energía (AAE), 2013), este subproducto es

usado en su gran mayoría para la producción de energía eléctrica y térmica (García-Maraver et al., 2012). Más del 90 % del *hueso de aceituna* es usado para la generación de energía térmica para propósitos industriales y para calefacción en edificios comerciales, residenciales y particulares (Lopez et al., 2010; Rosúa and Pasadas, 2012). Este subproducto se caracteriza por bajos contenidos de azufre y nitrógeno minimizando las emisiones de NO<sub>x</sub> y SO<sub>2</sub>, bajo contenido en cenizas y un poder calorífico de 19.2 MJ/kg (González et al., 2004; Mata-Sánchez et al., 2014; Moral and Méndez, 2006).

Sin embargo, para obtener un biocombustible óptimo y revalorizado, el hueso debe ser secado desde humedades en torno al 25 % hasta su humedad de equilibrio, entre el 8 % y 10 % (Gómez-de la Cruz et al., 2014b; Gómez-de la Cruz et al., 2015b). Disminuir su contenido de humedad implica mejores rendimientos en la combustión y una vida más larga en las calderas biomásicas ya que su principal inconveniente es la aparición de condensaciones (Gebreegziabher et al., 2013).

Para apoyar y afianzar las investigaciones sobre la cinética de secado de los subproductos originados en la extracción del aceite de oliva, se han realizado estudios de secado auxiliares con *posos de café*. Con una producción mundial de café de 8 millones de toneladas anuales (International Coffee Organization (ICO), 2014), los *posos de café* se están posicionando como uno de los recursos biomásicos más atractivos debido a la generación de residuos en la industria del café (Zuorro and Lavecchia, 2012). Cuatro tipos principales de subproductos biomásicos son obtenidos de los *posos de café*: biodiesel, bioetanol, bioaceites y combustible sólido peletizado (Kondamudi et al., 2008; Vardon et al., 2013). El biodiesel es producido usando métodos de transesterificación a partir del aceite contenido en los *posos de café*, alrededor del 15 % (Mussatto et al., 2011b). Los residuos sólidos desgrasados son utilizados para obtener bioetanol y bioaceites a partir de procesos como fermentación orgánica y pirólisis lenta, respectivamente. Los residuos sólidos restantes (sin lípidos y azúcares) son utilizados para la fabricación de pellets, los cuales son usados para generar energía eléctrica y térmica. Su poder calorífico inferior es de 25,240 kJ/kg a 0 % de contenido de humedad (Silva et al., 1998).

Los *posos de café* tienen un alto contenido de humedad, entre el 55 % y el 80 % dependiendo del proceso de fabricación usado. Generalmente, la industria del café soluble obtiene contenidos de humedad en los *posos de café* mayores que los obtenidos en el sector servicios. Por tanto, el proceso de secado se hace necesario para la posterior

fabricación de biocombustibles (Gómez-de la Cruz et al., 2015a). Primero, en el proceso de producción de biodiesel, bioetanol y bioaceites, la eliminación del contenido de humedad es esencial para que el disolvente sea más efectivo (Caetano et al., 2012; Caetano et al., 2013; Mussatto et al., 2011a). Y segundo, cuando los *posos de café* son directamente utilizados como pellets, deberían ser secados hasta su humedad de equilibrio con el fin de mejorar las características energéticas como biocombustible (Nebra et al., 2000; Sosa-Arno and Nebra, 2009). Además, el secado hace posible minimizar los costes de almacenaje, transporte y empaquetado.

## 2. OBJETIVOS.

Tras estudiar y analizar los principales modelos de transferencia de calor y masa en secaderos rotativos, se desprende que un análisis por secciones del trómel sería el modelo más adecuado para una futura investigación.

Por este motivo, el principal objetivo de esta Tesis Doctoral es estudiar las variables más importantes que se obtienen del estudio de la cinética de secado de subproductos de almazara y de otros residuos biomásicos para su posterior aplicación al modelado de secaderos rotativos. Estas variables se detallan a continuación:

-Obtención de las curvas de secado experimentales y aproximación matemática a partir de funciones empíricas. Esto permitirá obtener los tiempos necesarios de secado a partir de unas condiciones dadas entre secciones del trómel y definirá el tiempo de residencia entre secciones para obtener secados más efectivos.

-Cálculo de tasa de secado a partir de las curvas de secado experimentales y aproximación matemática de los resultados. La tasa de secado permitirá obtener la cantidad de agua evaporada entre secciones a partir de unas condiciones determinadas. Además, se pretende determinar y analizar las etapas de secado.

-Identificación de los valores de temperatura y humedad en los cuales aparecen tanto material volátil como la combustión de los subproductos. Ambas son variables muy importantes durante el secado en secaderos rotativos, ya que permiten mejorar la calidad de los subproductos, tanto en el aceite de *orujo* de oliva como el *orujillo* o *hueso de aceituna*.

-Cálculo y determinación de la difusividad de humedad efectiva a partir de funciones experimentales dependientes de la temperatura y humedad. Esta variable puede ser definida como una propiedad de transporte de humedad general en los subproductos de almazara, la cual explica los fenómenos de transferencia de masa y puede ser empleada en simulaciones numéricas o mediante técnicas de Mecánica de Fluidos Computacional (CFD) en el modelado de secaderos rotativos.

### 3. METODOLOGÍA.

#### 3.1 MATERIALES

Para la realización de los estudios de la cinética de secado se han utilizado muestras de *alpeorujo*, *hueso de aceituna* y *posos de café*.

Las muestras de *alpeorujo* fueron elaboradas a partir de *orujillo* seco y la adición de agua. El subproducto presentaba heterogeneidad macroscópica debido a diferentes tamaños de partículas. Para encontrar el contenido de humedad inicial, las muestras fueron secadas en un horno (Mettler GmbH + Co.KG, SNB 167 Model 100, Germany) a 105°C durante 24 horas. Un contenido medio de humedad inicial de  $60 \pm 5$  % (base húmeda) fue encontrado. El mismo procedimiento fue realizado para encontrar la humedad de equilibrio que fue estimada en  $8 \pm 1$  % (base húmeda).

El *hueso de aceituna* fue donado por varias almazaras de la provincia de Jaén y fueron recogidas durante la campaña de aceituna para asegurar el contenido de humedad original. Dos estudios han sido realizados con diferentes muestras. En el primer estudio, el *hueso de aceituna* presentaba aproximadamente un 30% en peso de pulpa, presentando un contenido medio de humedad inicial y de equilibrio de  $23 \pm 0.5$  % y  $8 \pm 0.5$  % (base húmeda), respectivamente. En el segundo estudio, el *hueso de aceituna* se presentó limpio (libre de pulpa) con un contenido medio de humedad inicial y de equilibrio de  $19 \pm 0.3$  % y  $7 \pm 0.5$  % (base húmeda), respectivamente. Además, en este último estudio se obtuvo la distribución de tamaño de partícula a partir de una cantidad de sólidos usando un tamiz vibratorio (Restch, Mod. Vibro). Los resultados mostraron que el *hueso de aceituna* tenía un tamaño de partícula entre 7 y 5 mm (9.7 %), 5-3 mm (43.1 %), 3-1 (36.9 %), y <1 mm (10.3 %). Aproximadamente, un tamaño medio de partícula de 3.1 mm fue encontrado.

Los *posos de café* fueron recogidos de varias cafeterías de la provincia de Jaén. Los ensayos fueron realizados inmediatamente después a su recogida para conservar la humedad inicial original. Para este residuo biomásico, se encontró una humedad media inicial y de equilibrio de  $58.5 \pm 1.5$  % y  $7 \pm 0.5$  % (base húmeda), respectivamente.

### 3.2 EQUIPO EXPERIMENTAL

Los experimentos fueron llevados a cabo en un túnel de secado experimental (figura 3.1). El equipo está formado por:

-Un ventilador centrífugo de media presión y simple aspiración, con caudal máximo de 475 m<sup>3</sup>/h. Su velocidad máxima de giro oscila entre las 2900 rpm a una potencia de 80 W. A este ventilador se le ha acoplado un tubo de PVC de 70 cm de longitud y con un diámetro de 10,4 cm para calcular la velocidad media de entrada del aire en el túnel. En su parte central se ha realizado un orificio para la introducción de un tubo de Pitot que obtenga el perfil de velocidades.

-Unas resistencias que calientan el aire de secado aspirado por el ventilador centrífugo. El modelo de las resistencias es T-MAX-L 45 de la empresa ELECTRICFOR. El modelo T-MAX-L 45 está compuesto por treinta resistencias dispuestas en tres fases: dos resistencias de 18 kW cada una y otra de 9 kW. En total puede desarrollar 45 kW.

-Un conducto que conduce el aire de secado. Tiene 2 m de longitud con una sección interior de 15 cm x 15 cm. Está dispuesto horizontalmente y está unido al conjunto de resistencias por una carcasa exterior. El conducto se ha aislado para evitar las pérdidas de calor. Al final del conducto se le ha practicado un orificio para introducir la varilla de sujeción de la cestilla a estudiar.



**Figura 3.1** Equipo experimental para la realización de los estudios de la cinética de secado.

La velocidad del aire del ventilador es controlada por un variador de frecuencia conectado al motor eléctrico. Para lograr la temperatura deseada en cada ensayo, el aire pasa por el grupo de resistencias y un controlador PID (proporcional-integral-derivativo) se encarga de actuar sobre las resistencias manteniendo una temperatura constante (ensayos isotérmicos). Para medir la temperatura se usa una sonda PT100 la cual está colocada justo antes de la posición de las muestras de estudio. Una vez alcanzadas las condiciones estacionarias para cada experimento, las muestras son introducidas (con el espesor de muestra correspondiente a cada ensayo) dentro del túnel en una cestilla metálica. La cesta es colocada sobre una balanza de precisión (Blauscal AH1200) con un error de aproximación de  $\pm 0.01 \cdot 10^{-3}$  kg. Así mismo, la balanza es conectada a un ordenador mediante puerto USB. El software diseñado para el equipo mide la variación de masa cada segundo y almacena la información de cada ensayo en archivos para su posterior tratamiento. Los experimentos son interrumpidos cuando el contenido de humedad se aproxima al de equilibrio o comienza la combustión de las muestras. Para obtener un contenido final exacto de humedad en la muestra, éstas son secadas hasta un 0 % de contenido de humedad en el horno durante 24 horas a 105°C. La figura 3.2 presenta el esquema del túnel de secado y su funcionamiento.

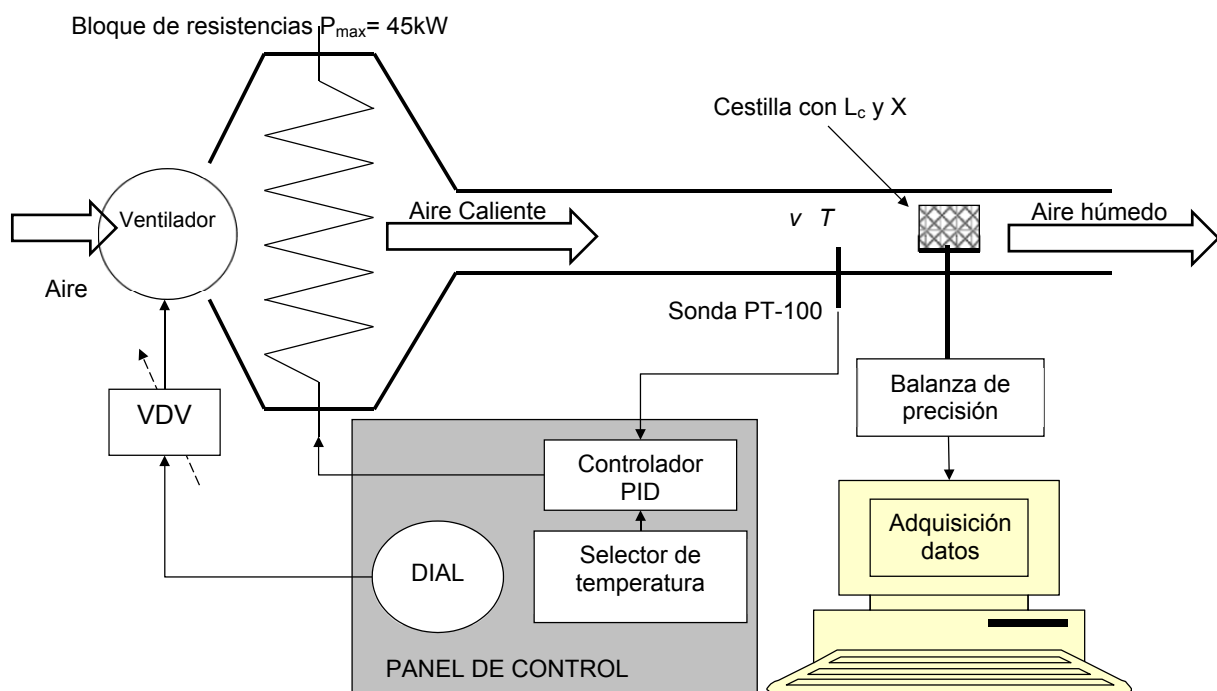


Figura 3.2 Esquema del proceso de trabajo en el túnel de secado.

### 3.3 DISEÑO EXPERIMENTAL.

Para el estudio de la cinética de secado en el *alpeorujo* se ha utilizado un diseño experimental compuesto central para tres variables: velocidad del aire de secado, temperatura del aire de secado y espesor de muestra (Wu and Hamada, 2009). Esta metodología está basada en los valores extremos, valores cúbicos y un valor central de estas tres variables (figura 3.3). Los valores extremos considerados en secaderos rotativos han sido:

- Temperatura del aire de secado: entre 100°C y 425°C.
- Velocidad del aire de secado: entre 1 m/s y 7 m/s.
- Espesor de muestra: entre 10 mm y 50 mm.

En total 15 experimentos de secado han sido propuestos. Con respecto a las temperaturas utilizadas, la máxima temperatura que se ha podido alcanzar en el túnel de secado ha sido de 425°C (acorde a temperaturas utilizadas en secaderos rotativos que utilizan gases de cogeneración para el secado), sin embargo las temperaturas alcanzadas en secaderos rotativos que utilizan el *orujillo* como combustible pueden llegar a duplicar esta temperatura. El máximo valor de la velocidad del aire no excede de los 7 m/s, teniendo en cuenta que varía a lo largo del trómel, mientras que los espesores de muestra utilizados están ligados a la cantidad de subproducto que voltean las aspás del trómel. Algunos trabajos en el secado de subproductos de almazara han tenido en cuenta la granulometría de las partículas, pero ya que su control dentro de los secaderos rotativos se hace muy difícil, solo tres parámetros han sido estudiados en este trabajo. La tabla 3.1 muestra los parámetros de secado para cada test.

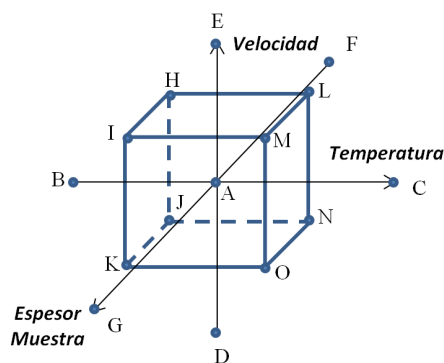


Figura 3.3 Diseño compuesto central para tres variables: velocidad, temperatura y espesor de muestra.

**Tabla 3.1** Ensayos de secado realizados en el túnel de secado para el *alpeorujo*.

TEST	Temperatura (°C)	Velocidad (m/s)	Espesor de Muestra (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

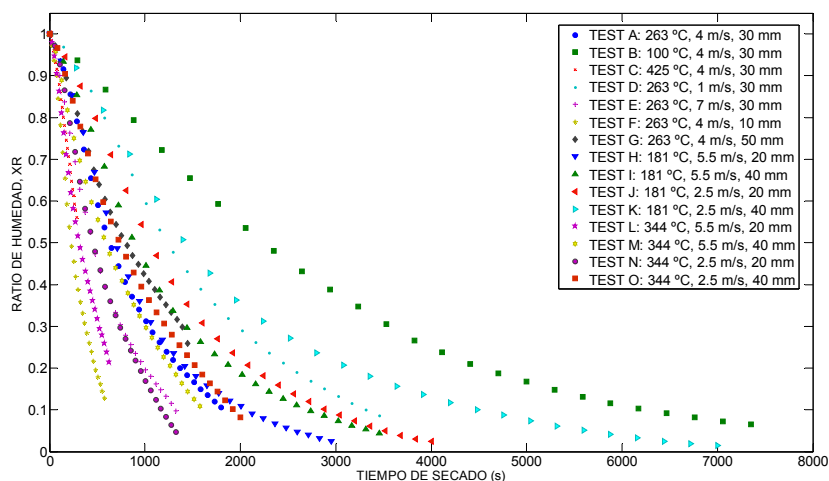
En el diseño experimental utilizado para el estudio de la cinética de secado en el *hueso de aceituna* se ha considerado un rango de temperaturas entre 100°C y 250°C, y un rango de espesores de muestra entre 10 mm y 30 mm. La velocidad del aire de secado propuesta ha sido de 1 m/s. Para cada espesor de muestra: 10 mm, 20 mm y 30 mm, cuatro ensayos con temperaturas constantes de 100°C, 150°C, 200°C y 250°C han sido realizados, en total 12 experimentos. Como puede observarse, estas condiciones son menos agresivas que las que se utilizan en el secado del *alpeorujo*. Esto se debe a la baja humedad que contiene el *hueso de aceituna* y a las posibles partículas más propicias para la combustión como finos o pulpa de aceituna.

Finalmente el diseño experimental propuesto para el secado de *posos de café* ha sido muy similar al propuesto para el *hueso de aceituna*. Las partículas de este residuo presentan tamaños microscópicos, y el uso de altas temperaturas y velocidades puede provocar un secado incorrecto y peligroso. Así, 16 experimentos fueron realizados. Para cada espesor de muestra: 5 mm, 10 mm, 15 mm y 20 mm, cuatro experimentos a 100°C, 150°C, 200°C y 250°C fueron llevados a cabo. La velocidad del aire de secado también se estableció en 1 m/s.

## 4. RESULTADOS Y DISCUSIÓN.

### 4.1 CURVAS DE SECADO.

La variable experimental ratio de humedad,  $XR = X_t/X_0$ , puede ser graficada como una función del tiempo. La [figura 4.1](#) muestra las curvas de secado en los experimentos realizados en el *alpeorujo*. Como puede observarse, conforme el tiempo de secado avanza, el ratio de humedad en cada experimento disminuye. Conforme las temperaturas y velocidades del aire de secado aumentan y los espesores de muestra disminuyen, los tiempos de secado disminuyen considerablemente. Sin embargo, cuando estas magnitudes son bajas, el tiempo necesario para llevar la humedad inicial hasta la de equilibrio aumenta.



**Figura 4.1** Curvas de secado en los ensayos realizados en el *alpeorujo*.

Durante la realización de los experimentos, algunas muestras comenzaron a arder a medida que la humedad decrecía. Las muestras en las que la aparición de material volátil y el inicio de la combustión se hicieron presentes pertenecen al “ensayo C” (425°C, 4 m/s, 30 mm), “ensayo E” (263°C, 7 m/s, 30 mm), “ensayo G” (263°C, 4 m/s, 50 mm) y “ensayo M” (344°C, 5.5 m/s, 40 mm). Por tanto, estos ensayos fueron detenidos en el momento del inicio de estos fenómenos.

Estos resultados indican que el riesgo de combustión de la muestra es más patente cuanto mayor es la temperatura, velocidad y tamaño de la muestra. Sin embargo, existe un parámetro esencial para que la combustión pueda ocurrir: la temperatura. Ésta

debe ser superior a 250°C. La [tabla 4.1](#) indica la humedad de parada en cada uno de los ensayos que presentaron combustión en la muestra de *alpeorujo* a secar.

**Tabla 4.1** Ensayos que presentan combustión.

ENSAYO	$T$ (°C)	$v$ (m/s)	$L_c$ (mm)	Humedad final de parada (Base húmeda)
C	425	4	30	38.62 %
E	263	7	30	10.87 %
G	263	4	50	21.53 %
M	344	5.5	40	14.9 %

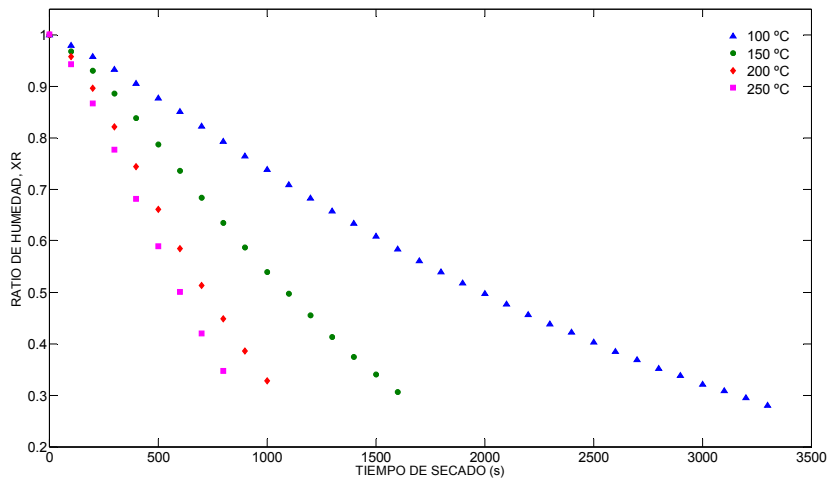
Como se observa, la muestra del “ensayo C” es retirada del túnel de secado cuando su humedad está alrededor del 40 %. Por tanto, no se obtienen resultados sobre el fenómeno del secado por debajo de esta humedad. Sin embargo, los resultados obtenidos en ese intervalo de humedad (61 % - 38.62 %) son suficientes para ser extrapolados al trómel de un secadero rotativo, ya que difícilmente se da una temperatura de 425°C a una humedad del 40 % dentro del mismo (la temperatura tiende a disminuir rápidamente a la entrada del trómel).

En el “ensayo E” el parámetro principal que desencadena la combustión de la muestra es la velocidad del aire, 7 m/s (velocidad máxima de los ensayos). Esta combustión es producida a una humedad aproximada del 11 %.

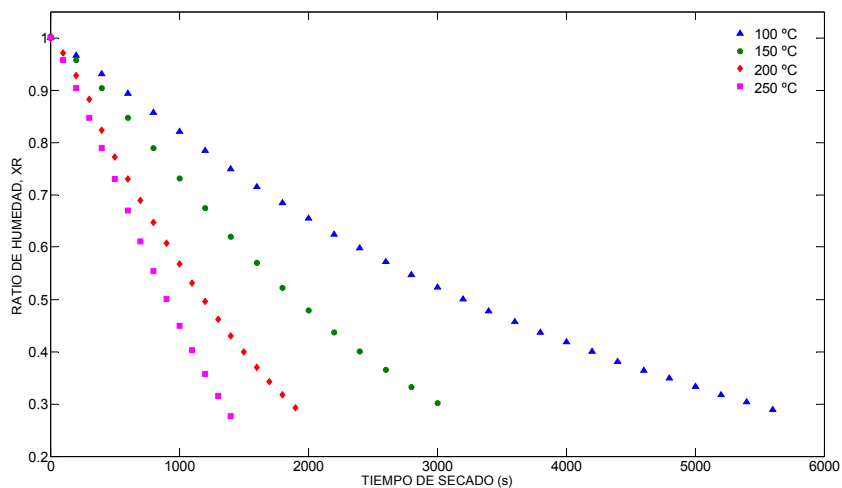
En el “ensayo G” aunque la velocidad es moderada, el tamaño de la muestra es considerable, 50 mm, comenzando a arder en torno al 20 % de humedad. Por último, en el “ensayo M”, el detonante de la combustión es una situación conjunta entre temperatura de secado, velocidad del aire y tamaño de la muestra.

Las [figuras 4.2, 4.3 y 4.4](#) muestran las curvas de secado para el *hueso de aceituna*. En ellas se puede ver que el tiempo de secado disminuye conforme la temperatura de secado aumenta. Por otro lado, el tiempo de secado se incrementa conforme lo hace el espesor de muestra. El tiempo de secado cae aproximadamente cuatro veces cuando la temperatura de secado se eleva de 100°C a 250°C, independientemente del espesor de muestra analizado. En los experimentos realizados en el *hueso de aceituna* con un 30 % de pulpa a 250°C, cuando la humedad de equilibrio fue alcanzada, la superficie exterior de la muestra comenzó a arder, independientemente del espesor de la muestra. Previamente, se apreció una pérdida de material volátil

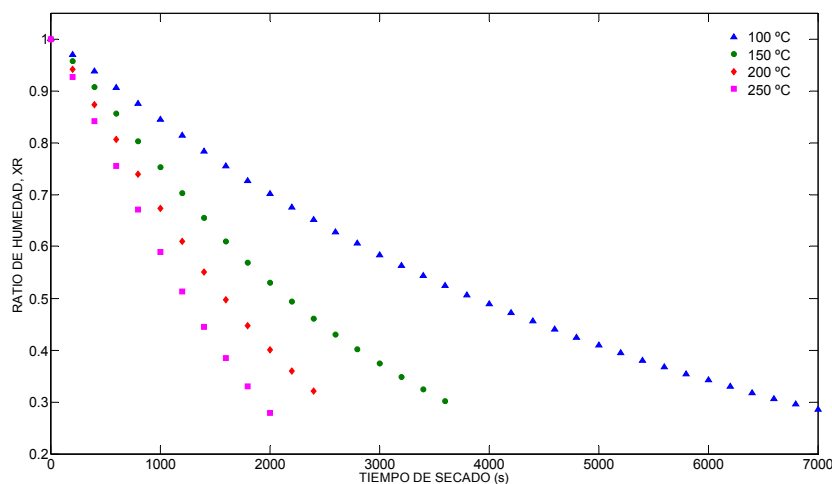
significativa. Este fenómeno alerta de la importancia en el control de las temperaturas del aire de secado, las cuales provocan que las características energéticas de este biocombustible empeoren. Sin embargo, cuando los estudios fueron realizados con *hueso de aceituna* limpio, la combustión no apareció. Esto implica que el riesgo de combustión durante el secado de *hueso de aceituna* está íntimamente ligado al porcentaje de finos y pulpa.



**Figura 4.2** Curvas de secado en los ensayos realizados en el *hueso de aceituna* para un espesor de muestra de 10 mm.

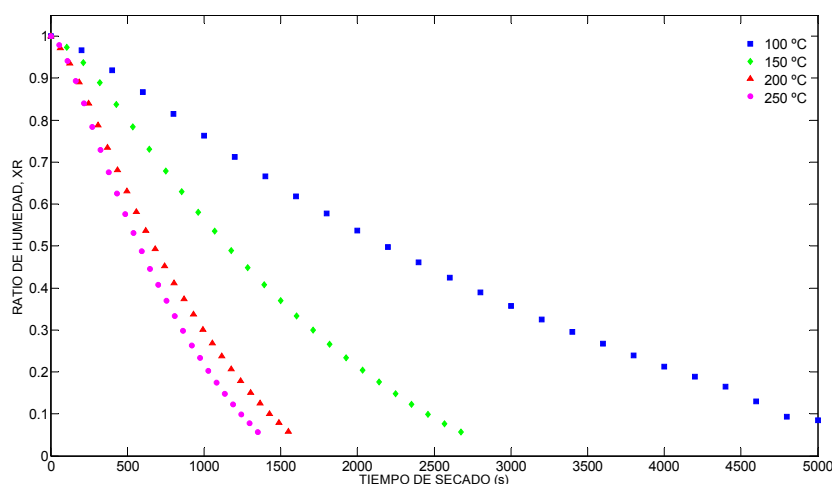


**Figura 4.3** Curvas de secado en los ensayos realizados en el *hueso de aceituna* para un espesor de muestra de 20 mm.

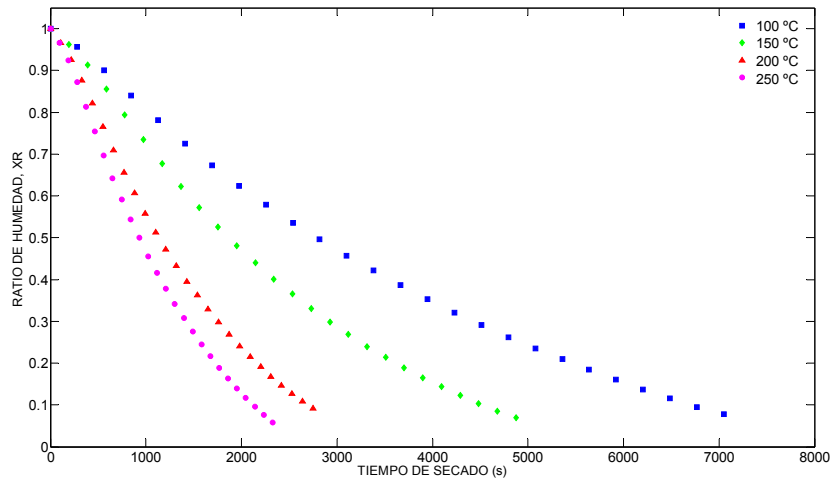


**Figura 4.4** Curvas de secado en los ensayos realizados en el *hueso de aceituna* para un espesor de muestra de 30 mm.

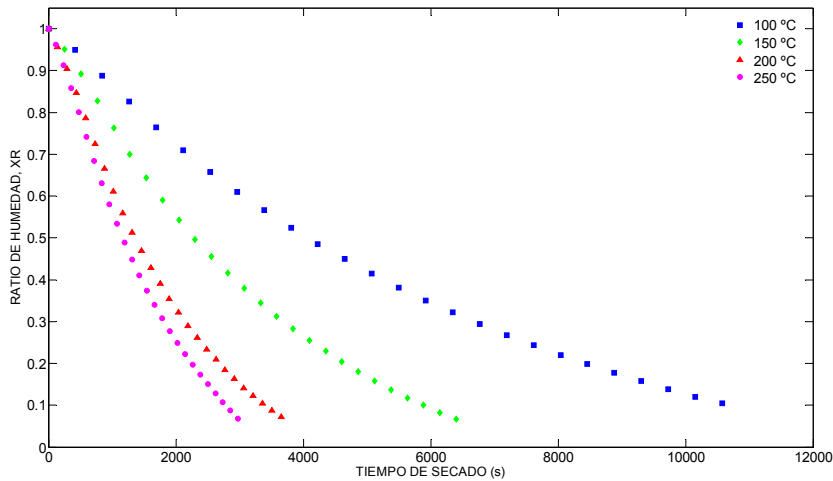
Similares resultados son obtenidos durante el secado de los *posos de café*. El secado es más rápido cuando la temperatura se eleva y el espesor de muestra disminuye. Además, una vez más se corrobora la aparición de la combustión en residuos biomásicos en los ensayos realizados a 250°C justo antes de que el proceso de secado haya acabado, es decir, antes de que se alcance el contenido de humedad de equilibrio. Así mismo, los ensayos son interrumpidos para no mostrar la pérdida de masa causada por el inicio de la combustión. Para temperaturas  $\leq 200^{\circ}\text{C}$ , la pérdida de volátiles no fue apreciada independientemente del contenido de humedad. Las figuras 4.5, 4.6, 4.7 y 4.8 muestran las curvas de secado de los *posos de café*.



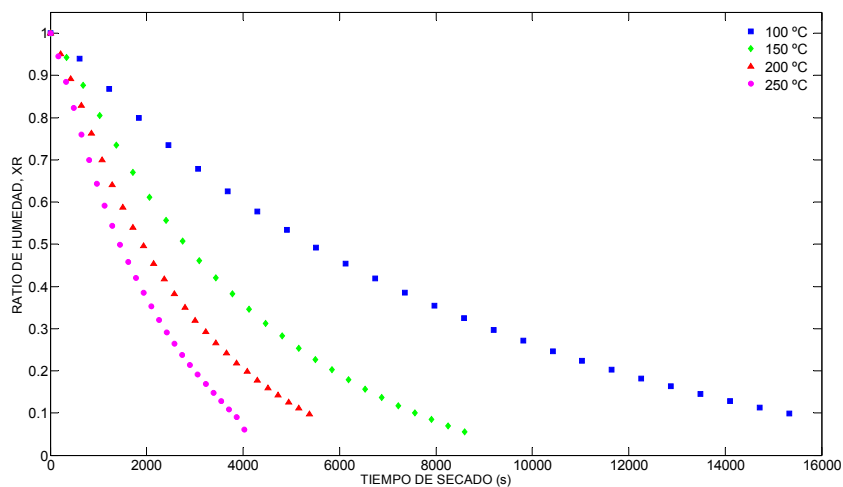
**Figura 4.5** Curvas de secado en los ensayos realizados en los *posos de café* para un espesor de muestra de 5 mm.



**Figura 4.6** Curvas de secado en los ensayos realizados en los *posos de café* para un espesor de muestra de 10 mm.



**Figura 4.7** Curvas de secado en los ensayos realizados en los *posos de café* para un espesor de muestra de 15 mm.



**Figura 4.8** Curvas de secado en los ensayos realizados en los *posos de café* para un espesor de muestra de 20 mm.

Hasta la fecha, no existen modelos matemáticos físicos que describan el comportamiento en el secado de capa delgada. El secado es un proceso complejo en los que intervienen conjuntamente fenómenos de convección y difusión, además de otros fenómenos significativos como capilaridad, flujos secuenciales de vaporización-condensación, etc. Sin embargo, es muy importante modelar eficazmente las etapas durante el proceso de secado. Para ello, las curvas de secado son ajustadas con funciones matemáticas semi-teóricas, semi-empíricas y empíricas ( $XR = f(t)$ ). Los modelos propuestos en la [tabla 1.3](#) son utilizados para aproximar las curvas de secado mediante análisis de regresión no lineal. Para verificar la calidad del ajuste, se han utilizado los criterios del coeficiente de determinación ([Ec. \(34\)](#)),  $R^2$ , y la raíz cuadrada del error cuadrático medio [Ec. \(35\)](#),  $RMSE$ . En esta Memoria, un nuevo modelo matemático empírico ha sido propuesto, el modelo gaussiano de doble término (*Two Term Gaussian*), el cual ha presentado los mejores resultados de ajuste en todos los experimentos realizados.

$$R^2 = \sum_{i=1}^N \frac{(XR_{cal,i} - \overline{XR_{exp,i}})^2}{(\overline{XR_{exp,i}} - \overline{XR_{exp,i}})^2} \quad (34)$$

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^N (XR_{exp,i} - XR_{cal,i})^2} \quad (35)$$

donde  $N$  es el número de datos y los subíndices *exp* y *cal* significan experimental y calculada, respectivamente. Las [tablas 4.2, 4.3 y 4.4](#) muestran los valores de  $R^2$  y  $RMSE$  de los diferentes modelos matemáticos de capa delgada para el *alpeorajo*, *hueso de aceituna* y *posos de café*, respectivamente. El modelo empírico *Two Term Gaussian* presenta unos valores de  $R^2$  prácticamente iguales a la unidad, con unos valores de  $RMSE$  muy bajos. Por tanto, puede decirse que esta función matemática aproxima de una forma excelente el comportamiento de secado desde la humedad inicial hasta la de equilibrio.

**Tabla 4.2** Criterios estadísticos,  $R^2$  y  $RMSE$ , para evaluar el ajuste de los modelos matemáticos de capa delgada en cada experimento para el *alpeorujo*.

TEST	MODELOS MATEMÁTICOS									
	Lewis		Page		Modified Page		Henderson and Pabis		Logarithmic	
	$R^2$	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	0,017	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
TEST	MODELOS MATEMÁTICOS									
	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	<b>1,0000</b>	<b>0,0011</b>
B	0,9993	0,0073	0,9999	0,0024	0,9999	0,0025	0,9999	0,0023	<b>0,9999</b>	<b>0,0022</b>
C	0,9983	0,0056	0,9999	0,0014	0,9999	0,0014	0,9999	0,0014	<b>0,9999</b>	<b>0,0015</b>
D	0,9965	0,0168	0,9968	0,0161	0,9996	0,0054	0,9992	0,0082	<b>1,0000</b>	<b>0,0012</b>
E	0,9972	0,0142	0,9985	0,0103	0,9996	0,0054	0,9991	0,0078	<b>0,9999</b>	<b>0,0024</b>
F	0,9963	0,0167	0,9955	0,0184	0,9991	0,0084	0,9981	0,0119	<b>0,9999</b>	<b>0,0026</b>
G	0,9982	0,0092	0,9981	0,0094	0,9988	0,0075	0,9988	0,0076	<b>0,9998</b>	<b>0,0032</b>
H	0,9904	0,0278	0,9973	0,0148	0,9995	0,0064	0,9989	0,0096	<b>1,0000</b>	<b>0,0017</b>
I	0,9925	0,0242	0,9977	0,0133	0,9995	0,0064	0,9990	0,0090	<b>1,0000</b>	<b>0,0014</b>
J	0,9951	0,0205	0,9971	0,0157	0,9996	0,0057	0,9992	0,0084	<b>1,0000</b>	<b>0,0010</b>
K	0,9837	0,0352	0,9982	0,0118	0,9993	0,0075	0,9986	0,0103	<b>1,0000</b>	<b>0,0012</b>
L	0,9979	0,0109	0,9809	0,0333	0,9993	0,0062	0,9988	0,0084	<b>0,9999</b>	<b>0,0027</b>
M	0,9975	0,0128	0,9998	0,0038	0,9987	0,0094	0,9987	0,0091	<b>0,9999</b>	<b>0,0023</b>
N	0,9969	0,0160	0,9977	0,0137	0,9979	0,0132	0,9982	0,0123	<b>0,9999</b>	<b>0,0026</b>
O	0,9981	0,0116	0,9993	0,0070	0,9985	0,0104	0,9989	0,0088	<b>0,9999</b>	<b>0,0022</b>

**Tabla 4.3** Criterios estadísticos,  $R^2$  y  $RMSE$ , para evaluar el ajuste de los modelos matemáticos de capa delgada en cada experimento para el *hueso de aceituna*.

Modelo matemático	T (°C)	Espesor de la muestra					
		$L_c = 10\text{ mm}$		$L_c = 20\text{ mm}$		$L_c = 30\text{ mm}$	
		$R^2$	RMSE	$R^2$	RMSE	$R^2$	RMSE
Lewis	100	0.987114	0.024637	0.998546	0.008	0.999742	0.003308
	150	0.965110	0.040503	0.983932	0.027523	0.993971	0.016644
	200	0.95409	0.04765	0.990925	0.019756	0.986919	0.024244
	250	0.950947	0.049019	0.968336	0.039168	0.980718	0.030844
Page	100	0.999949	0.001548	0.999859	0.002489	0.999867	0.002376
	150	0.99993	0.001803	0.999929	0.001826	0.999726	0.003545
	200	0.99995	0.001572	0.998141	0.008944	0.999962	0.001306
	250	0.999844	0.002763	0.999742	0.003535	0.999897	0.002248
M. Page	100	0.999949	0.001548	0.999859	0.002489	0.999867	0.002376
	150	0.99993	0.001803	0.999929	0.001826	0.999726	0.003545
	200	0.99995	0.001572	0.998141	0.008944	0.999962	0.001306
	250	0.999844	0.002763	0.999742	0.003535	0.999897	0.002248
Henderson and Pabis	100	0.995672	0.014279	0.999735	0.003416	0.999941	0.001573
	150	0.985325	0.026275	0.994617	0.015933	0.998453	0.008431
	200	0.981012	0.030658	0.993280	0.017004	0.995003	0.014987
	250	0.977943	0.032888	0.985979	0.026073	0.991982	0.019894
Logarithmic	100	0.999317	0.005671	0.999851	0.002559	0.999948	0.001484
	150	0.998574	0.008192	0.999060	0.006657	0.999357	0.005436
	200	0.998129	0.009626	0.999484	0.004711	0.999431	0.005056
	250	0.998166	0.009486	0.999116	0.006547	0.999297	0.005891
Wang and Singh	100	0.999569	0.004506	0.999816	0.002842	0.999654	0.003834
	150	0.99864	0.008	0.999355	0.005516	0.999701	0.003706
	200	0.998162	0.009541	0.999609	0.004102	0.999643	0.004003
	250	0.998174	0.009468	0.999186	0.006284	0.999497	0.004979
Two term	100	0.992431	0.018891	0.999984	0.000831	0.999948	0.001481
	150	0.999952	0.001495	0.989663	0.022086	0.996754	0.012216
	200	0.966741	0.040613	0.999713	0.00351	0.984757	0.026187
	250	0.959972	0.044355	0.999816	0.002982	0.990431	0.021744
Approach of Diffusion	100	0.999942	0.001650	0.999984	0.000835	0.999954	0.001388
	150	0.999882	0.00235	0.999976	0.001041	0.999979	0.000979
	200	0.999741	0.003579	0.998914	0.006835	0.999983	0.000871
	250	0.999349	0.005648	0.999663	0.004038	0.99996	0.0014
Midilli et al.	100	0.999962	0.001332	0.999954	0.001414	0.999958	0.001323
	150	0.999969	0.0012	0.999975	0.001085	0.999958	0.001389
	200	0.999953	0.001516	0.999539	0.00445	0.999975	0.001055
	250	0.999945	0.001635	0.999869	0.002518	0.99995	0.001565
Two term Gaussian	100	<b>0.999985</b>	<b>0.000823</b>	<b>0.999991</b>	<b>0.000616</b>	<b>0.999996</b>	<b>0.000366</b>
	150	<b>0.999986</b>	<b>0.000799</b>	<b>0.999993</b>	<b>0.000581</b>	<b>0.999987</b>	<b>0.000757</b>
	200	<b>0.999982</b>	<b>0.000942</b>	<b>0.999858</b>	<b>0.002472</b>	<b>0.999992</b>	<b>0.000625</b>
	250	<b>0.999989</b>	<b>0.000719</b>	<b>0.999992</b>	<b>0.00064</b>	<b>0.999999</b>	<b>0.000704</b>

**Tabla 4.4** Criterios estadísticos,  $R^2$  y  $RMSE$ , para evaluar el ajuste de los modelos matemáticos de capa delgada en cada experimento para los *posos de café*.

Modelo matemático	T (°C)	Espesor de la muestra							
		$L_c = 5\text{ mm}$		$L_c = 10\text{ mm}$		$L_c = 15\text{ mm}$		$L_c = 20\text{ mm}$	
		$R^2$	RMSE	$R^2$	RMSE	$R^2$	RMSE	$R^2$	RMSE
Lewis	100	0.971925	0.045390	0.982064	0.035993	0.992241	0.022717	0.996713	0.014620
	150	0.952396	0.063247	0.976087	0.043227	0.985963	0.032333	0.989783	0.027602
	200	0.951738	0.063585	0.971853	0.046357	0.980330	0.038849	0.992132	0.023431
	250	0.952856	0.063032	0.965480	0.053302	0.975165	0.043956	0.989064	0.027931
Page	100	0.997339	0.013974	0.997937	0.012205	0.999137	0.007573	0.999286	0.006813
	150	0.998301	0.011950	0.999255	0.007630	0.999063	0.008350	0.999166	0.007882
	200	0.998247	0.012122	0.999645	0.005203	0.999504	0.006164	0.999705	0.004537
	250	0.998414	0.011563	0.998779	0.010023	0.999030	0.008686	0.998954	0.008638
Modified Page	100	0.997339	0.013974	0.997937	0.012205	0.999137	0.007573	0.999286	0.006813
	150	0.998301	0.011950	0.999255	0.007630	0.999063	0.008350	0.999166	0.007882
	200	0.998247	0.012122	0.999645	0.005203	0.999504	0.006164	0.999705	0.004537
	250	0.998414	0.011563	0.998779	0.010023	0.999030	0.008686	0.998954	0.008638
Henderson and Pabis	100	0.984942	0.033244	0.990248	0.026541	0.996177	0.015945	0.998190	0.010848
	150	0.975731	0.045167	0.989223	0.029022	0.993427	0.022125	0.995207	0.018907
	200	0.975236	0.045562	0.987885	0.030418	0.991239	0.025930	0.997404	0.013460
	250	0.977075	0.043971	0.983326	0.037052	0.988492	0.029927	0.994372	0.020039
Logarithmic	100	0.999622	0.005264	0.999875	0.002996	0.999907	0.002482	0.999872	0.002885
	150	0.998749	0.010253	0.999231	0.007753	0.999705	0.004685	0.999786	0.003990
	200	0.998662	0.010591	0.998638	0.010199	0.999204	0.007814	0.999526	0.005752
	250	0.998738	0.010317	0.998937	0.009356	0.999311	0.007321	0.999757	0.004163
Wang and Singh	100	0.999542	0.005792	0.999541	0.005752	0.999167	0.007440	0.998270	0.010608
	150	0.999183	0.008287	0.999523	0.006100	0.999201	0.007712	0.998636	0.010084
	200	0.999072	0.008822	0.999258	0.007527	0.999493	0.006238	0.998898	0.008767
	250	0.999159	0.008421	0.999408	0.006979	0.999501	0.006227	0.999046	0.008248
Two term	100	0.902333	0.084684	0.999876	0.002991	0.999917	0.002339	0.999948	0.001830
	150	0.998844	0.009857	0.999309	0.007346	0.999735	0.004440	0.999797	0.003883
	200	0.998752	0.010232	0.998774	0.009677	0.999325	0.007197	0.999575	0.005445
	250	0.998791	0.010097	0.999056	0.008816	0.999370	0.007002	0.999758	0.004151
Approach of Diffusion	100	0.996704	0.015552	0.997821	0.012546	0.999240	0.007106	0.999423	0.006125
	150	0.996036	0.018252	0.998881	0.009348	0.998916	0.008983	0.999122	0.008090
	200	0.995971	0.018377	0.999341	0.007093	0.999309	0.007281	0.999594	0.005321
	250	0.996420	0.017374	0.997763	0.013570	0.998606	0.010412	0.998997	0.008458
Midilli et al.	100	0.999719	0.004535	0.999906	0.002602	0.999922	0.002270	0.999666	0.004661
	150	0.999918	0.002619	0.999871	0.003164	0.999861	0.003216	0.999878	0.003009
	200	0.999868	0.003324	0.999858	0.003288	0.999882	0.002998	0.999741	0.004248
	250	0.999842	0.003643	0.999854	0.003466	0.999847	0.003441	0.999837	0.003408
Two term Gaussian	100	0.999785	0.003969	0.999962	0.001650	0.999980	0.001134	0.999983	0.001032
	150	0.999966	0.001691	0.999981	0.001215	0.999976	0.001325	0.999979	0.001245
	200	0.999975	0.001436	0.999990	0.000835	0.999992	0.000765	0.999992	0.000718
	250	0.999967	0.001651	0.999966	0.001654	0.999973	0.001442	0.999971	0.001427

## 4.2 TASA DE SECADO.

La tasa de secado indica la cantidad de agua evaporada por unidad de tiempo. Esta variable es muy importante para optimizar y controlar el proceso de secado en cualquier tipo de secadero. La tasa de secado puede ser calculada experimentalmente como indica la Ec. (2). La figura 4.9 muestra la tasa de secado experimental para cada uno de los ensayos realizados en el *alpeorujo*. Como se aprecia, la tasa de secado es mayor conforme aumenta la temperatura y la velocidad del aire de secado, mientras que disminuye cuando el espesor de la muestra aumenta.

La tasa de secado puede ser graficada con respecto al tiempo, pero el tiempo es normalmente reemplazado por el ratio de humedad. De este modo, la dependencia temporal puede ser eliminada y la tasa de secado puede ser expresada como una función del contenido de humedad. Las curvas de la tasa de secado pueden ser ajustadas a partir de la derivada con respecto al tiempo de los modelos matemáticos de la tabla 1.3 (Ec. (3)). Sin embargo, aunque la mayoría de los modelos matemáticos de ajuste de las curvas de secado presentan buenos resultados, cuando la derivada de las funciones son obtenidas, los errores cometidos pueden ser considerables. La figura 4.10 muestra la tasa de secado experimental y los cuatro mejores modelos de ajuste frente al ratio de humedad para un ensayo en el *hueso de aceituna* a 100°C y 10 mm de espesor. Como se puede apreciar, el modelo que ofreció el mejor resultado de ajuste en las curvas de secado presentó también el mejor ajuste en las curvas de la tasa de secado, el modelo *Two Term Gaussian*.

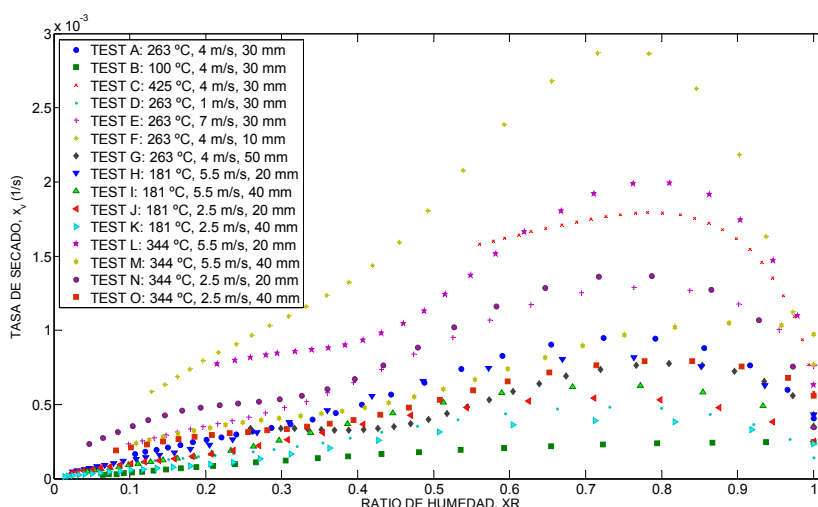
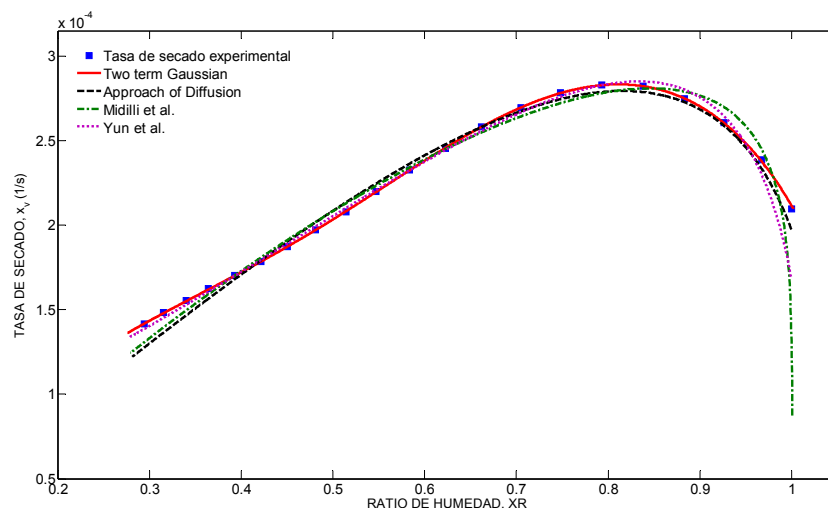
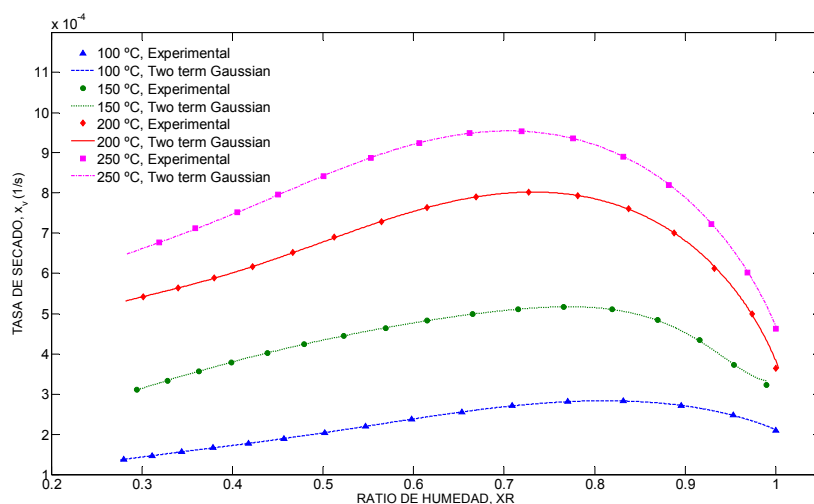


Figura 4.9 Tasa de secado frente al ratio de humedad en los ensayos realizados en el *alpeorujo*.

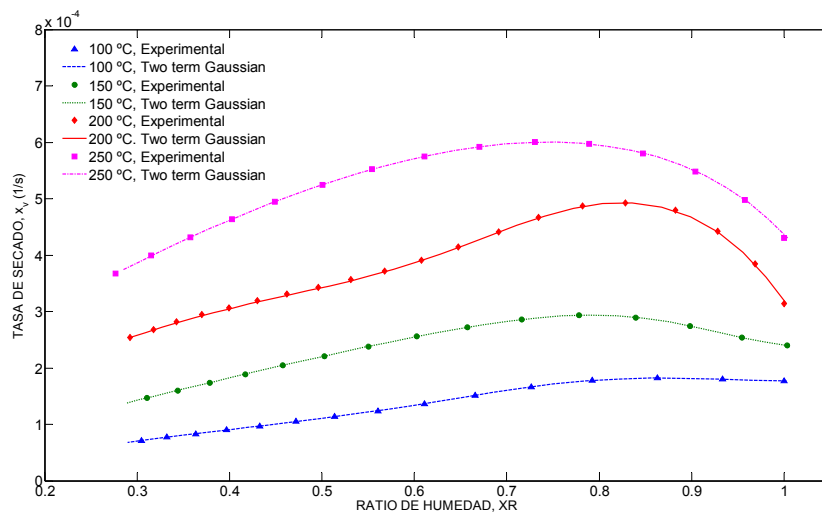


**Figura 4.10** Tasa de secado experimental y los cuatro mejores modelos de capa delgada de ajuste para un ensayo realizado a 10 mm de espesor y 100°C en el *hueso de aceituna*.

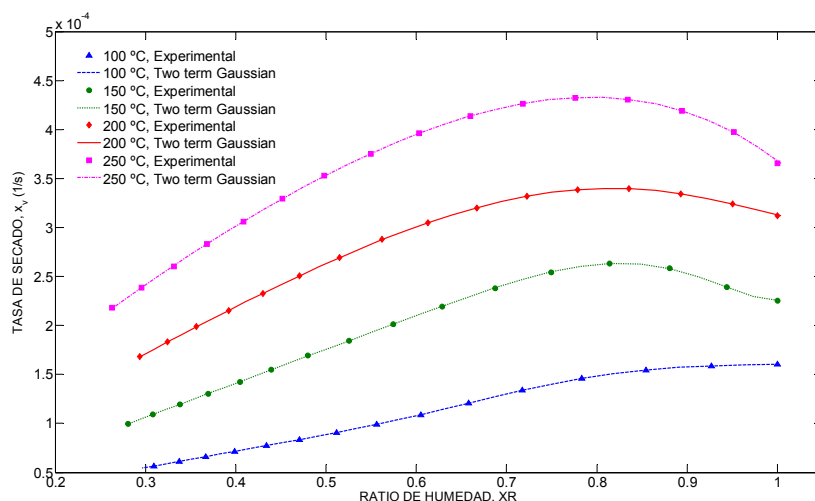
Las figuras 4.11, 4.12 y 4.13 muestran las curvas de la tasa de secado experimental y sus aproximaciones con el modelo *Two Term Gaussian* para cada espesor de muestra: 10 mm, 20 mm y 30 mm, respectivamente. Los resultados muestran que la tasa de secado está íntimamente ligada a la temperatura de secado y al espesor de muestra. Existen altas tasas de secado cuando la temperatura de secado se incrementa y el espesor de muestra decrece.



**Figura 4.11** Tasa de secado frente al ratio de humedad para 10 mm de espesor a diferentes temperaturas con velocidad constante,  $v = 1 \text{ m/s}$ , en el *hueso de aceituna*.

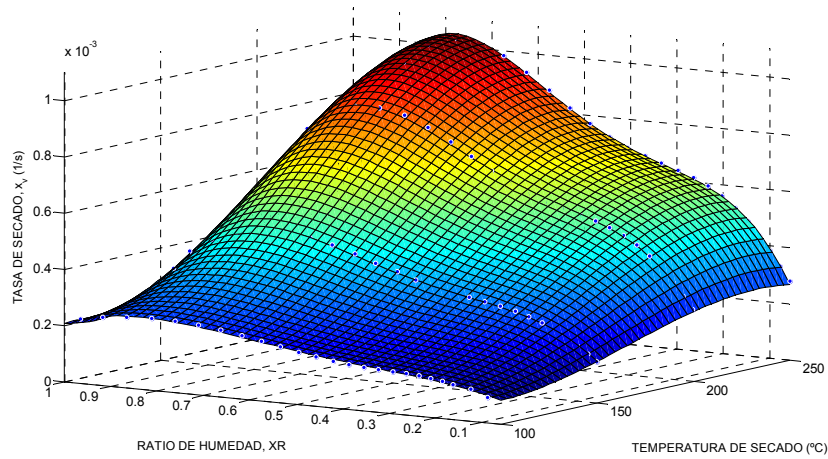


**Figura 4.12** Tasa de secado frente al ratio de humedad para 20 mm de espesor a diferentes temperaturas con velocidad constante,  $v = 1 \text{ m/s}$ , en el *hueso de aceituna*.

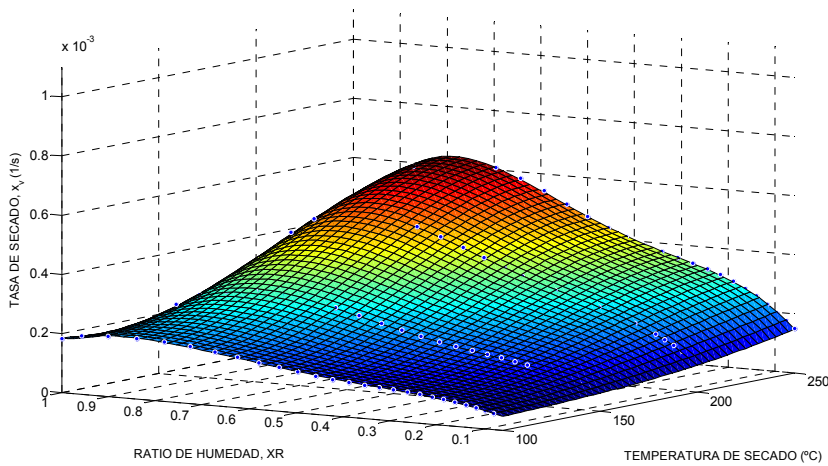


**Figura 4.13** Tasa de secado frente al ratio de humedad para 30 mm de espesor a diferentes temperaturas con velocidad constante,  $v = 1 \text{ m/s}$ , en el *hueso de aceituna*.

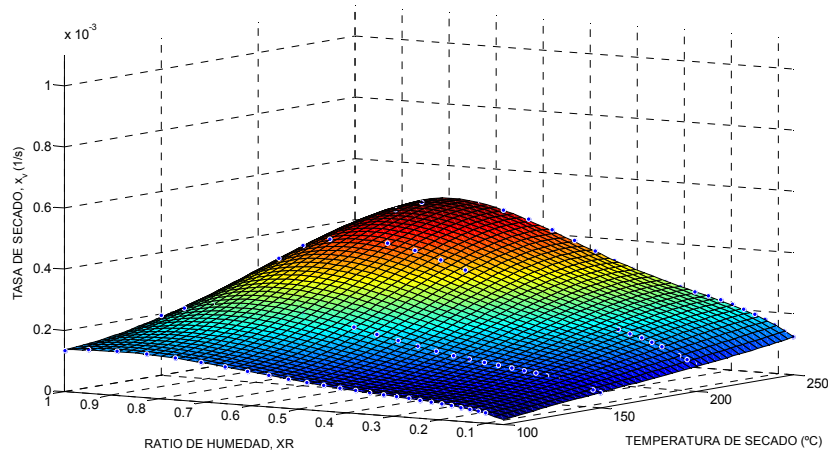
Los estudios realizados en el secado de los *posos de café* revelan que la tasa de secado puede ser expresada como una función del ratio de humedad, temperatura y velocidad del aire de secado y espesor de la muestra. Las [figuras 4.14](#), [4.15](#), [4.16](#) y [4.17](#) muestran la tasa de secado de los *posos de café* en función del ratio de humedad y la temperatura del aire de secado para cada uno de los espesores de muestra, respectivamente, cuando la velocidad del aire de secado es de 1 m/s.



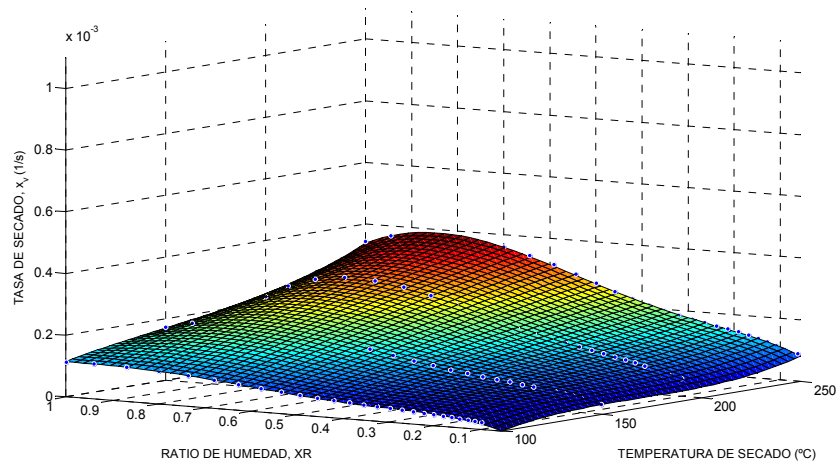
**Figura 4.14** Tasa de secado frente al ratio de humedad y la temperatura de secado para 5 mm de espesor con velocidad constante,  $v = 1 \text{ m/s}$ , en los *posos de café*.



**Figura 4.15** Tasa de secado frente al ratio de humedad y la temperatura de secado para 10 mm de espesor con velocidad constante,  $v = 1 \text{ m/s}$ , en los *posos de café*.



**Figura 4.16** Tasa de secado frente al ratio de humedad y la temperatura de secado para 15 mm de espesor con velocidad constante,  $v = 1 \text{ m/s}$ , en los *posos de café*.



**Figura 4.17** Tasa de secado frente al ratio de humedad y la temperatura de secado para 20 mm de espesor con velocidad constante,  $v = 1 \text{ m/s}$ , en los *posos de café*.

La variación de la tasa de secado, a temperatura constante, con respecto al contenido de humedad puede ser explicada a partir de las etapas de secado. Tanto durante el secado de subproductos de almazara como el *alpeorujo* y el *hueso de aceituna* como en los *posos de café*, tres etapas bien diferenciadas pueden ser distinguidas: *periodo de calentamiento*, *primer periodo de caída de la tasa de secado* y *segundo periodo de caída de la tasa de secado*. Los estudios revelan que no existe un periodo en el que la tasa de secado es constante. Así mismo, una cuarta etapa de aparición de volátiles puede aparecer a altas temperaturas. Sin embargo, como se comentó anteriormente, esta etapa no será reflejada en estos estudios debido a que los ensayos fueron interrumpidos cuando se produjo el comienzo de estos fenómenos. Las etapas de secado de estos subproductos se detallan a continuación:

-En el *periodo de calentamiento*, toda la superficie de la muestra está totalmente húmeda, y la temperatura de la muestra inicial tiene una temperatura ambiental. Conforme el ensayo avanza en el tiempo, la muestra comienza a calentarse y la tasa de secado se incrementa rápidamente (altas presiones de vapor) hasta que un valor máximo es alcanzado. A partir de este valor, la tasa de secado comienza a decrecer. Para temperaturas cercanas a los  $100^\circ\text{C}$ , este periodo no existe prácticamente y solo a pequeños espesores de muestra puede ser apreciado ligeramente (figuras 4.11 y 4.14). Esto puede ser explicado teniendo en cuenta el concepto de superficie específica (superficie de la muestra en contacto con el aire de secado dividido por el volumen de la muestra), ya que este valor es mayor conforme el espesor de la muestra disminuye.

-*El primer periodo de caída de la tasa de secado* ocurre principalmente porque la superficie de la muestra todavía tiene un cierto contenido de humedad en contacto con el aire de secado y existe un ligero movimiento de agua desde dentro de la muestra a la superficie, principalmente por difusión capilar (Strumillo C and Kudra T., 1986). El comienzo de la ausencia de agua en la superficie provoca que la tasa de secado comience a disminuir.

-Cuando *el segundo periodo de caída de la tasa de secado* es manifestado, toda la superficie de la muestra está totalmente seca. La migración del agua (vapor o líquido dependiendo de la temperatura del aire de secado) es producida desde dentro de la muestra a la superficie a través de la estructura porosa de las partículas mediante mecanismos de difusión.

Una última etapa ha sido encontrada durante el secado de los *posos de café, periodo de contracción*. Debido a la naturaleza microscópica de sus partículas, cuando el contenido de humedad se aproxima al de equilibrio, las muestras sufren contracciones. Este fenómeno provocado por el colapso por gravedad de las muestras limita la transferencia de humedad hacia el exterior y la tasa de secado cae de nuevo conforme el espesor de la muestra disminuye.

### 4.3 DIFUSIVIDAD DE HUMEDAD EFECTIVA.

El secado de subproductos de almazara es producido por procesos complejos simultáneos de transferencia de calor y humedad que son a menudo no estacionarios y cuya naturaleza y propiedades del material tan diferentes complican la descripción e identificación del proceso de secado. Los fenómenos más importantes dados durante el proceso de secado son generalmente de difusión, capilaridad y de condensación- evaporación durante *el periodo de caída de la tasa de secado*. Sin embargo, otra multitud de procesos transcurren durante el proceso de secado como: movimientos de líquido por capilaridad, difusión de líquido debido a gradientes de concentración, flujos de líquido o vapor debido a diferencias en la presión total, difusión de vapor debido a diferencias en la concentración de vapor y a los gradientes de la presión parcial de vapor, difusión Knudsen y termodifusión.

En este sentido, se propone la difusividad de humedad efectiva como una propiedad de transporte de masa general que explique e incluya todos estos fenómenos durante el secado. Para obtener experimentalmente esta propiedad, se utiliza la segunda ley de la difusión de Fick (Ec.(4)), la cual es utilizada normalmente para describir la distribución de humedad interna, el transporte de humedad y los tiempos de secado. Considerando la geometría de losa infinita propuesta en los experimentos de secado de los subproductos de almazara y de los *posos de café*, la solución se ha propuesto en la Ec.(5). Para largos periodos de tiempo y tomando logaritmos neperianos a ambos lados, Ec.(6), si se representan los valores del  $\ln XR$  y el tiempo de secado, la difusividad de humedad efectiva puede ser calculada a partir de la pendiente de la función lineal que aproxima los datos experimentales obtenidos, Ec. (36):

$$D_{eff} = \frac{s \cdot L_c^2}{\pi^2} \quad (36)$$

donde  $s$  representa el valor de la pendiente. Este procedimiento de cálculo es llamado el *Método Simplificado* porque obtiene un valor medio para todo el proceso de secado. Las tablas 4.5, 4.6 y 4.7 representan los valores medios de la difusividad de humedad efectiva en cada uno de los ensayos en el *alpeorujo*, *hueso de aceituna* y *posos de café*. Los valores del coeficiente de determinación en el ajuste de la función lineal superaron los valores de 0.97.

**Tabla 4.5** Valores medios de la difusividad de humedad efectiva en cada experimento durante el secado de *alpeorujo*.

TEST	$D_{eff}$ (m <sup>2</sup> /s)
A	$1.29 \cdot 10^{-7}$
B	$4.47 \cdot 10^{-8}$
C	$2.05 \cdot 10^{-7}$
D	$7.68 \cdot 10^{-7}$
E	$1.79 \cdot 10^{-7}$
F	$4.09 \cdot 10^{-8}$
G	$2.44 \cdot 10^{-7}$
H	$6.91 \cdot 10^{-8}$
I	$1.97 \cdot 10^{-7}$
J	$4.83 \cdot 10^{-8}$
K	$1.23 \cdot 10^{-7}$
L	$1.10 \cdot 10^{-7}$
M	$2.44 \cdot 10^{-7}$
N	$1.06 \cdot 10^{-7}$
O	$2.15 \cdot 10^{-7}$

**Tabla 4.6** Valores medios de la difusividad de humedad efectiva en cada experimento durante el secado del *hueso de aceituna*.

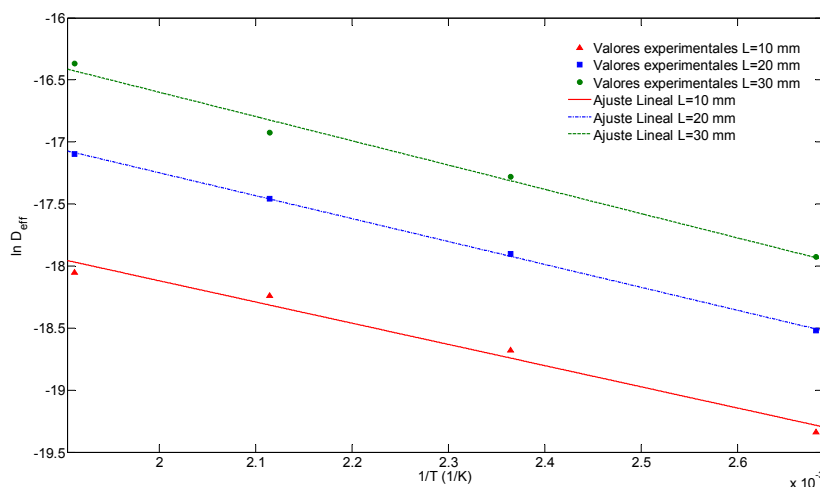
	Espesor de la muestra (mm)											
	10				20				30			
Temperatura (°C)	100	150	200	250	100	150	200	250	100	150	200	250
$D_{eff} \times 10^8$ (m <sup>2</sup> /s)	0.4	0.77	1.2	1.45	0.91	1.68	2.62	3.76	1.64	3.13	4.47	5.98

**Tabla 4.7** Valores medios de la difusividad de humedad efectiva en cada experimento durante el secado de los *posos de café*.

	Espesor de la muestra (mm)															
	5				10				15				20			
Temperatura (°C)	100	150	200	250	100	150	200	250	100	150	200	250	100	150	200	250
$D_{eff} \times 10^9$ (m <sup>2</sup> /s)	1.29	3.66	4.71	5.94	1.96	5.33	9.17	12.9	3.12	8.62	13.6	19.4	3.89	11.6	19.4	28.8

Los resultados muestran que los valores de difusividad de humedad efectiva aumentan conforme lo hace la temperatura y la velocidad del aire de secado, así como el espesor de la muestra. La [Ec.\(7\)](#) tipo Arrhenius permite obtener una relación de la difusividad de humedad efectiva y la temperatura. El valor de la energía de activación, energía necesaria para producir el movimiento de humedad en el medio, puede ser obtenido a partir de la representación del  $\ln D_{eff}$  frente a la inversa de la temperatura absoluta  $1/T$  para cada espesor de muestra. La [figura 4.18](#) muestra la función lineal de aproximación para obtener la energía de activación en los ensayos con *hueso de*

*aceituna*. Las [tablas 4.8 y 4.9](#) indican los valores de la energía de activación para cada uno de los espesores de muestra del *hueso de aceituna* y los *posos de café*.



**Figura 4.18** Relación de Arrhenius entre el logaritmo de la difusividad de humedad efectiva y la inversa de la temperatura para diferentes espesores de muestra de *hueso de aceituna*.  $v = 1 \text{ m/s}$ .

**Tabla 4.8** Valores de energía de activación para diferentes espesores de muestra en el *hueso de aceituna* y su coeficiente de determinación en el ajuste de la función lineal.

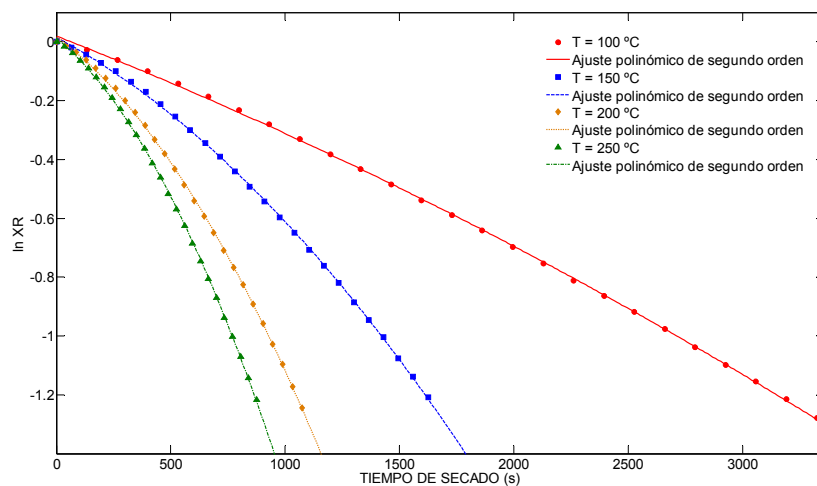
	Espesor de la muestra (mm)		
	10	20	30
Energía de activación (J/mol)	14,208	15,356	16,270
$R^2$	0.9801	0.995	0.9884

**Tabla 4.9** Valores de energía de activación para diferentes espesores de muestra en los *posos de café* y su coeficiente de determinación en el ajuste de la función lineal.

	Espesor de la muestra (mm)			
	5	10	15	20
Energía de activación (J/mol)	12,288	12,778	15,181	16,869
$R^2$	0.9928	0.9903	0.9958	0.9919

Comúnmente, en el campo del modelado numérico de la transferencia de calor y masa en procesos de secado, los valores de la difusividad de humedad efectiva suelen considerarse constantes (Kaya et al., 2008) o están basados en una relación tipo Arrhenius dependiente de la temperatura (Hussain and Dincer, 2003; Kaya et al., 2006), como se han calculado anteriormente. Sin embargo, la difusividad efectiva presenta una

fuerte dependencia al contenido de humedad y al grado de contracción de la muestra, además de la temperatura del aire de secado (Efremov and Kudra, 2004; Vasić et al., 2014). Por este motivo, la difusividad de humedad efectiva para el *hueso de aceituna* limpio ha sido calculada como una función dependiente del tiempo. Para ello, una nueva metodología ha sido desarrollada en esta Memoria, la cual consiste en una modificación del *Método Simplificado*. Este método consiste en ajustar los datos experimentales  $\ln XR - t$ , reemplazando la función lineal de ajuste por una función parabólica. La [figura 4.19](#) muestra la gráfica  $\ln XR - t$  para los ensayos realizados para un espesor de muestra de 10 mm y el ajuste polinómico de segundo orden propuesto en cada experimento.



**Figura 4.19** Curvas de secado logarítmicas a diferentes temperaturas y su ajuste por medio de funciones parabólicas para un espesor de muestra de 10 mm en el *hueso de aceituna*.

Por lo tanto, en el caso general de curvas logarítmicas de secado no lineales, el *Método Simplificado Modificado* puede ser aplicado para estimar la dependencia del tiempo de la difusividad de humedad efectiva a partir de la derivada de las ecuaciones parabólicas de aproximación como se detalla a continuación:

$$D_{eff}(t) = -\frac{d(\ln(XR))}{dt} \cdot \frac{L_c^2}{\pi^2} = -(2p_1 \cdot t + p_2) \cdot \frac{L_c^2}{\pi^2} \quad (37)$$

donde  $p_1$  y  $p_2$  son los coeficientes de la derivada de la ecuación parabólica. La [tabla 4.10](#) muestra los valores de la derivada de la función parabólica de aproximación y sus valores de  $R^2$  y  $RMSE$  en cada experimento.

**Tabla 4.10** Valores de la pendiente,  $R^2$  y  $RMSE$  en la aproximación mediante polinomio de segundo orden de las curvas logarítmicas de secado.

Espesor de la muestra (mm)	Temperatura (°C)	$\frac{d(\ln(XR))}{dt}$	$R^2$	$RMSE$
10	100	$2(-2.64 \cdot 10^{-8}) \cdot t - 3.059 \cdot 10^{-4}$	0.9998	0.005195
	150	$2(-2.033 \cdot 10^{-7}) \cdot t - 4.27 \cdot 10^{-4}$	0.9998	0.005238
	200	$2(-5.48 \cdot 10^{-7}) \cdot t - 5.889 \cdot 10^{-4}$	0.9998	0.005661
	250	$2(-8.797 \cdot 10^{-7}) \cdot t - 6.408 \cdot 10^{-4}$	0.9999	0.004508
20	100	$2(-1.345 \cdot 10^{-9}) \cdot t - 2.16 \cdot 10^{-4}$	0.9999	0.002951
	150	$2(-3.15 \cdot 10^{-8}) \cdot t - 3.156 \cdot 10^{-4}$	0.9997	0.006567
	200	$2(-1.017 \cdot 10^{-7}) \cdot t - 4.479 \cdot 10^{-4}$	0.9999	0.004074
	250	$2(-2.969 \cdot 10^{-7}) \cdot t - 5.112 \cdot 10^{-4}$	0.9999	0.002742
30	100	$2(-2.227 \cdot 10^{-14}) \cdot t - 1.802 \cdot 10^{-4}$	1	0.002616
	150	$2(-8.185 \cdot 10^{-9}) \cdot t - 3.118 \cdot 10^{-4}$	0.9997	0.006493
	200	$2(-4.707 \cdot 10^{-8}) \cdot t - 3.697 \cdot 10^{-4}$	0.9997	0.004657
	250	$2(-1.033 \cdot 10^{-7}) \cdot t - 4.406 \cdot 10^{-4}$	0.9999	0.004193

Los resultados obtenidos por el *Método Simplificado Modificado* han sido comparados con los calculados por el *Método de las Pendientes*, el cual es ampliamente utilizado en la literatura sobre el secado en una gran variedad de productos agrícolas y alimentos (Azzouz et al., 2002; Babalis and Belessiotis, 2004; Pinto and Tobinaga, 2006). El *Método de las Pendientes* ha sido aplicado para estimar la difusividad de humedad efectiva en las curvas logarítmicas de secado no lineales como una función del ratio de humedad. La pendiente de las curvas de secado experimentales  $(dXR/dt)_{exp}$ , la tasa de secado experimental, es determinada a partir de los datos experimentales. Y la pendiente de las curvas teóricas  $(dXR/dFo)_{teo}$  es calculada por diferenciación del ratio de humedad, Ec. (38), con respecto al número de *Fourier* de difusión (Ec. (39)). La difusividad de humedad efectiva puede ser obtenida para un valor del ratio de humedad a partir de la Ec. (40).

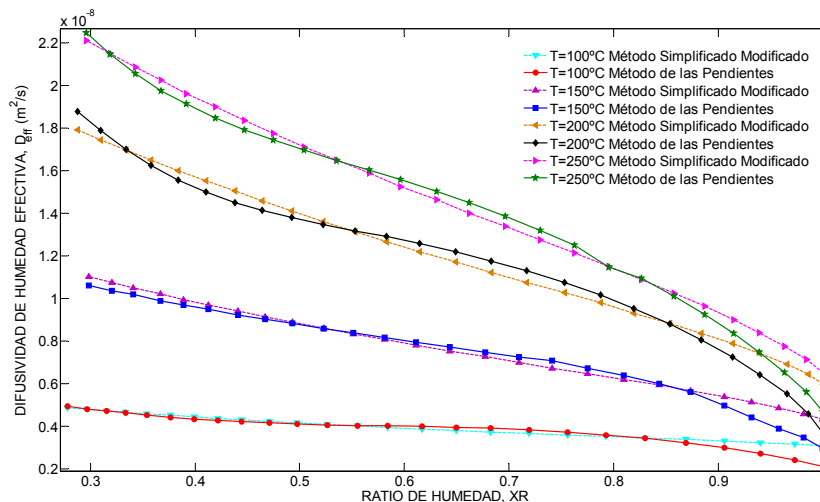
$$XR = \frac{8}{\pi^2} \exp\left(-\frac{\pi^2 D_{eff} t}{L_c^2}\right) \quad (38)$$

$$Fo = \frac{D_{eff} \cdot t}{L_c^2} \quad (39)$$

$$D_{eff} = \frac{(dXR/dt)_{exp}}{(dXR/dFo)_{teo}} \cdot L_c^2 \quad (40)$$

Tanto el *Método Simplificado Modificado* como el *Método de las Pendientes* pueden ser utilizados en materiales con un alto grado de contracción de la muestra

substituyendo el valor constante del espesor de la muestra,  $L_c$ , por una función dependiente del tiempo,  $L_c(t)$ , la cual indica la variación de espesor durante la realización de los experimentos de secado. Sin embargo, los resultados obtenidos en los experimentos de secado en el *hueso de aceituna* limpio no mostraron este comportamiento debido a su naturaleza granular. Ya que cada valor del tiempo de secado está ligado a un valor de ratio de humedad, la difusividad de humedad efectiva puede ser expresada como:  $D_{eff} = D_{eff}(XR)$  para cada experimento. La [figura 4.20](#) detalla los valores de la difusividad de humedad efectiva frente al ratio de humedad para cada temperatura de secado y un espesor de muestra de 10 mm, calculada a partir de estos métodos. Como puede apreciarse, los resultados obtenidos fueron muy similares entre ambos métodos.



**Figura 4.20** Comparación entre el *Método de las Pendientes* y el *Método Simplificado Modificado* en el cálculo de la difusividad de humedad efectiva como una función del ratio de humedad para un espesor de muestra de 10 mm en el *hueso de aceituna*.

Sin embargo, las técnicas numéricas relacionadas con la transferencia de calor y masa en medios de naturaleza y composición compleja requieren un cálculo más exhaustivo de las propiedades de las cuales estos fenómenos dependen. El diseño experimental propuesto a partir de ensayos isotérmicos llevados a cabo en el *hueso de aceituna* ha permitido estimar la difusividad de humedad efectiva como una función del ratio de humedad y la temperatura del aire de secado para cada espesor de muestra,  $D_{eff} = D_{eff}(T, XR)$ , por medio de un análisis de regresión múltiple. Además, a altas temperaturas, la temperatura de la muestra es rápidamente alcanzada, y la diferencia de

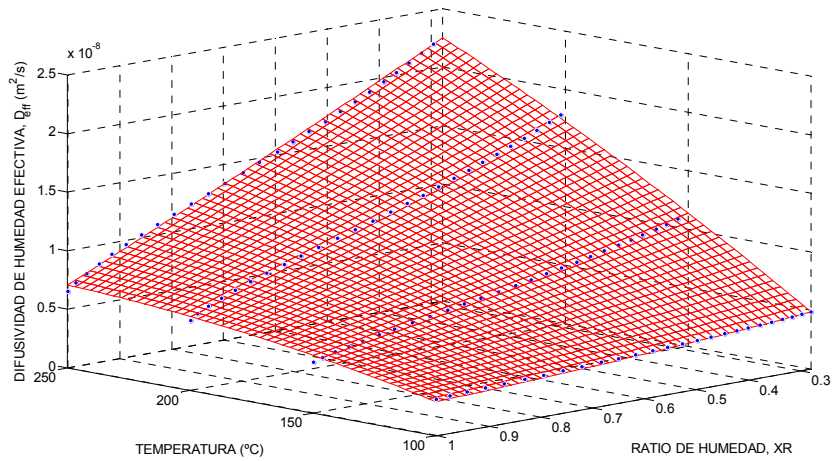
temperatura entre la superficie y el interior de la muestra es difícilmente apreciada conforme el experimento de secado se realiza (Ruiz-López et al., 2012). Un modelo polinómico multivariable de segundo orden con un nivel de confianza del 95% fue propuesto (Ec.(41)). Este modelo presentó excelentes resultados de ajuste para la función experimental  $D_{eff} = D_{eff}(T, XR)$ , eliminando el uso de un número elevado de coeficientes (Tabla 4.11).

$$D_{eff}(T, XR) = a_0 + a_1 \cdot T + a_2 \cdot XR + a_3 \cdot T^2 + a_4 \cdot T \cdot XR + a_5 \cdot XR^2 \quad (41)$$

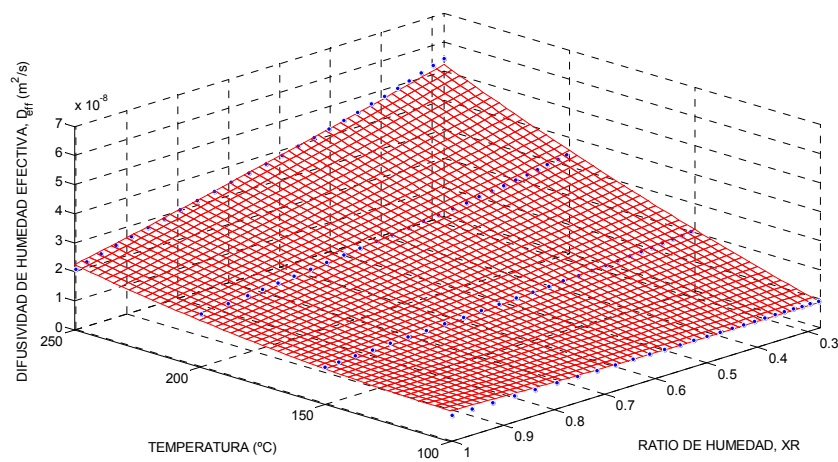
**Tabla 4.11** Parámetros que define el modelo polinómico multivariable de segundo orden, Ec.(41), y los criterios estadísticos en cada espesor de muestra en el *hueso de aceituna*.

Espesor muestra (mm)	Tipo de método	$a_0$	$a_1$	$a_2$	$a_3$	$a_4$	$a_5$	$R^2$	RMSE
10	Método Simplificado Modificado	$-1.231 \cdot 10^{-8}$	$1.96 \cdot 10^{-10}$	$7.833 \cdot 10^{-9}$	$-1.252 \cdot 10^{-13}$	$-1.244 \cdot 10^{-10}$	$1.472 \cdot 10^{-9}$	0.9963	$3.144 \cdot 10^{-10}$
10	Método de las Pendientes	$-1.482 \cdot 10^{-8}$	$1.946 \cdot 10^{-10}$	$1.779 \cdot 10^{-8}$	$-1.183 \cdot 10^{-13}$	$-1.273 \cdot 10^{-10}$	$-6.626 \cdot 10^{-9}$	0.9884	$5.73 \cdot 10^{-10}$
20	Método Simplificado Modificado	$-1.519 \cdot 10^{-8}$	$2.061 \cdot 10^{-10}$	$2.33 \cdot 10^{-8}$	$4.634 \cdot 10^{-13}$	$-2.882 \cdot 10^{-10}$	$5.985 \cdot 10^{-9}$	0.9958	$8.08 \cdot 10^{-10}$
20	Método de las Pendientes	$-1.904 \cdot 10^{-8}$	$1.962 \cdot 10^{-10}$	$4.156 \cdot 10^{-8}$	$4.944 \cdot 10^{-13}$	$-2.92 \cdot 10^{-10}$	$-9.609 \cdot 10^{-9}$	0.9962	$7.795 \cdot 10^{-10}$
30	Método Simplificado Modificado	$-3.198 \cdot 10^{-8}$	$4.972 \cdot 10^{-10}$	$2.586 \cdot 10^{-8}$	$3.181 \cdot 10^{-15}$	$-3.532 \cdot 10^{-10}$	$1.104 \cdot 10^{-8}$	0.9971	$9.626 \cdot 10^{-10}$
30	Método de las Pendientes	$-4.199 \cdot 10^{-8}$	$4.689 \cdot 10^{-10}$	$7.212 \cdot 10^{-8}$	$7.193 \cdot 10^{-14}$	$-3.554 \cdot 10^{-10}$	$-2.753 \cdot 10^{-9}$	0.9966	$1.032 \cdot 10^{-9}$

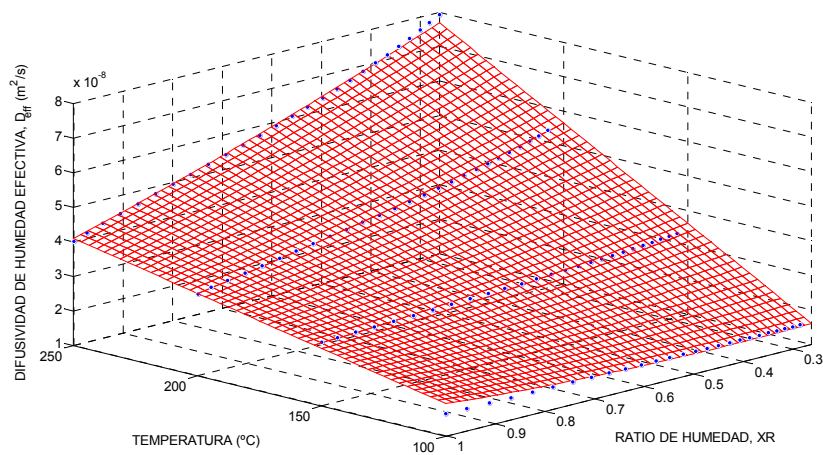
Las figuras 4.21, 4.22 y 4.23 muestran las funciones  $D_{eff} = D_{eff}(T, XR)$ , para cada espesor de muestra, obtenidas a partir del *Método Simplificado Modificado*. Estas superficies matemáticas permiten obtener los valores de difusividad de humedad efectiva en el rango de las condiciones actuales de operación en el secado de *hueso de aceituna* limpio. Un comportamiento similar ha sido obtenido en el secado de quitosano para una temperatura, velocidad y espesor de 60°C, 1.5 m/s y 4 mm (Batista et al., 2007), respectivamente, y durante la cocción de la torta blanca para temperaturas entre 50°C y 160°C, velocidad del aire de 0.56 m/s y un espesor de 3 mm (Sakin et al., 2007).



**Figura 4.21** Difusividad de humedad efectiva en términos del ratio de humedad y la temperatura del aire de secado para un espesor de muestra en el *hueso de aceituna* de 10 mm.



**Figura 4.22** Difusividad de humedad efectiva en términos del ratio de humedad y la temperatura del aire de secado para un espesor de muestra en el *hueso de aceituna* de 20 mm.



**Figura 4.23** Difusividad de humedad efectiva en términos del ratio de humedad y la temperatura del aire de secado para un espesor de muestra en el *hueso de aceituna* de 30 mm.

Como se comentó en la [sección 4.2](#), los mecanismos de transporte de masa durante el secado en el *periodo de calentamiento*, el *primer periodo de caída de la tasa de secado* y el *segundo periodo de caída de la tasa de secado* son bastante complejos y a menudo son muy difíciles de identificarlos. Los fenómenos de secado en el *hueso de aceituna* limpio pueden ser detallados a partir del comportamiento de la difusividad de humedad efectiva.

Las [figuras 4.21, 4.22 y 4.23](#) indican que los valores de la difusividad de humedad efectiva se incrementan conforme el ratio de humedad decrece y la temperatura de secado aumenta. Al comienzo del proceso de secado de cada experimento, un corto periodo de tiempo es necesario para establecer las condiciones de secado estacionarias. Esta etapa es conocida como *periodo de calentamiento*. El aire de secado es impulsado hacia la muestra y el calor es transferido a la superficie. El calor latente de vaporización provoca la evaporación del agua. Conforme el contenido de humedad disminuye en la superficie, una región de presiones bajas de vapor de agua es originada. Un gradiente de presión en el agua es manifestado desde el agua interna de la muestra al aire de secado. Así pues, esta etapa está gobernada por difusión a través de la capa límite debido a la diferencia de concentraciones de humedad entre la superficie de la muestra y el aire de secado. La [figura 4.20](#) muestra el *periodo de calentamiento* entre  $XR = 1$  y  $XR = 0.85$  donde la difusividad de humedad efectiva es baja.

El *primer periodo de caída de la tasa de secado* está caracterizado por un incremento moderado de la difusividad de humedad efectiva debido al transporte de humedad por difusión capilar. En esta etapa, conforme ocurre la evaporación, la superficie de la muestra está prácticamente saturada debido al flujo constante de agua desde el interior de la muestra. Por tanto, el principal mecanismo de transporte de masa en este periodo es originado por el gradiente del potencial capilar. Por otro lado, un flujo líquido en los poros, el cual es creado debido a la diferencia de presión causada por fricción, aparece. Conforme estos dos mecanismos actúan, la superficie de la muestra abandona el estado de saturación donde ya solo existe un estado líquido continuo dentro del material (*estado funicular* (Strumillo C and Kudra T., 1986)). A altas temperaturas, mayores a 100°C, el *primer periodo de caída de la tasa de secado* puede estar incluido en el *periodo de calentamiento* (Arjona et al., 1999). Este fenómeno puede ser mostrado para valores de difusividad de humedad efectiva entre  $XR = 1$  y  $XR = 0.4$ , mientras que a bajas temperaturas esta etapa se manifiesta para valores entre  $XR = 0.9$  y  $XR = 0.4$ .

Finalmente, en el *segundo periodo de caída de la tasa de secado*, la superficie de la muestra está totalmente seca y los continuos canales de agua dentro del material han desaparecido (*estado pendular* (Strumillo C and Kudra T., 1986)). Esta etapa puede ser observada cuando la difusividad de humedad efectiva alcanza un ratio de humedad de 0.4 (figura 4.20). Conforme el ratio de humedad decrece por debajo de este valor, los valores de la difusividad de humedad efectiva se incrementan ligeramente. A bajas temperaturas, la difusión del líquido es producida en los poros debido al gradiente de concentración, y, a altas temperaturas, el vapor es transportado por diferencias en la presión total (*flujo hidrodinámico*).

## 5. CONCLUSIONES.

Esta Tesis Doctoral aporta el cálculo de variables útiles para completar y estudiar a fondo los modelos matemáticos de transferencia de calor y masa en secaderos rotativos de subproductos de almazara en busca de un secado más óptimo y eficiente.

Para ello, se recurre al análisis experimental de la cinética de secado en el *alpeorujo*, *hueso de aceituna* y otros residuos biomásicos como los *posos de café*, la cual arroja resultados sobre los tiempos de secado, las tasas de secado, las condiciones óptimas de secado e incluso variables importantes en los fenómenos de transferencia de masa como la difusividad de humedad efectiva y que serán de aplicación especialmente en el estudio de los modelos por secciones en secaderos rotativos.

La sinergia entre los principales modelos matemáticos en secaderos rotativos y los estudios de la cinética de secado para cada subproducto permitirá mejorar el diseño, control y optimización del proceso de secado en este tipo de secaderos.

No obstante, las principales conclusiones obtenidas tras la realización de esta Memoria pueden ser detalladas a continuación:

-El estudio de la cinética de secado de subproductos de almazara depende principalmente de tres factores importantes como la temperatura del aire de secado, la velocidad del aire de secado y el espesor de muestra estudiado, siendo el primer factor la fuerza rectora del proceso.

-Para el estudio de la cinética de secado en el *alpeorujo*, se realizó un diseño de experimentos acorde a las condiciones dadas en secaderos rotativos. Sin embargo, la limitación del equipo experimental solo permitió alcanzar una temperatura máxima de 425°C. En el caso de la cinética de secado del *hueso de aceituna* y de los *posos de café* se optó por unas condiciones de secado menos agresivas y más acorde a las condiciones reales en secaderos rotativos.

-Estos trabajos han permitido saber las condiciones límites correspondientes a la temperatura de secado y la humedad del subproducto en las cuales aparece la liberación de volátiles y la combustión de las muestras. Todos los resultados muestran que temperaturas iguales o superiores a 250°C provocan estos fenómenos cuando el

contenido de humedad se acerca al de equilibrio. Sin embargo, en las condiciones propuestas en el estudio del *alpeorujo* donde en algunos ensayos se manifiestan altas temperaturas y velocidades del aire de secado, la combustión puede aparecer a mayores contenidos de humedad.

-Se ha presentado un nuevo modelo matemático de capa delgada empírico, *Two Term Gaussian*, el cual presenta los mejores resultados de ajuste de los modelos estudiados hasta la fecha en la literatura. Esto radica en la obtención de funciones matemáticas que representen fielmente los tiempos y las tasas de secado experimentales.

-Los estudios realizados con los *posos de café* han contribuido a entender e interpretar los resultados obtenidos en el secado de los subproductos de almazara, así como corroborar las condiciones óptimas de secado.

-Finalmente, la difusividad de humedad efectiva ha sido obtenida como una propiedad de la transferencia de masa global durante el proceso de secado. Esta propiedad ayudará en la iniciación en el estudio del secado mediante simulaciones numéricas a partir de técnicas de Mecánica de Fluidos Computacional (CFD). Además, un nuevo método ha sido propuesto en la obtención de esta propiedad como una función dependiente del tiempo, el *Método Simplificado Modificado*, el cual presenta resultados muy similares al *Método de las Pendientes* y es más sencillo de aplicar.

## 6. TRABAJOS FUTUROS.

Los resultados obtenidos en la presente Tesis Doctoral serán aplicados al estudio experimental en secaderos rotativos. En concreto, el Proyecto de Investigación de Excelencia concedido por la Consejería de Innovación, Ciencia y Empresa de la Junta de Andalucía con el título “Modelado y Control de Secadero Rotativo de Orujo” ha financiado el diseño, construcción y fabricación de un secadero rotativo a escala ubicado en el laboratorio de Máquinas y Motores Térmicos del Departamento de Ingeniería Mecánica y Minera de la Universidad de Jaén (figura 6.1).



**Figura 6.1** Secadero rotativo experimental para el secado de subproductos de almazara.

El equipo está formado principalmente por:

-Un horno basado en unas resistencias que calientan el aire de secado aspirado por los ventiladores centrífugos. El modelo T-MAX-L 45 está compuesto por treinta resistencias dispuestas en tres fases: dos resistencias de 18 kW cada una y otra de 9 kW. En total puede desarrollar 45 kW.

-Un trómel giratorio de velocidad variable de 50 cm de diámetro y 3.5 m de largo, compuesto en su interior de aspas que voltean el material. El trómel tiene varios

sensores, para captación de temperaturas y otras variables, repartidos en toda su longitud.

-Dos ventiladores centrífugos. El primero colocado a la salida del trómel, el cual provoca el arrastre del material. El segundo colocado cerca de las resistencias para suministrar el aire secundario del horno y para medir velocidades del aire de secado.

-Un separador ciclónico que permite la separación por rotación y gravedad de las partículas de menor tamaño durante el secado.

-Una tolva de administración de los subproductos a secar conectado a un tornillo sinfín.

-Un tornillo sinfín de salida de producto.

-Un equipo electrónico que automatiza y controla el secadero rotativo a escala.

En primer lugar, se realizará la caracterización del secadero rotativo a partir de variables como: las temperaturas y velocidades del aire de secado, flujos de subproducto, velocidad de giro del trómel, pérdidas de calor y valores del coeficiente volumétrico de transferencia de calor y tiempos de residencia.

Conocidas las principales variables a considerar dentro del secadero rotativo como la velocidad y temperatura del aire de secado, espesor de material que voltean las aspas y el contenido de humedad del subproducto en cada una de las secciones, se podrán aplicar los conocimientos adquiridos en el estudio de la cinética de secado para cada subproducto. Se trata así de conocer el proceso de secado en el interior del trómel y a lo largo de su longitud. El conocimiento de las variables más importantes que influyen durante el secado en el interior de secaderos rotativos permitirá optimizar y controlar su funcionamiento más eficientemente.

Además, con la obtención de propiedades en los subproductos como la difusividad de humedad efectiva y otras todavía por calcular como el calor específico, difusividad térmica y conductividad térmica, se pretende realizar simulaciones numéricas mediante técnicas como la Mecánica de Fluidos Computacional que ayuden a

explicar y visualizar el fenómeno del secado tanto en el túnel de secado como en el secadero rotativo experimental.

Como trabajos auxiliares, sería necesario aproximar los valores de tiempo de secado y tasa de secado para cualquier condición a partir de los diseños experimentales obtenidos en el estudio de la cinética de secado mediante la utilización de redes neuronales.

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## 8. ANEXOS.

### 8.1 ARTÍCULOS PUBLICADOS EN EL JOURNAL CITATION REPORTS.

**JOURNAL:** Energy. ISSN: 0360-5442.

**FACTOR DE IMPACTO:** 4.159

**CATEGORÍAS:** Energy and Fuel (13/83) y Thermodynamics (2/55)

**TÍTULO:** Drying kinetics of olive stone: a valuable source of biomass obtained in the olive oil extraction. Volumen 75. Páginas: 146-152. Año: 2014.

DOI: [10.1016/j.energy.2014.06.085](https://doi.org/10.1016/j.energy.2014.06.085)

**AUTORES:** F.J. Gómez-de la Cruz, P.J. Casanova-Peláez, J.M. Palomar-Carnicero, F. Cruz-Peragón.

**JOURNAL:** Fuel Processing Technology. ISSN: 0378-3820.

**FACTOR DE IMPACTO:** 3.019

**CATEGORÍAS:** Energy and Fuel (27/83), Chemistry Applied (12/71) y Engineering Chemical (18/133)

**TÍTULO:** A vital stage in the large-scale production of biofuels from spent coffee grounds: The drying kinetics. Volumen 130. Páginas 188-196. Año: 2015.

DOI: [10.1016/j.fuproc.2014.10.012](https://doi.org/10.1016/j.fuproc.2014.10.012)

**AUTORES:** F.J. Gómez-de la Cruz, F. Cruz-Peragón, P.J. Casanova-Peláez, J.M. Palomar-Carnicero.

**JOURNAL:** Applied Thermal Engineering. ISSN: 1359-4311.

**FACTOR DE IMPACTO:** 2.624

**CATEGORÍAS:** Energy and Fuel (34/83), Engineering Mechanical (10/128), Mechanics (8/139) y Thermodynamics (6/55)

**TÍTULO:** Modeling of olive-oil mill waste rotary dryers: Green energy recovery systems. Volumen 80. Páginas 362-373. Año: 2015.

DOI: [10.1016/j.applthermaleng.2015.01.035](https://doi.org/10.1016/j.applthermaleng.2015.01.035)

**AUTORES:** F.J. Gómez-de la Cruz, P.J. Casanova-Peláez, J.M. Palomar-Carnicero, F. Cruz-Peragón.

**JOURNAL:** Fuel Processing Technology. ISSN: 0378-3820.

**FACTOR DE IMPACTO:** 3.019

**CATEGORÍAS:** Energy and Fuel (27/83), Chemistry Applied (12/71) y Engineering Chemical (18/133)

**TÍTULO:** Experimental determination of effective moisture diffusivity during the drying of clean olive stone: dependence of temperature, moisture content and sample thickness. In press. Año: 2015.

*DOI:10.1016/j.fuproc.2015.03.018*

**AUTORES:** F.J. Gómez-de la Cruz, J.M. Palomar-Carnicero, P.J. Casanova-Peláez, F. Cruz-Peragón.

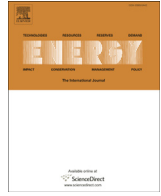
## 8.2 ARTÍCULOS PUBLICADOS EN OTRAS REVISTAS.

**JOURNAL:** International Scientific Journal - Journal of environmental science.

**ISBN-10:** 1499721986

**TÍTULO:** Obtaining of the drying rate of Alpeorujo for applications on rotary dryers. Volumen 3. Páginas 191-197. Año: 2014.

**AUTORES:** F.J. Gómez-de la Cruz, P.J Casanova-Peláez, J.M. Palomar-Carnicero, S. Sánchez, R. Pacheco, F. Cruz-Peragón.



# Drying kinetics of olive stone: A valuable source of biomass obtained in the olive oil extraction



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## ABSTRACT

Olive stone is a by-product of the olive grove especially suitable for thermal purpose in industrial, residential and home. To avoid maintenance problems in boilers and to increase the combustion efficiency, olive stone needs to be dried to moisture equilibrium, about 8% (wet basis). The thin layer drying kinetics was investigated in a drying tunnel. Isothermal drying tests were performed with different drying air temperatures: 100, 150, 200 and 250 °C for each sample thickness: 10, 20 and 30 mm. Drying curves were analyzed from the different mathematical models studied by the researchers to date. A new mathematical model is proposed in this work, Two Term Gaussian, which presents the best results of fit. The drying rate is calculated and analyzed. The effective diffusivity values range from  $3.98 \cdot 10^{-9}$  to  $5.97 \cdot 10^{-8}$  m<sup>2</sup>/s. Furthermore, the activation energies values were 14,208, 15,356 and 16,270 J/mol for each sample thickness: 10, 20 and 30 mm, respectively.

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## 1. Introduction

Olive grove is the main source of biomass residues in Andalusia (Spain). There are more than 1.4 million hectares for the olive grove cultivation in Andalusia which produce an average total of 4,700,000 tons of olives per year [1]. The current process of olive oil extraction, two-phase system, separates the virgin olive oil from alpeorujo (olive cake and vegetable water mixture). The alpeorujo is formed by all wastes of the olives: skin, pulp, crushed olive stones, olive oil content about 5% and vegetable water with organics compounds.

The average annual production of olive stone ranges from 360,000 tons per year [2]. The olive stone and olive pulp are separated from the alpeorujo by physical media in olive oil mill and extracted factories [3]. This by-product is used for many applications: activated carbon, liquid and gas fuel produced by pyrolysis, plastic filled, cosmetics, abrasives and furfural production [4]. However, the vast majority of production is spent for the production of electrical and thermal renewable energy [5,6]. As first use, olive stone is employed as biofuel to provide heat in the beating stage, in the olive oil mills, and to produce heat in the drying of the

alpeorujo, in the extracted factories [1]. More than 90% of the olive stone is used for thermal energy for industrial purposes and for space heating in commercial building, residential and homes [7,8].

This biomass product highlighted by low percentages of nitrogen and sulfur minimizing emission of NO<sub>x</sub> and SO<sub>2</sub> which produce acid rain and cause the destruction of the ozone layer [9,10]. Furthermore, the olive stone combustion does not contribute to climate change with the CO<sub>2</sub> emissions to the atmosphere. The net calorific value is estimated at 19,200 kJ/kg [11].

Drying of the olive stone is necessary for two fundamental reasons. First, olive stone is accompanied by the olive pulp when is separated from the alpeorujo. The olive pulp contains some content of olive oil that needs to be extracted. To do so, it is essential the drying until equilibrium moisture content, where the solvent is more effective. On the other hand, high moisture content in the olive stone decreases the combustion yield [12] and provokes water vapor condensations that are unfavorable in the heating boilers. Moreover, the removal of the moisture content improves the storage and transport conditions.

So far, there are no works in the drying of olive stone in the literature. All researches in the drying of by-products of the olive grove focus on the drying of olive cake (40–50% moisture content, three-phase system) [13–16] and alpeorujo (60–70% moisture content, two-phase system) [17–19].

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## Nomenclature

$a, b, c, d, e, f, n$	coefficients of the mathematical models
$k, k_0, k_1, k_2, k_3$	constants of the mathematical models ( $s^{-1}$ )
$D_{\text{eff}}$	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 ( $^{\circ}C, K$ )
$v$	velocity ( $m s^{-1}$ )
$X_e$	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 $\cdot$ s))

This work presents a study about the drying kinetics of the olive stone (with olive pulp) in a drying tunnel, although the results can serve as starting point in other drying systems like: fluidized bed dryers, solar dryers and rotary dryers. First, drying curves were fitted with the main mathematical model in the drying of agricultural products. A new mathematical model, which obtains the best results of fit, is proposed, Two Term Gaussian. Second, the drying rate was calculated and analyzed. Third, the effective diffusivity values were obtained for each test. Finally, the activation energies were found for each sample thickness.

## 2. Materials and methods

### 2.1. Materials

Olive stone samples with olive pulp were kindly donated by an olive oil mill and a company of drying of the olive stone in the province of Jaen (Spain). The weight of the olive pulp was estimated at approximately 30% of the total weight. The olive stone size ranges from 1 to 3 mm of diameter. To find out the initial moisture content, the samples were dried in an oven (Memmert GmbH + Co.KG, SNB 167 Model 100, Germany) at  $105^{\circ}C$  for 24 h. Drying samples were performed in triplicate. An average moisture content of  $23 \pm 0.5\%$  (wet basis) was found. The same procedure was applied to obtain the equilibrium moisture content which was estimated at  $8 \pm 0.5\%$  (wet basis).

### 2.2. Drying equipment and experimental procedure

Drying tests were carried out in a drying tunnel (Fig. 1). The drying equipment is formed by: a blower, electric resistances and a tunnel of 2 m of length with thermal insulation and 0.15 m of square section. The air velocity of the blower was controlled by a VFD (Variable Frequency Drive) connected to an electric AC motor. To achieve the desired temperature in each test, air was passed through a group of three independent resistances: 9 (first stage), 18 (second stage) and 18 kW (third stage), 45 kW in total. To control the constant temperature in each test, a PID (Proportional-Integral-Differential) controller acted over the resistances, measuring the

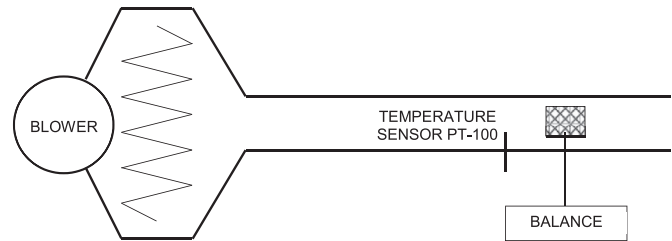


Fig. 1. Drying tunnel scheme.

temperature using a PT 100 sensor. The sensor was positioned just before the point of drying of samples. Once the test conditions were correct, the sample was introduced (with the corresponding thickness in each test) into the tunnel in a steel basket of 7 cm of square section. The basket was placed over a precision balance (Blauscal AH1200) with an error of  $\pm 0.01$  g. It was connected to personal computer by USB port. Software measured the variation of mass every second and stored the test information in files. Drying experiments were stopped when the equilibrium moisture content of the sample was approximately accomplished. However, to obtain the exact moisture content, when the experiments were finished, samples were dried until 0% moisture content in the oven at  $105^{\circ}C$  for 24 h.

Twelve experiments were performed. For each sample thickness: 10, 20, 30 mm, four tests with constant drying air temperatures at 100, 150, 200 and  $250^{\circ}C$  were carried out. The drying air velocity was established in  $1 \pm 0.1$  m/s.

## 3. Results and discussion

### 3.1. Drying curves

Drying curves represent the moisture ratio function versus drying time. The moisture ratio can be expressed as (Eq. (1)):

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

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. Nevertheless, the 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.

Figs. 2–4 show the drying curves for sample thicknesses: 10, 20 and 30 mm, respectively. As can be seen, the drying time decreases when the drying air temperature increases. On the other hand, drying times increase when the sample thickness increases. The drying time falls to approximately four times when the drying temperature rises from  $100^{\circ}C$  to  $250^{\circ}C$ , for each of the sample thicknesses. When the moisture content, in tests carried out at  $250^{\circ}C$ , reaches the equilibrium moisture content, the external surface of the sample begins to burn, regardless of the sample thickness. Tests were stopped at the beginning of the combustion. This indicates that the drying air temperature should be controlled, because this phenomenon causes energy characteristics of the olive stone getting worse.

Drying is a complex physic process which depends on diffusion and convection phenomena. Drying curves were fitted with the main mathematical models in the drying of products. Thirteen mathematical models were used to approximate the drying curves by non-linear regression analysis. Table 1 indicates the names of the fit mathematical models and their expressions. A new mathematical model is presented in this work, Two Term Gaussian.

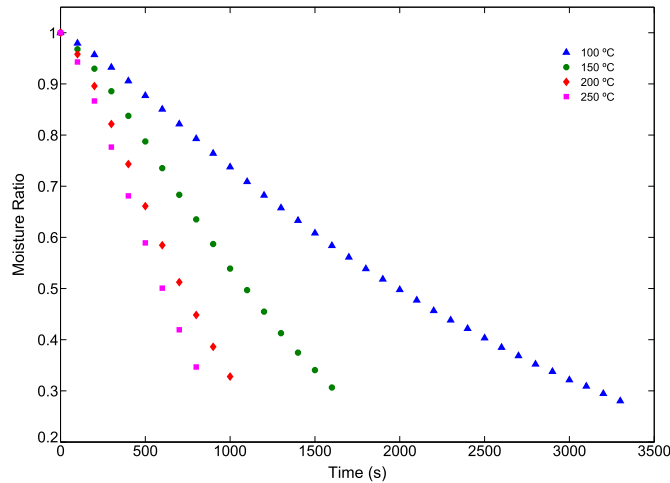


Fig. 2. Drying curves for  $L = 10$  mm at different temperatures with constant air velocity,  $v = 1$  m/s.

To verify the quality of fit, the coefficient of determination,  $R^2$ , and the root mean square error, RMSE, were used. Table 2 shows the values of  $R^2$  and RMSE for the different mathematical models applied to each of the twelve drying curves. The mathematical models which present the best results of fit were: Two Term Gaussian, Yun et al., Midilli et al., and Approach of Diffusion. Six significant figures were used to evaluate the fits, due to excellent approximations of these models. The models proposed by Yun et al., Midilli et al. and Approach of Diffusion obtained an average value of  $R^2$  and RMSE of 0.999972 and 0.000938, 0.999917 and 0.001706, and 0.999777 and 0.002551, respectively. However, Two Term Gaussian model is presented as the best fit model for drying curves of the olive stone with an average value of  $R^2$  and RMSE of 0.999978 and 0.000837, respectively.

### 3.2. Drying rate

The drying rate is a variable very important to optimize and control the drying process. This value indicates the moisture content variation with respect to time, in other word, the amount of evaporated water per time unit. The drying rate of the olive stone can be expressed as (Eq. (2)):

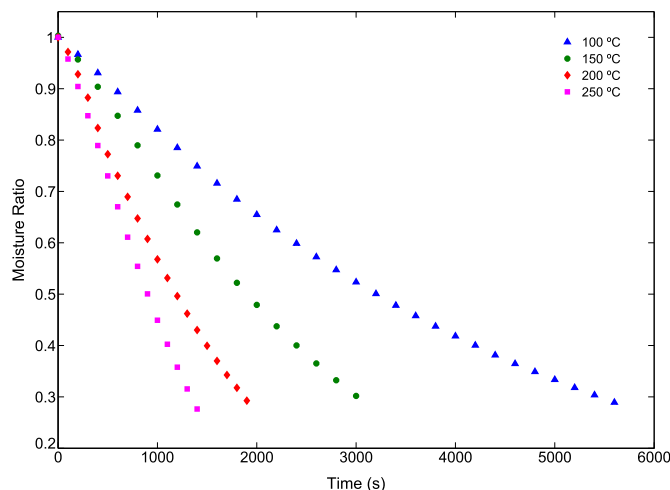


Fig. 3. Drying curves for  $L = 20$  mm at different temperatures with constant air velocity,  $v = 1$  m/s.

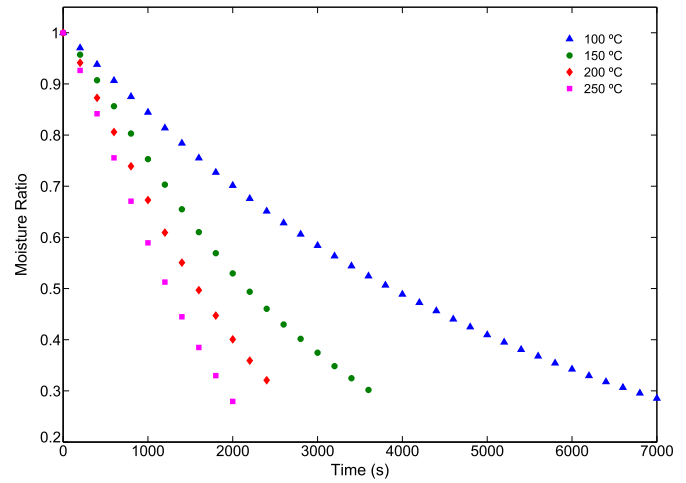


Fig. 4. Drying curves for  $L = 30$  mm at different temperatures with constant air velocity,  $v = 1$  m/s.

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

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. The negative sign was included to indicate the drying rate with positive values.

The drying rate curves can be fitted from the derivate with respect to time of the models of Table 1. Nevertheless, although the four best fit models of drying curve presented results above  $R^2 = 0.998$ , when their derivate functions were obtained, the errors made were considerable. Fig. 5 shows the experimental drying rate and the four best fit models versus moisture ratio for the test of 100 °C of temperature and 10 mm of sample thickness. As can be seen, the mathematical model which offered the best fit in the drying curves was the best fit in the drying rate curves as well, Two Term Gaussian.

Figs. 6–8 show the experimental drying rate curves and their approximations with the Two Term Gaussian model for each of the thicknesses: 10, 20 and 30 mm, respectively. Results indicate that the drying rate is intimately connected with the drying temperature and the sample thickness. There are high drying rates when

Table 1  
Mathematical models of the drying curves.

Model name	Equation	References
Lewis	$XR = \exp(-kt)$	[20]
Page	$XR = \exp(-kt^n)$	[21]
Modified Page	$XR = \exp(-(kt)^n)$	[22]
Henderson and Pabis	$XR = a \cdot \exp(-kt)$	[23]
Logarithmic	$XR = a \cdot \exp(-kt) + c$	[13]
Wang and Singh	$XR = 1 + at + bt^2$	[24]
Two term	$XR = a \cdot \exp(-k_0t) + c \cdot \exp(-k_1t)$	[25]
Two term exponential	$XR = a \cdot \exp(-kt) + (1 - a) \cdot \exp(-kat)$	[26]
Approach of Diffusion	$XR = a \cdot \exp(-kt) + (1 - a) \cdot \exp(-kbt)$	[27]
Modified Henderson and Pabis	$XR = a \cdot \exp(-kt) + b \cdot \exp(-k_2t) + c \cdot \exp(-k_3t)$	[19]
Midilli et al.	$XR = a \cdot \exp(-kt^n) + bt$	[28]
Yun et al.	$XR = a + bt + ct^2/1 + dt + et^2$	[29]
Two term Gaussian	$XR = a \cdot \exp[-((t - b)/c)^2] + d \cdot \exp[-((t - e)/f)^2]$	Present work

**Table 2**  
Fit values of the mathematical models in each experiment,  $R^2$  and RMSE.

Mathematical model	$T$ (°C)	Sample thickness					
		$L = 10$ mm		$L = 20$ mm		$L = 30$ mm	
		$R^2$	RMSE	$R^2$	RMSE	$R^2$	RMSE
Lewis	100	0.987114	0.024637	0.998546	0.008	0.999742	0.003308
	150	0.965110	0.040503	0.983932	0.027523	0.993971	0.016644
	200	0.95409	0.04765	0.990925	0.019756	0.986919	0.024244
	250	0.950947	0.049019	0.968336	0.039168	0.980718	0.030844
Page	100	0.999949	0.001548	0.999859	0.002489	0.999867	0.002376
	150	0.99993	0.001803	0.999929	0.001826	0.999726	0.003545
	200	0.99995	0.001572	0.998141	0.008944	0.999962	0.001306
	250	0.999844	0.002763	0.999742	0.003535	0.999897	0.002248
M. Page	100	0.999949	0.001548	0.999859	0.002489	0.999867	0.002376
	150	0.99993	0.001803	0.999929	0.001826	0.999726	0.003545
	200	0.99995	0.001572	0.998141	0.008944	0.999962	0.001306
	250	0.999844	0.002763	0.999742	0.003535	0.999897	0.002248
Henderson and Pabis	100	0.995672	0.014279	0.999735	0.003416	0.999941	0.001573
	150	0.985325	0.026275	0.994617	0.015933	0.998453	0.008431
	200	0.981012	0.030658	0.993280	0.017004	0.995003	0.014987
	250	0.977943	0.032888	0.985979	0.026073	0.991982	0.019894
Logarithmic	100	0.999317	0.005671	0.999851	0.002559	0.999948	0.001484
	150	0.998574	0.008192	0.999060	0.006657	0.999357	0.005436
	200	0.998129	0.009626	0.999484	0.004711	0.999431	0.005056
	250	0.998166	0.009486	0.999116	0.006547	0.999297	0.005891
Wang and Singh	100	0.999569	0.004506	0.999816	0.002842	0.999654	0.003834
	150	0.99864	0.008	0.999355	0.005516	0.999701	0.003706
	200	0.998162	0.009541	0.999609	0.004102	0.999643	0.004003
	250	0.998174	0.009468	0.999186	0.006284	0.999497	0.004979
Two term	100	0.992431	0.018891	0.999984	0.000831	0.999948	0.001481
	150	0.999952	0.001495	0.989663	0.022086	0.996754	0.012216
	200	0.966741	0.040613	0.999713	0.00351	0.984757	0.026187
	250	0.959972	0.044355	0.999816	0.002982	0.990431	0.021744
Two term exponential	100	0.999804	0.003036	0.999676	0.003774	0.999813	0.002812
	150	0.999865	0.00252	0.999819	0.002916	0.999382	0.005328
	200	0.999723	0.003701	0.998869	0.006974	0.999969	0.001171
	250	0.99932	0.005771	0.999627	0.004247	0.999949	0.001585
Approach of Diffusion	100	0.999942	0.001650	0.999984	0.000835	0.999954	0.001388
	150	0.999882	0.00235	0.999976	0.001041	0.999979	0.000979
	200	0.999741	0.003579	0.998914	0.006835	0.999983	0.000871
	250	0.999349	0.005648	0.999663	0.004038	0.99996	0.0014
M. Henderson and Pabis	100	0.999213	0.006091	0.993138	0.017390	0.992621	0.017715
	150	0.999207	0.006113	0.999970	0.00117	0.999066	0.006553
	200	0.99897	0.007153	0.99277	0.017657	0.998634	0.007839
	250	0.964289	0.041942	0.984707	0.027268	0.993343	0.018144
Midilli et al.	100	0.999962	0.001332	0.999954	0.001414	0.999958	0.001323
	150	0.999969	0.0012	0.999975	0.001085	0.999958	0.001389
	200	0.999953	0.001516	0.999539	0.00445	0.999975	0.001055
	250	0.999945	0.001635	0.999869	0.002518	0.99995	0.001565
Yun et al.	100	0.999982	0.000898	0.99999	0.000635	0.999993	0.000526
	150	0.999983	0.000884	0.999989	0.000709	0.999986	0.000794
	200	0.999972	0.001164	0.999817	0.002808	0.999991	0.000642
	250	0.999986	0.000829	0.999991	0.000651	0.999989	0.000722
Two term Gaussian	100	0.999985	0.000823	0.999991	0.000616	0.999996	0.000366
	150	0.999986	0.000799	0.999993	0.000581	0.999987	0.000757
	200	0.999982	0.000942	0.999858	0.002472	0.999992	0.000625
	250	0.999989	0.000719	0.999992	0.00064	0.99999	0.000704

the drying temperature is increased and the sample thickness is decreased.

The drying rate curves show the dependency with the moisture ratio. The variation of the drying rate with respect to the moisture ratio can be explained from the stages proposed in the drying of the alpeorajo [17], although there are some differences due to the nature of the products. These stages are: warming-up period, first

falling rate period and second falling rate period. There is no constant rate period.

In the warming-up period, the entire sample surface is totally wet, and the test starts at room temperature. As the test proceeds, the solid begins to heat and the drying rate increases rapidly (higher vapor pressures) until a maximum value is reached. From this value, the drying rate begins to decrease. In the tests performed

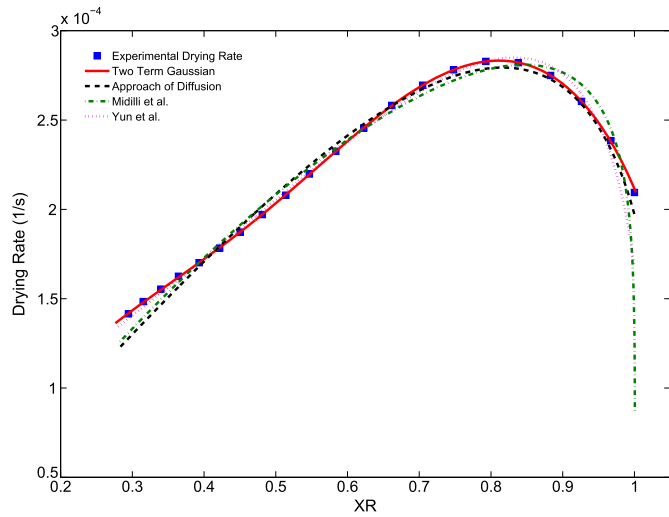


Fig. 5. Experimental drying rate and the four best fit mathematical models.  $T = 100\text{ }^{\circ}\text{C}$ ,  $L = 10\text{ mm}$  and  $v = 1\text{ m/s}$ .

at  $100\text{ }^{\circ}\text{C}$ , this period is appreciated when the sample thickness is  $10\text{ mm}$ , can hardly be differentiated when the sample thickness is  $20\text{ mm}$  (it appears that there is a constant drying rate) and does not exist when the sample thickness is  $30\text{ mm}$ . This can be explained taking into account the concept of specific surface (sample surface in contact with the drying air divided by sample volume). This value is greater as the sample thickness is lower. For remaining temperatures and sample thicknesses, this period can be clearly identified. In fact, warming-up period grows as the drying temperature increases, regardless of the sample thickness. This means that the maximum value of drying rate moves in downward direction of the moisture ratio.

The first falling rate period appears at low temperatures mainly by two reasons. First, the sample surface still has moisture content in contact with the drying air. And second, there is continuous movement of moisture content from within the porous medium to the surface [30]. In the drying of the olive stone, the two falling rate periods (first and second) can hardly be differentiated with each other. The first falling rate period can

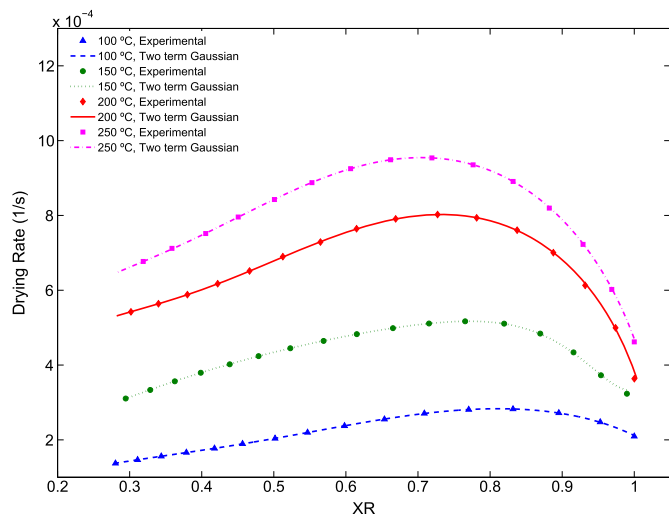


Fig. 6. Drying rate versus moisture ratio for  $L = 10\text{ mm}$  at different temperatures with constant air velocity,  $v = 1\text{ m/s}$ .

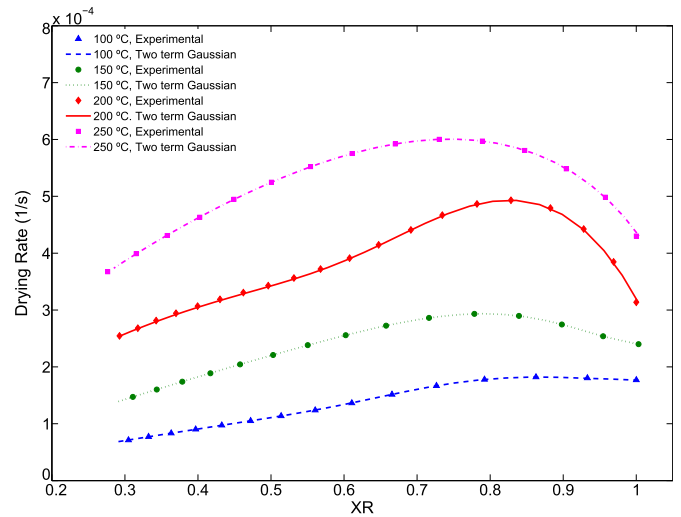


Fig. 7. Drying rate versus moisture ratio for  $L = 20\text{ mm}$  at different temperatures with constant air velocity,  $v = 1\text{ m/s}$ .

be seen for temperatures  $\leq 200\text{ }^{\circ}\text{C}$  and sample thicknesses  $\leq 20\text{ mm}$ . For values above  $200\text{ }^{\circ}\text{C}$  and  $20\text{ mm}$  this stage does not exist.

When the second falling rate period is manifested, the sample surface is totally dry. The diffusion is the predominant phenomenon in this stage. The vapor and liquid migration is produced from inside the sample to the surface. In this stage, the drying rate drops rapidly as the sample thickness grows.

### 3.3. Effective diffusivity and energy activation

The effective diffusivity values can be obtained for each of the twelve tests carried out in the drying of the olive stone. For that, the partial differential equation, Fick's second law of diffusion (Eq. (3)), should be solved. The solution calculated by Crank [31] for the one-dimensional mass transport in infinite slab geometry, in spherical coordinates, is considered (Eq. (4)). However, some suppositions should be done: negligible shrinkage, migration by diffusion and constant temperature and diffusion coefficients.

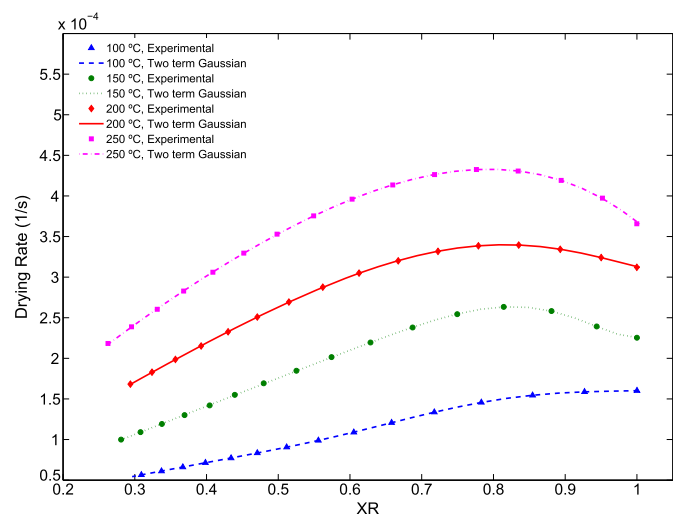


Fig. 8. Drying rate versus moisture ratio for  $L = 30\text{ mm}$  at different temperatures with constant air velocity,  $v = 1\text{ m/s}$ .

**Table 3**  
Effective Diffusivity values for each test.

Temperature (°C)	Sample thickness (mm)											
	10				20				30			
	100	150	200	250	100	150	200	250	100	150	200	250
$D_{\text{eff}} \times 10^8 \text{ (m}^2/\text{s)}$	0.4	0.77	1.2	1.45	0.91	1.68	2.62	3.76	1.64	3.13	4.47	5.98

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

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

where  $D_{\text{eff}}$  is the effective diffusivity ( $\text{m}^2/\text{s}$ ),  $L$  is thickness of slab (m) and  $t$  is the drying time (s). This mathematical model considers the mass transfer (moisture ratio) by diffusion phenomena. Although the drying rate in the olive stone presents a warming-up period, the vast majority of the drying process is produced in the falling rate period where the mass transfer is by diffusion. Eq. (4) can be simplified to only the first term of the series when the drying times are long (Eq. (5)):

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

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

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

Table 3 indicates the effective diffusivity values for each test. The coefficient of determination in the approximation of the linear function of the curves  $\ln(\text{XR})$  versus time was higher than 0.98. The effective diffusivity values increase when the drying air temperatures and the sample thicknesses increase as well. Although does not exist results in the literature about the drying of the olive stone, similar values can be found for olive cake with different conditions of drying air temperature, sample thickness and drying air velocity.

Thus, Akgun and Doymaz [13] obtained values between  $4.95 \cdot 10^{-10}$  -  $1.42 \cdot 10^{-9} \text{ m}^2/\text{s}$  for temperatures ranging from 50 to 110 °C, a sample thickness of 8 mm and a velocity of 1.2 m/s. Gögüs and Maskan [14] calculated, for temperature between 60 and 80 °C, sample thickness of 6, 9 and 12 mm and velocity of 1.5 m/s, effective diffusivity values between  $1.84 \cdot 10^{-7}$  -  $3.94 \cdot 10^{-7} \text{ m}^2/\text{s}$ . Meziane [15] found values that varied between  $0.68 \cdot 10^{-7}$  -  $2.15 \cdot 10^{-7} \text{ m}^2/\text{s}$  for tests carried out at temperatures and sample thicknesses between 50 and 80 °C and 41, 52 and 63 mm, respectively, with a drying air velocity of 1 m/s.

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

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

where  $D_0$  is the pre-exponential factor ( $\text{m}^2/\text{s}$ ),  $R$  is the universal gas constant ( $\text{J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$ ),  $T$  is the absolute temperature (K) and  $E_a$  is the activation energy ( $\text{J} \cdot \text{mol}^{-1}$ ). The activation energy was found out for each sample thickness. Fig. 9 shows the function  $\ln D_{\text{eff}}$  versus  $1/T$  for each sample thickness value: 10, 20 and 30 mm. The  $\ln D_{\text{eff}}$  decreases linearly with the reciprocal of the temperature. Table 4 indicates values obtained of the activation energy after analyzing the slope of the linear function of approximation. The coefficients of determination values are shown too.

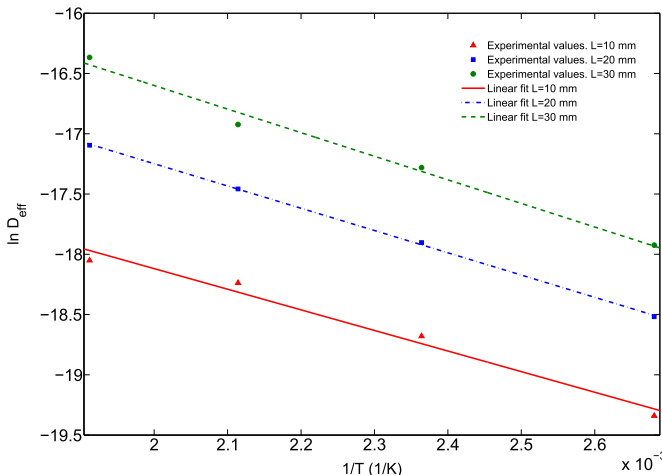
With respect to the activation energy, similar values can be found for olive cake. Akgun and Doymaz [13] obtained a value of 17,970 J/mol for a sample thickness of 8 mm and drying air velocity of 1.2 m/s. Meziane [15], with a drying air velocity in his experiments of 1 m/s, obtained values of 34,050, 36,840, 38,100 J/mol for 41, 52 and 63 mm of sample thickness, respectively. Gögüs and Maskan [14], for air velocity of 1.5 m/s and sample thicknesses of 6, 9 and 12 mm calculated values of 25,400, 25,700, 29,200 J/mol, respectively.

**4. Conclusion**

Drying kinetics of olive stone with olive pulp was studied from different conditions of temperature and sample thickness. Thirteen mathematical models were analyzed to fit the drying curves. The drying rate curves were fitted with the derivate of the models which obtained the best results of fit in the drying curves. Two term Gaussian model presented the best results. The effective diffusivity values were calculated for each test between  $3.98 \cdot 10^{-9}$  -  $5.97 \cdot 10^{-8} \text{ m}^2/\text{s}$ . Finally, the activation energy was estimated for each sample thickness: 14,208 J/mol ( $L = 10 \text{ mm}$ ), 15,356 J/mol ( $L = 20 \text{ mm}$ ) and 16,270 J/mol ( $L = 30 \text{ mm}$ ).

**Table 4**  
Activation Energy values for different sample thicknesses.

	Sample thickness (mm)		
	10	20	30
Activation energy (J/mol)	14,208	15,356	16,270
$R^2$	0.9801	0.995	0.9884



**Fig. 9.** Arrhenius-type relationship between effective diffusivity logarithmic and the reciprocal of absolute temperature for different thicknesses.  $v = 1 \text{ m/s}$ .

## Acknowledgments

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## A vital stage in the large-scale production of biofuels from spent coffee grounds: The drying kinetics



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### ABSTRACT

Spent coffee grounds are being consolidated as one of the most abundant bioresources in the world for use as green energy. Biodiesel, bioethanol, bio-oil and fuel pellet are biofuels derived of this waste. To get them, spent coffee grounds need to be dried due to their high moisture content. This work analyzes their drying kinetics from isothermal drying experiments in a convective dryer at different temperatures: 100, 150, 200 and 250 °C, and sample thicknesses: 5, 10, 15 and 20 mm. Drying curves were fitted with the main mathematical models in the drying of agricultural products where the Two Term Gaussian model got the best results of fit. Drying rate was calculated and analyzed. Effective moisture diffusivities were calculated in a range between  $1.29 \cdot 10^{-9}$  to  $28.8 \cdot 10^{-9}$  m<sup>2</sup>/s. Activation energies were 12.29, 12.78, 15.18 and 16.87 kJ/mol for each sample thickness: 5, 10, 15, and 20 mm, respectively.

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### 1. Introduction

Coffee is one of the agricultural products intended mainly for beverage industry. Coffee plants are cultivated in more than 70 countries with an average annual production of 8 million tons in the world [1]. 50% of the world coffee production is destined for elaborating the instant coffee, while the other 50% is used in cafeterias, restaurant and homes to make directly beverages of the ground coffee [2]. Spent coffee grounds are the waste obtained in the coffee industry. This waste is an environmental problem due to its high biochemical oxygen demand (BOD) formed by toxic organic compounds such as: caffeine, polyphenols and tannins [3]. At present, spent coffee grounds are been used as horticultural production, animal feed, fertilizers, activated carbons and biochar [4].

However, in recent years spent coffee grounds are positioned as one of the biomass products more attractive due to the generation of waste in the coffee industry [5]. Four main types of biomass products are obtained from this residue: biodiesel, bioethanol, bio-oil and fuel pellet [6,7]. Biodiesel is produced using transesterification methods from oil contained in the spent coffee grounds, about 15% [8]. Defatted solid wastes are used to obtain bioethanol and bio-oil from processes like organic fermentation and slow pyrolysis, respectively. The

leftover solid wastes (without lipids and sugars) are utilized to make fuel pellet, which is used as biofuel to generate electric and thermal energy. Fuel pellet has a net calorific value of 25 240 kJ/kg at 0% moisture content [9].

Spent coffee grounds have a high moisture content, between 55 and 80% depending on the process used. Generally, the instant coffee industry obtains moisture contents in the spent coffee grounds higher than those generated by the coffee bars from ground coffee. The drying process of the spent coffee grounds is necessary to make biofuels. First, in the production process of biodiesel, bio-oil and bioethanol, elimination of the moisture content is essential for the solvent becomes effective [10–12]. And second, when spent coffee grounds are directly utilized as fuel pellet, should be dried up to the equilibrium moisture content to improve the energetic characteristics as biofuel [13,14]. Furthermore, the drying makes possible to minimize the storage, packing and transport costs. Fig. 1 shows the process to obtain biofuels from spent coffee grounds.

Although there are some specific studies related to the drying and roasted of coffee [15,16], there are no works in the field of drying of the spent coffee grounds, where the nature of the particles is completely different from the coffee beans.

This paper presents a study about the drying kinetics of the spent coffee grounds in a convective dryer. Sixteen isothermal drying tests were carried out at different drying air temperatures and sample thicknesses with constant drying air velocity. Drying curves were fitted with the main mathematical models in the drying of agricultural

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## 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 moisture 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 ( $^{\circ}C, K$ )
$v$	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 ( $s^{-1}$ )

products. Two Term Gaussian model obtains the best results of fit. The experimental drying rate and its mathematical function of fit were analyzed and calculated. The effective moisture diffusivity values were found out for each test. Finally, the activation energies were found for each sample thickness.

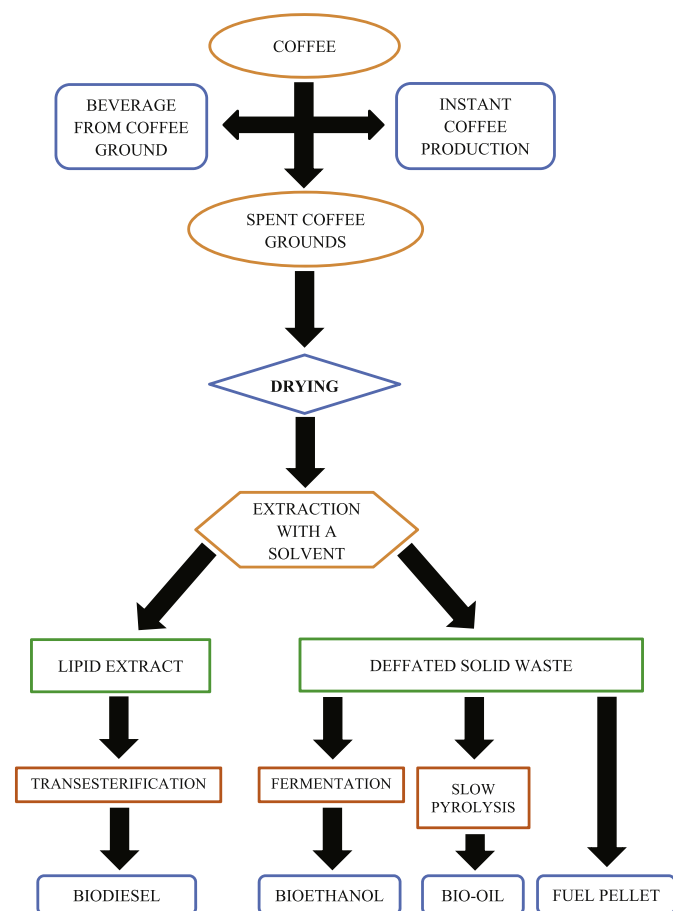


Fig. 1. Stages in the biofuel production from spent coffee grounds.

## 2. Materials and methods

### 2.1. Materials

Spent coffee ground samples (grounds obtained after brewing) were kindly provided by a cafeteria and a restaurant in the province of Jaen (Spain). To know the initial moisture content, the samples were dried in an oven (Memmert GmbH + Co.KG, SNB 167 Model 100, Germany) at 105 °C for 24 h. Drying samples were performed in triplicate. An average moisture content of  $58.5 \pm 1.5\%$  (wet basis) was found. The same procedure was applied to obtain the equilibrium moisture content which was estimated at  $7 \pm 0.5\%$  (wet basis) for a relative humidity of 50% and a temperature of 20 °C of surrounding air.

### 2.2. Experimental set-up and procedure

Drying experiments were carried out in a convective dryer (Fig. 2). The drying equipment consists of a blower, electric resistances and a tunnel of 2 m of length with thermal insulation and 0.15 m of square section. To achieve the desired temperature in each test, air was passed through a group of three independent resistances: 9 kW (first stage), 18 kW (second stage) and 18 kW (third stage), 45 kW in total. To control the constant temperature in each test, a PID (Proportional-Integral-Differential) controller acted on 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. Once the test conditions were correct, the sample was introduced (with the corresponding thickness in each test) into the tunnel in a steel basket of 7 cm of square section. The basket was placed over a precision balance (Blauscal AH1200) with an error of  $\pm 0.01$  g. It was connected to personal computer by USB port. Software measured the variation of mass every second and stored the test information in files. Drying experiments were stopped when the equilibrium moisture content of the sample was approximately accomplished. However, to obtain the exact moisture content, when the experiments were finished, samples were dried until 0% moisture content in the oven at 105 °C for 24 h.

Sixteen experiments were performed. For each sample thickness: 5, 10, 15, 20 mm, four tests with constant drying air temperatures at 100, 150, 200 and 250 °C were carried out. The drying air velocity was established in  $1 \pm 0.1$  m/s.

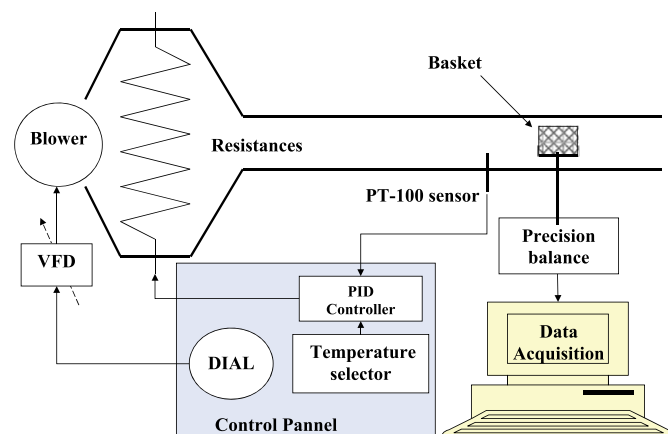


Fig. 2. Convective dryer scheme.

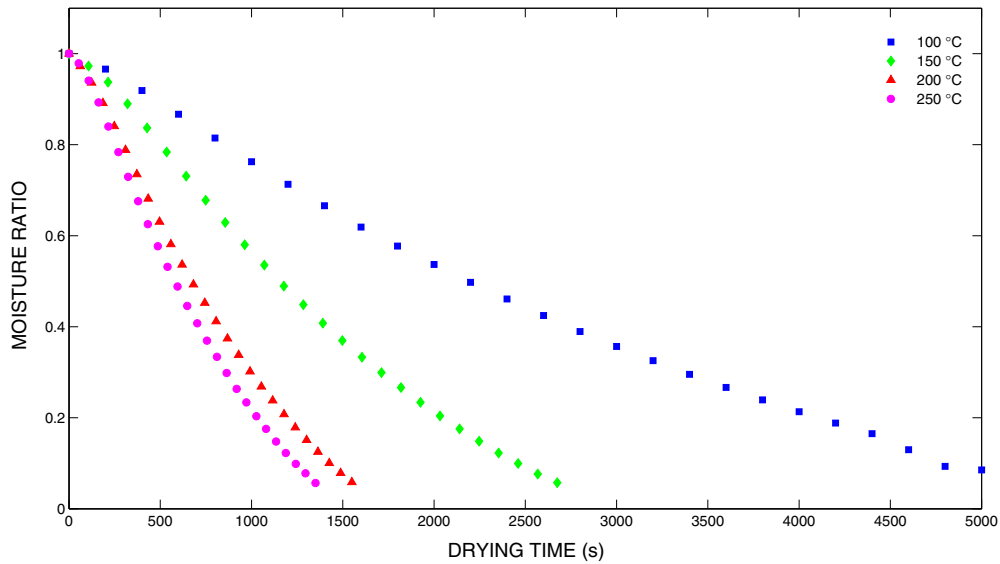


Fig. 3. Drying curves at different temperatures with constant air velocity,  $v = 1$  m/s.  $L = 5$  mm.

### 3. Results and discussion

#### 3.1. Drying curves

The dimensionless moisture ratio was plotted with respect to drying time,  $XR = f(t)$ . This variable can be expressed as (Eq. (1)):

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

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. However, when the equilibrium moisture content value,  $X_e$ , is small with respect to other variables, the dimensionless moisture ratio can be expressed as  $XR = X_t / X_0$ .

Figs. 3–6 show the spent coffee ground drying curves for each sample thickness: 5, 10, 15 and 20 mm, respectively. Four drying curves belonging to the temperatures of 100, 150, 200 and 250 °C were represented for each sample thickness in each figure. The moisture content decreased continuously with the drying time. As can be seen,

the drying time increases when the drying air temperature decreases. In fact, the drying time at 100 °C is approximately four times greater than at 250 °C, regardless of the sample thickness. The drying time increases when the sample thickness increases as well. This means that the ideal drying of spent coffee grounds begins by cutting back the sample thickness and rising the drying air temperature. Nevertheless, the drying air temperature should be controlled in the drying process. The combustion phenomenon appears in tests carried out at 250 °C, regardless of the sample thickness. The samples began to burn when the moisture content closed to equilibrium moisture content. Drying curves at 250 °C do not show the mass loss caused by the combustion because the drying tests were stopped at the beginning of this chemical reaction. However, moments before combustion, the loss of volatiles was appreciated. For temperatures  $\leq 200$  °C, the loss of volatiles was not appreciated regardless of the moisture content.

To date, there are no physic models to describe exactly the drying process. Drying is a complex physic process which depends on diffusion and convection phenomena. It is very important to model the stages in the drying process effectively. For that, drying curves are usually fitted with empirical, semi-empirical or semi-theoretical mathematical

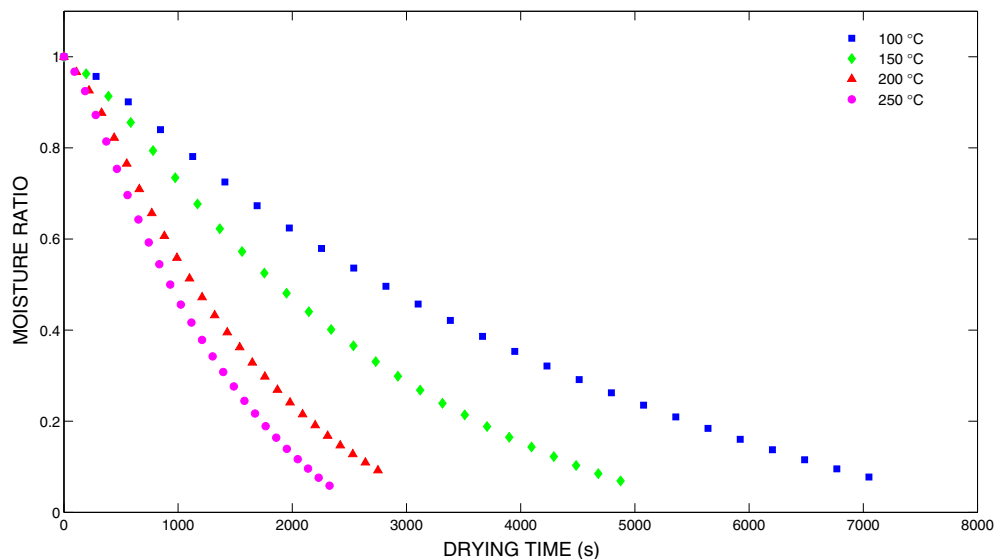


Fig. 4. Drying curves at different temperatures with constant air velocity,  $v = 1$  m/s.  $L = 10$  mm.

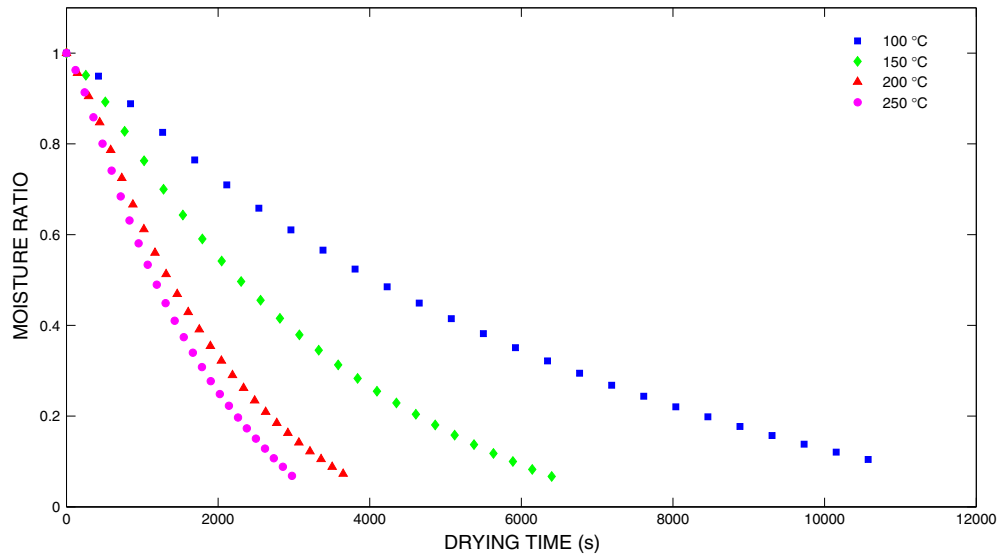


Fig. 5. Drying curves at different temperatures with constant air velocity,  $v = 1$  m/s.  $L = 15$  mm.

functions. Drying curves were fitted with the main mathematical models in the drying of agricultural products. Table 1 indicates the mathematical models of fit used in this work.

Ten mathematical models were used to approximate the drying curves by non-linear regression analysis. The criteria such as coefficient of determination,  $R^2$ , and root mean square error, RMSE, were used to verify the quality of fit. Table 2 shows these values for the different mathematical models applied to each drying curve. To evaluate the fits, six significant figures were used due to excellent approximations of these models. The mathematical models which presented the best results of fit were: Two Term Gaussian, Midilli et al., Two Term and Logarithmic. However, Two Term Gaussian model is presented as the best fit model for drying curves of the spent coffee grounds with an average value of  $R^2$  and RMSE of 0.999965 and 0.00145, respectively. The models proposed by Midilli et al., Two Term and Logarithmic obtained an average value of  $R^2$  and RMSE of 0.999842 and 0.003369, 0.999404 and 0.005637, and 0.999345 and 0.006614, respectively.

### 3.2. Drying rate

The drying rate indicates the amount of evaporated water per time unit. This variable is very important to optimize and control the drying process. The drying rate of the spent coffee grounds can be expressed as the moisture content variation with respect to time (Eq. (2)):

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

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.

The drying rate can be plotted with respect to drying time, but drying time is usually replaced by the dimensionless moisture ratio. Thus, the dependence of time is removed and the drying rate can be expressed in function of dimensionless moisture ratio, drying air

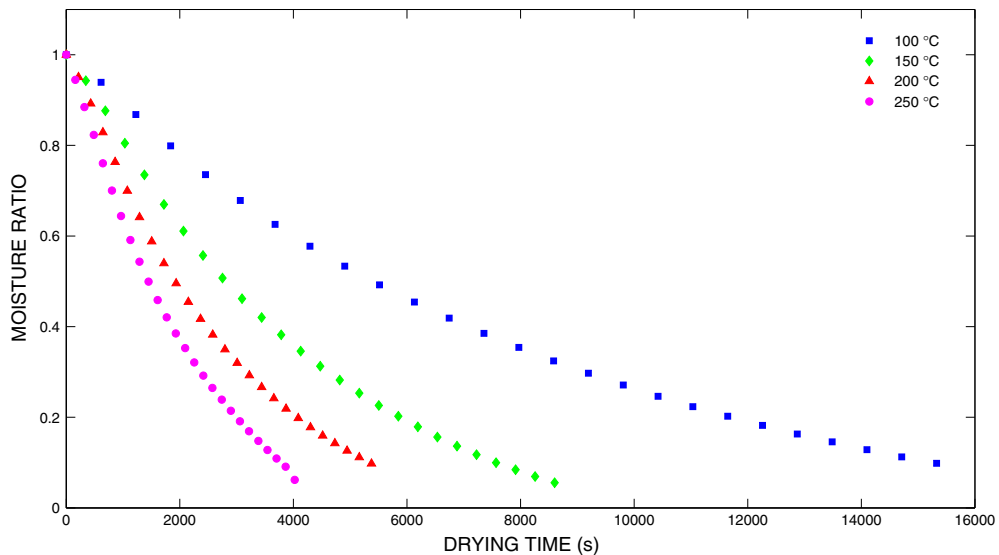


Fig. 6. Drying curves at different temperatures with constant air velocity,  $v = 1$  m/s.  $L = 20$  mm.

**Table 1**  
Mathematical models applied to spent coffee ground drying kinetics.

Model name	Equation	References
Lewis	$XR = \exp(-kt)$	[17]
Page	$XR = \exp(-kt^n)$	[18]
Modified page	$XR = \exp(-(kt)^n)$	[19]
Henderson and Pabis	$XR = a \cdot \exp(-kt)$	[20]
Logarithmic	$XR = a \cdot \exp(-kt) + c$	[21]
Wang and Singh	$XR = 1 + at + bt^2$	[22]
Two term	$XR = a \cdot \exp(-k_0t) + c \cdot \exp(-k_1t)$	[23]
Approach of diffusion	$XR = a \cdot \exp(-kt) + (1 - a) \cdot \exp(-kbt)$	[24]
Midilli et al.	$XR = a \cdot \exp(-kt^n) + bt$	[25]
Two term Gaussian	$XR = a \cdot \exp\left[-\left(\frac{t-b}{c}\right)^2\right] + d \cdot \exp\left[-\left(\frac{t-e}{f}\right)^2\right]$	[26]

temperature, sample thickness and drying air velocity. Figs. 7–10 show the drying rate of the spent coffee grounds in function of dimensionless moisture ratio and drying air temperature for each of the thicknesses: 5, 10, 15 and 20 mm, respectively, when drying air velocity is equal to 1 m/s. As a result, there are high drying rates when the drying temperature is raised and the sample thickness is reduced.

The variation of the drying rate, at constant temperature, with respect to the dimensionless moisture ratio can be explained from drying stages [27,28]. In the drying of spent coffee grounds four stages are differentiated: *warming-up period*, *first falling rate period*, *second*

*falling rate period* and *shrinkage period*. As can be seen, there is no constant drying rate period in the drying of spent coffee grounds.

The *warming-up period* appears for dimensionless moisture ratio values which lie between 0.75 and 1. This stage is mainly governed by convection phenomenon. The entire sample surface of the spent coffee grounds is totally wet at the beginning of the experiments. When the test starts at room temperature, the sample begins to heat and the drying rate rises quickly until a maximum value is reached. This is due to the higher vapor pressures which are caused by high drying air temperature, above 100 °C. This explains why this period does not exist at temperature below 100 °C. In fact, when the drying air temperature is about 100 °C, this stage can hardly be differentiated for the sample thicknesses of 5, 10 and 15 mm and does not exist when the sample thickness is 20 mm (Fig. 10). The specific surface (sample surface in contact with the drying air divided by sample volume) can explain this phenomenon, since this value is lower as the sample thickness is greater. For the remaining temperatures and sample thicknesses, *warming-up period* grows as the drying temperature increases and the sample thickness decreases. This means that the maximum value of drying rate moves in a downward direction of the dimensionless moisture ratio.

The *first falling rate period* is governed by convection and diffusion phenomena. This period occurs mainly because the sample surface still has moisture content in contact with the drying air and there is water movement from inside of the sample to the surface [29]. The

**Table 2**  
Statistics criteria to evaluate the quality of fit of the mathematical models in each experiment, R<sup>2</sup> and RMSE values.

Mathematical model	T (°C)	Sample thickness							
		L = 5 mm		L = 10 mm		L = 15 mm		L = 20 mm	
		R <sup>2</sup>	RMSE	R <sup>2</sup>	RMSE	R <sup>2</sup>	RMSE	R <sup>2</sup>	RMSE
Lewis	100	0.971925	0.045390	0.982064	0.035993	0.992241	0.022717	0.996713	0.014620
	150	0.952396	0.063247	0.976087	0.043227	0.985963	0.032333	0.989783	0.027602
	200	0.951738	0.063585	0.971853	0.046357	0.980330	0.038849	0.992132	0.023431
	250	0.952856	0.063032	0.965480	0.053302	0.975165	0.043956	0.989064	0.027931
Page	100	0.997339	0.013974	0.997937	0.012205	0.999137	0.007573	0.999286	0.006813
	150	0.998301	0.011950	0.999255	0.007630	0.999063	0.008350	0.999166	0.007882
	200	0.998247	0.012122	0.999645	0.005203	0.999504	0.006164	0.999705	0.004537
	250	0.998414	0.011563	0.998779	0.010023	0.999030	0.008686	0.998954	0.008638
Modified page	100	0.997339	0.013974	0.997937	0.012205	0.999137	0.007573	0.999286	0.006813
	150	0.998301	0.011950	0.999255	0.007630	0.999063	0.008350	0.999166	0.007882
	200	0.998247	0.012122	0.999645	0.005203	0.999504	0.006164	0.999705	0.004537
	250	0.998414	0.011563	0.998779	0.010023	0.999030	0.008686	0.998954	0.008638
Henderson and Pabis	100	0.984942	0.033244	0.990248	0.026541	0.996177	0.015945	0.998190	0.010848
	150	0.975731	0.045167	0.989223	0.029022	0.993427	0.022125	0.995207	0.018907
	200	0.975236	0.045562	0.987885	0.030418	0.991239	0.025930	0.997404	0.013460
	250	0.977075	0.043971	0.983326	0.037052	0.988492	0.029927	0.994372	0.020039
Logarithmic	100	0.999622	0.005264	0.999875	0.002996	0.999907	0.002482	0.999872	0.002885
	150	0.998749	0.010253	0.999231	0.007753	0.999705	0.004685	0.999786	0.003990
	200	0.998662	0.010591	0.998638	0.010199	0.999204	0.007814	0.999526	0.005752
	250	0.998738	0.010317	0.998937	0.009356	0.999311	0.007321	0.999757	0.004163
Wang and Singh	100	0.999542	0.005792	0.999541	0.005752	0.999167	0.007440	0.998270	0.010608
	150	0.999183	0.008287	0.999523	0.006100	0.999201	0.007712	0.998636	0.010084
	200	0.999072	0.008822	0.999258	0.007527	0.999493	0.006238	0.998898	0.008767
	250	0.999159	0.008421	0.999408	0.006979	0.999501	0.006227	0.999046	0.008248
Two term	100	0.902333	0.084684	0.999876	0.002991	0.999917	0.002339	0.999948	0.001830
	150	0.998844	0.009857	0.999309	0.007346	0.999735	0.004440	0.999797	0.003883
	200	0.998752	0.010232	0.998774	0.009677	0.999325	0.007197	0.999575	0.005445
	250	0.998791	0.010097	0.999056	0.008816	0.999370	0.007002	0.999758	0.004151
Approach of diffusion	100	0.996704	0.015552	0.997821	0.012546	0.999240	0.007106	0.999423	0.006125
	150	0.996036	0.018252	0.998881	0.009348	0.998916	0.008983	0.999122	0.008090
	200	0.995971	0.018377	0.999341	0.007093	0.999309	0.007281	0.999594	0.005321
	250	0.996420	0.017374	0.997763	0.013570	0.998606	0.010412	0.998997	0.008458
Midilli et al.	100	0.999719	0.004535	0.999906	0.002602	0.999922	0.002270	0.999666	0.004661
	150	0.999918	0.002619	0.999871	0.003164	0.999861	0.003216	0.999878	0.003009
	200	0.999868	0.003324	0.999858	0.003288	0.999882	0.002998	0.999741	0.004248
	250	0.999842	0.003643	0.999854	0.003466	0.999847	0.003441	0.999837	0.003408
Two term Gaussian	100	0.999785	0.003969	0.999962	0.001650	0.999980	0.001134	0.999983	0.001032
	150	0.999966	0.001691	0.999981	0.001215	0.999976	0.001325	0.999979	0.001245
	200	0.999975	0.001436	0.999990	0.000835	0.999992	0.000765	0.999992	0.000718
	250	0.999967	0.001651	0.999966	0.001654	0.999973	0.001442	0.999971	0.001427

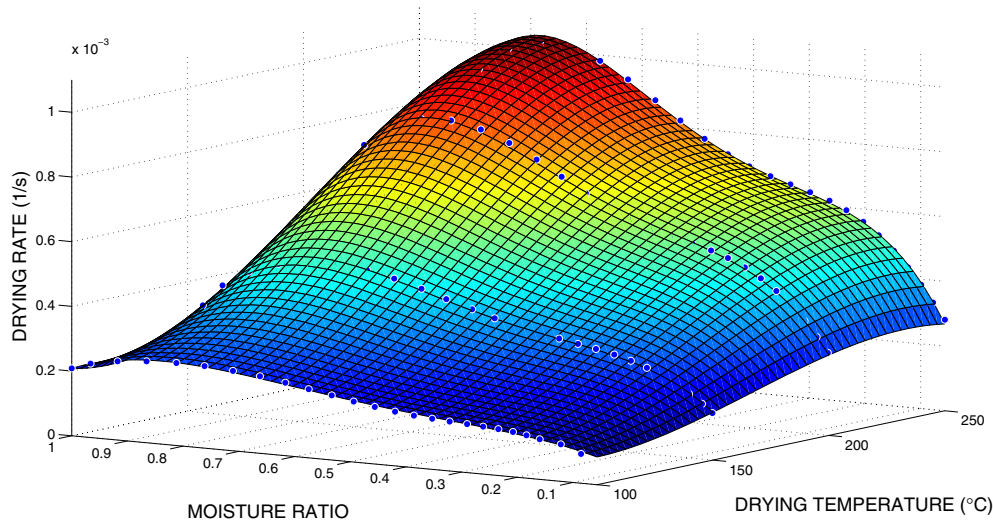


Fig. 7. Drying rate surface as a function of dimensionless moisture ratio and temperature, with constant air velocity,  $v = 1$  m/s.  $L = 5$  mm.

difference between the *first* and *second falling rate periods* is clearly appreciated when the sample thickness is equal to 5 mm. As the sample thickness increases, the two falling rate periods (first and second) can hardly be differentiated with each other. In this case when the temperatures are high, above 200 °C, this period does not exist because it is included in the *warming-up period*. At low temperatures, below 200 °C, the limit between both periods is well-defined.

In the *second falling rate period* the diffusion is the predominant phenomenon in the drying of the spent coffee grounds. In this stage the sample surface is totally dry. The water (vapor and liquid depending on the drying air temperature) migration is produced from inside the sample to the surface through the micro-holes between the spent coffee ground particles.

Finally, in the *shrinkage period*, the drying rate falls quickly as the sample thickness diminishes. In this stage, due to the microscopic nature of spent coffee ground particles, when the moisture content close to equilibrium moisture content, the samples suffer shrinkages [6]. This phenomenon is exacerbated as the sample thickness decreases. In fact, for the samples with thicknesses of 5 and 10 mm, a severe shrinkage provoked by the collapse, by gravity, of the spent coffee

ground particles prevents the fast mass transfers to the outside. However, the shrinkage of the samples with thicknesses of 15 and 20 mm is hardly appreciated.

### 3.3. Effective moisture diffusivity and activation energy

The effective moisture diffusivity values can be found out for each of the sixteen tests carried out in the drying of the spent coffee grounds. The diffusion phenomenon can be explained from Fick's second law of diffusion (Eq. (3)). The solution of this partial differential equation (Eq. (4)), was calculated by Crank [30] for the one-dimensional mass transport in infinite slab geometry, in spherical coordinates. However, some suppositions should be done: constant temperature and diffusion coefficients, migration by diffusion and negligible shrinkage.

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

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

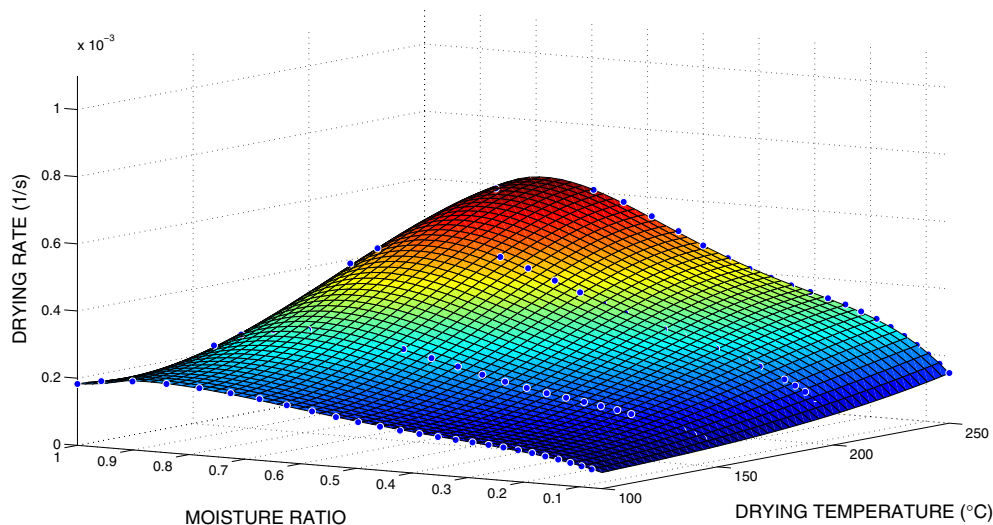


Fig. 8. Drying rate surface as a function of dimensionless moisture ratio and temperature, with constant air velocity,  $v = 1$  m/s.  $L = 10$  mm.

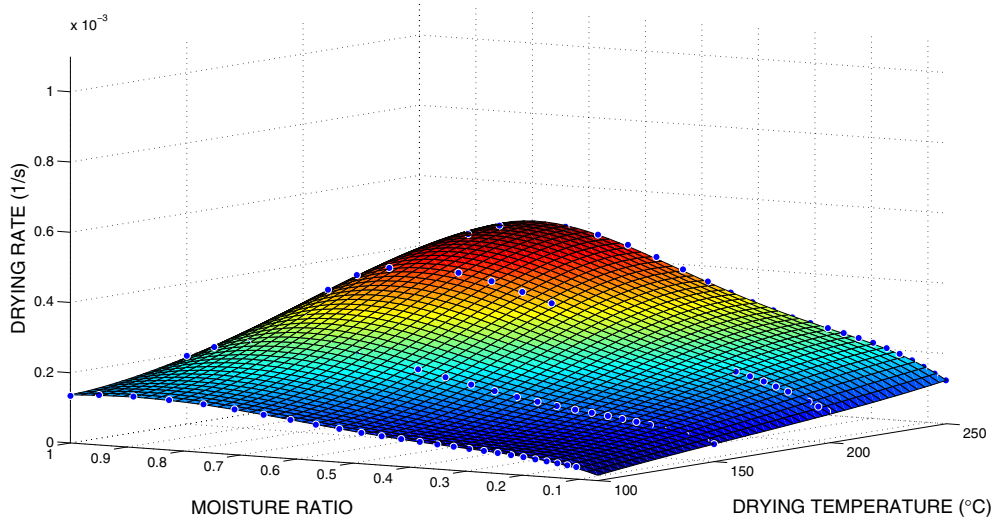


Fig. 9. Drying rate surface as a function of dimensionless moisture ratio and temperature, with constant air velocity,  $v = 1$  m/s.  $L = 15$  mm.

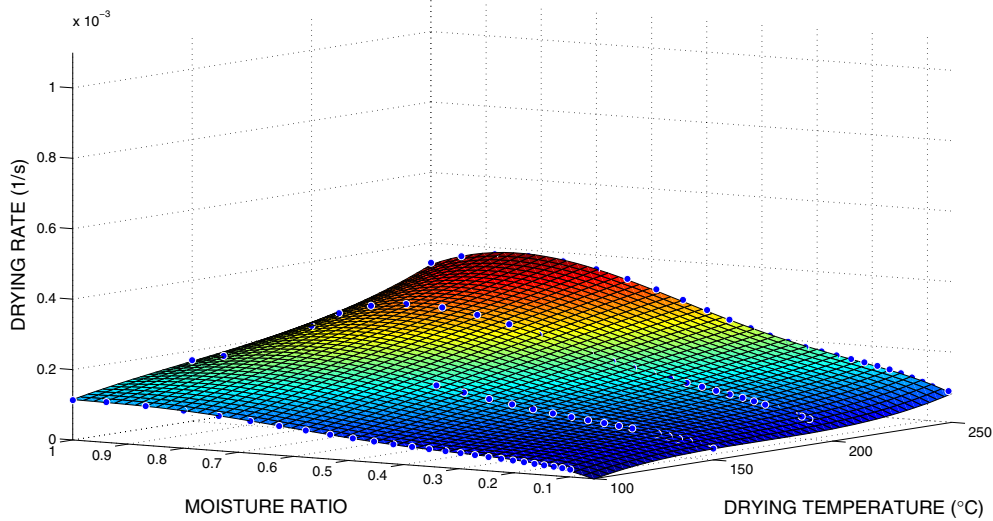


Fig. 10. Drying rate surface as a function of dimensionless moisture ratio and temperature, with constant air velocity,  $v = 1$  m/s.  $L = 20$  mm.

where  $D_{eff}$  is the effective moisture diffusivity ( $m^2/s$ ),  $t$  is the drying time (s) and  $L$  is thickness of slab (m). Although the drying rate in the spent coffee grounds presents a *warming-up period* and a *shrinkage period* in the samples, the vast majority of the drying process is produced in the falling rate period where the mass transfer is by diffusion phenomenon. Eq. (4) can be simplified to only the first term of the series when the drying times are considerable (Eq. (5)):

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

If the logarithm is applied on both sides of the equation, its plot can be represented as a linear function whose slope allows calculating the effective moisture diffusivity as (Eq. (6)):

$$D_{eff} = -\frac{\text{Slope} \cdot L^2}{\pi^2} \tag{6}$$

Table 3 indicates the effective moisture diffusivity values for each test. The coefficient of determination in the approximation of the linear function of the curves  $\ln(XR)$  versus time was higher than 0.97. Fig. 11 shows the effective moisture diffusivity in function of

Table 3  
Effective moisture diffusivity values for each test.

	Sample thickness (mm)															
	5				10				15				20			
Temperature (°C)	100	150	200	250	100	150	200	250	100	150	200	250	100	150	200	250
$D_{eff} \times 10^9$ ( $m^2/s$ )	1.29	3.66	4.71	5.94	1.96	5.33	9.17	12.9	3.12	8.62	13.6	19.4	3.89	11.6	19.4	28.8

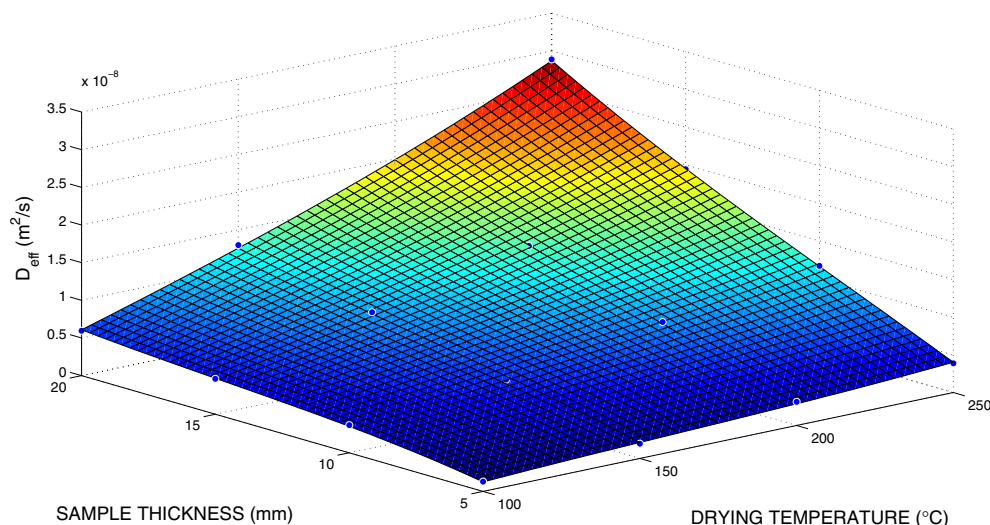


Fig. 11. Effective moisture diffusivity surface as a function of temperature and sample thickness.

drying air temperature and sample thickness. These values increase when the drying air temperatures and the sample thicknesses increase as well.

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

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

where  $D_0$  is the pre-exponential factor ( $\text{m}^2/\text{s}$ ),  $R$  is the universal gas constant ( $\text{J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$ ),  $T$  is the absolute temperature (K) and  $E_a$  is the activation energy ( $\text{J} \cdot \text{mol}^{-1}$ ). The activation energy was found out for each sample thickness. The function  $\ln D_{eff}$  versus  $1/T$  was obtained for each sample thickness. Table 4 indicates the values obtained of the activation energy after analyzing the slope of the linear approximation function. The coefficients of determination values are shown as well. As can be seen, the activation energy values grow as the sample thickness increases.

There are no results in the literature about the effective moisture diffusivity and the activation energy of the spent coffee grounds. For coffee bean, the mean effective moisture diffusivity was calculated in  $7.173 \cdot 10^{-10} \text{ m}^2/\text{s}$  [16]. Similar values of effective moisture diffusivity and activation energy can be found in other agricultural products. For olive-waste cake, these values were between  $1.71 \cdot 10^{-9}$ – $2.03 \cdot 10^{-9} \text{ m}^2/\text{s}$ , with temperatures between 50 and 90 °C, and a value of activation energy of 12430 J/mol for a sample thickness of 13 mm [31]. For corn, the values were found between  $0.948 \cdot 10^{-10}$ – $1.768 \cdot 10^{-10} \text{ m}^2/\text{s}$ , with temperatures in the range of 55–70 °C, and a value of activation energy of 29560 J/mol for a sample thickness of 3.4 mm [32]. And for vegetable wastes, the values calculated were between  $6.03 \cdot 10^{-9}$  and  $31.5 \cdot 10^{-9} \text{ m}^2/\text{s}$ , in a range of temperature of 50–150 °C, and an activation energy of 19820 J/mol for a sample thickness of 15 mm [33].

Table 4

Activation energy values for different sample thicknesses.

	Sample thickness (mm)			
	5	10	15	20
Activation energy (J/mol)	12288	12778	15181	16869
R <sup>2</sup>	0.9928	0.9903	0.9958	0.9919

#### 4. Conclusion

The drying process is fundamental for the biofuel production from spent coffee grounds. Their drying kinetics was studied in this work. Drying curves were fitted with the mathematical models proposed by the researchers to date. The Two Term Gaussian model achieved excellent results in all experiments. The drying rate was calculated and its stages were analyzed. Values of effective moisture diffusivity and activation energy were found between  $1.29 \cdot 10^{-9}$  and  $28.8 \cdot 10^{-9} \text{ m}^2/\text{s}$  and 12.29 and 16.87 kJ/mol, respectively.

Results can serve as starting point in other drying systems like: fluidized bed dryers, solar dryers and rotary dryers. Spent coffee grounds from services sector can present different characteristics such as initial moisture content and particle size with respect to those generated in instant coffee industry. For future studies, additional researches with spent coffee grounds from instant coffee industry should be made, and then, compare them with this work.

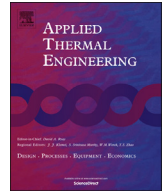
#### Acknowledgments

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## Modeling of olive-oil mill waste rotary dryers: Green energy recovery systems



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### HIGHLIGHTS

- Olive-oil mill waste rotary dryers play a crucial role in the olive oil industry.
- This review analyzes the main mathematical model in the trommel and furnace.
- Knowing the drying kinetics of olive-oil mill wastes is vital to complete the trommel models.
- Improving the design implies the automation, optimization and control in these dryers.
- The use of Computational Fluid Dynamics techniques can open new research.

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### ABSTRACT

Olive-oil mill waste rotary dryers play an important role in the environmental protection and sustainable development in the olive sector. Drying of these wastes is vital for three main reasons: the extraction of olive oil contained in them (olive pomace oil) by the use of solvents, the obtaining of a biomass fuel called dry de-oiled pomace and the elimination of a highly polluting by-product due to its high biological oxygen demand (BOD). However, because of the transition from three-phase system to two-phase system, their drying presents serious problems due to high moisture contents and their nature and composition. The main objective of this work is to study and analyze the research carried out on drying kinetics of olive-oil mill wastes, which is vital to understanding the drying phenomenon, and on the main models of heat and mass transfer in the olive-oil mill waste rotary dryers, based mainly on the trommel and furnace. The synergy between these two general studies will serve as a starting point for improving the design and optimization of the drying process in this type of dryers.

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### 1. Introduction

Until the nineties, the extraction process of olive oil was carried out in three phases: virgin olive oil, olive pomace and olive mill wastewater (OWW) [1,2]. The olive pomace was a by-product formed by a small quantity of pieces of pit, pulp of olive and a certain amount of olive oil. Olive mill wastewater, formed by vegetation water and organic compounds, constituted a hazardous contaminant due to its high biological oxygen demand (BOD) and chemical oxygen demand (COD) [3].

The current process of olive oil extraction, “two-phase system”, separates the virgin olive oil from two-phase olive mill waste. Two-phase system was instituted with the objective of eliminating the environmental problems caused by the three-phase system, and to improve the use of water resources more efficiently [4–6]. Two-phase olive mill waste is a thick sludge that contains pieces of pit, pulp, skin, vegetation water and about 5% olive oil content. In order to extract the oil contained in two-phase olive mill waste, it is necessary to dry the product to values close to 8% moisture content. Subsequently, it is mixed with a solvent, usually hexane, which facilitates its extraction. Initially, the extracted oil is refined, and then combined with virgin oils. The production of olive pomace oil represents 10% of all olive oils. The price of olive pomace oil is slightly lower than virgin olive oil. Nowadays, olive pomace oil is

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**Nomenclature***Latin letters*

$a, b, c, d, e, f, n$	coefficients of the mathematical models
$A$	empirical constant correlation Saeman and Mitchell
$B$	fuel wet fraction
$c_p$	specific heat capacity at constant pressure ( $\text{J kg}^{-1} \text{K}^{-1}$ )
$D$	trommel diameter (m)
$d_p$	particle size (m)
$D_{eff}$	effective moisture diffusivity ( $\text{m}^2 \text{s}^{-1}$ )
$D_0$	pre-exponential factor of the Arrhenius equation ( $\text{m}^2 \text{s}^{-1}$ )
$E_a$	activation energy ( $\text{J mol}^{-1}$ )
$F$	fuel fraction
$G$	mass flow, per unit area ( $\text{kg s}^{-1} \text{m}^{-2}$ )
$H$	product moisture content, kg moisture/kg wet matter
$H_i$	kg Hydrogen/kg fuel
$k, k_0, k_1, k_2, k_3$	constants of the mathematical models ( $\text{s}^{-1}$ )
$K'$	empirical constant Kelly correlation
$K$	empirical constant for calculation volumetric heat transfer coefficient
$L$	trommel length (m)
$L_c$	thickness of the slab (m)
$LMTD$	log mean temperature difference
$m$	mass (kg)
$\dot{m}$	mass flow ( $\text{kg s}^{-1}$ )
$N$	trommel speed (rpm)
$NCV$	net calorific value ( $\text{J kg}^{-1}$ )
$P$	pressure ( $\text{J m}^{-3}$ )
$Q_G$	heat gas combustion (J)
$Q_H$	heat which raises the temperature in the gas (J)
$Q_L$	heat which raises liquid phase temperature in the product (J)
$Q_p$	heat which raises the product temperature (J)
$Q_v$	heat which evaporates a liquid portion in the product (J)
$R$	universal gas constant ( $\text{J kg}^{-1} \text{K}^{-1}$ )

$S$	trommel slope
$t$	time (s)
$T$	temperature (K)
$U$	volumetric heat transfer coefficient ( $\text{W m}^{-3} \text{K}^{-1}$ )
$v$	velocity ( $\text{m s}^{-1}$ )
$V$	volume ( $\text{m}^3$ )
$X$	product moisture content, kg moisture/kg dry matter
$XR$	dimensionless product moisture ratio
$x_v$	drying rate ( $\text{s}^{-1}$ )
$Y$	air moisture content, kg moisture/kg dry matter
$z$	amount of ash

*Greek letters*

$\eta$	combustion yield
$\lambda$	latent heat of vaporization of water ( $\text{J kg}^{-1}$ )
$\nu$	empirical constant for calculation volumetric heat transfer coefficient
$\rho$	density ( $\text{kg m}^{-3}$ )
$\bar{\tau}$	mean residence time (s)

*Subscripts*

$a$	air
$da$	dry air
$dp$	dry product
$e$	equilibrium
$ev$	evaporated
$f$	fuel
$g$	gas
$in$	inlet
$out$	outlet
$p$	product ( <i>olive-oil mill waste</i> )
$t$	in each instant of time
$v$	vapor
$va$	vapor in the air
$vg$	vapor in the gas
$w$	water
$0$	initial

being considered in the production of biodiesel [7–9]. After performing these processes, a new biomass product with a net calorific value of  $17.5 \text{ MJ kg}^{-1}$  is obtained. Dry de-oiled pomace is a green energy source to be considered in Mediterranean countries where olive oil production is important. This biofuel has special interest as fuel in boilers for residential heating [10], fuel consumption in drying furnaces, and fuel for cogeneration [11] and generation [12] plants (Table 1). 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, and the energy released can be regarded as clean. Furthermore, both dry de-oiled pomace and olive stone can be transformed via gasification processes for using in internal combustion engines and microturbines [13–16]. Table 2 indicates the annual consumption per type of biomass in Andalusia (Spain) and its most important characteristics [17].

Olive-oil mill waste rotary dryers were designed to dry olive pomace (“three-phase system”), which had a moisture content of about 40%. With the current “two-phase system” the product to dry contains a moisture content of 60%. Subsequently, olive stones separation in the olive mill [18], about 7% of two-phase olive mill waste, further increases the moisture content (net calorific value of olive stone is slightly higher than dry de-oiled pomace,  $18,800 \text{ kJ kg}^{-1}$ , but its production is three times lower). This entails

a high energy cost due to the elimination of high water contents. Another significant drawback is the contribution of sugars which promote agglomerations during the drying process (increasing the particle size to dry). These agglomerations increase the product adhesion to the trommel walls. Therefore, the new extraction process (two-phase system) represents an additional difficulty in the drying process of olive-oil mill wastes. To solve the problem, the researchers focus on the automation of rotary dryers: furnace and trommel [19–25], and on the control of the most important parameters during the drying process. Such parameters are controlled in the trommel and are: the inlet drying air temperature, the outlet drying air temperature, the drying air velocity, olive-oil mill waste temperatures and the outlet olive-oil mill waste moisture content.

The performance improvement of the facilities is, currently, one of the major objectives, being necessary to achieve an optimization between energy cost, used in the olive-oil mill waste drying process, and the products obtained: olive pomace oil and dry de-oiled pomace (biomass). As discussed above, the main problem is the high moisture content of this waste. This work aims to expose and analyze the main contributions on the drying kinetics and the heat and mass transfer models. The analysis carried out on drying kinetics will help to improve and complete the mathematical models in rotary dryers by means of the obtaining of drying times and

**Table 1**  
Electrical energy generation plants with biomass in Andalusia (Spain) [17].

Biomass plant	Power (MW)	Biomass type	Locality	Province
Albaida Natural Resources	1.70	Green house residues	Níjar	Almería
Baena Agroenergy	25.00	Two-phase olive mill waste	Baena	Córdoba
Egabrense Bioenergy	8.00	Dry de-oiled pomace	Cabra	Córdoba
Severaes Biomass Energy	0.10	Olive tree pruning	Cañete de las Torres	Córdoba
Santamaría Bioenergy	14.30	Dry de-oiled pomace	Lucena	Córdoba
Santa María Muñoz's brothers and sons	1.72	Dry de-oiled pomace	Lucena	Córdoba
Palenciana Agroenergy	5.37	Two-phase olive mill waste	Palenciana	Córdoba
El Tejar Auto-generation	5.65	Two-phase olive mill waste	Palenciana	Córdoba
Vetejar Biomass	12.90	Two-phase olive mill waste	Palenciana	Córdoba
Puente Genil Biomass	9.82	Dry de-oiled pomace	Puente Genil	Córdoba
Ence Energy and Cellulose	67.95	Wood	San Juan del Puerto	Huelva
Tradema Biomass Energy	2.00	Wood	Linares	Jaén
Linares Bioenergy	15.00	Dry de-oiled pomace, agricultural and forestry residues, energy crops	Linares	Jaén
La Loma Energy	16.00	Dry de-oiled pomace	Villanueva del Arzobispo	Jaén
Aldebarán, Energy of the Guadalquivir River	6.00	Olive tree pruning, chip, cereal straw	Andújar	Jaén
Fuente de Piedra Biomass	8.04	Dry de-oiled pomace	Fuente de Piedra	Málaga
Extragol Bioenergy	9.15	Dry de-oiled pomace, pruning residues, energy crops	Villanueva de Algaidas	Málaga
Total	208.7			

drying rates. Particular emphasis has been placed on the control of the temperatures and moisture contents in which the release of volatiles occurs. These temperatures are vital to control the quality of olive pomace oil and dry de-oiled pomace. All these considerations indicated in this review should be taken into account in future works.

The rest of the paper is arranged as follows. Section 2, the drying kinetics of olive-oil mill wastes, both olive pomace (three-phase system) and two-phase olive mill waste, and experimental contributions of various authors are discussed. Section 3 analyzes the different study models of the trommel in rotary dryers, applying the differential equations to the whole trommel or discretizing it in sections. Section 4 studies the control models in the furnace, which are fed by dry de-oiled pomace. Finally, in Section 5, relevant findings are spelled.

## 2. Drying kinetics of olive-oil mill wastes

The drying process of olive-oil mill wastes is usually a complex phenomenon where convection and diffusion phenomena intervene. This is due to the heterogeneity of the product such as particles formed of pits and pulp of different size, sample sizes, vegetation water, sugars and to the different conditions of drying air temperature and velocity. During its tour in the trommel, the energy exchange (heat transferred to olive-oil mill wastes from hot gases) and the mass exchange (the evaporated water from olive-oil mill wastes) occur. Thus, these wastes are heated and dried, and the drying air is moistened and cooled.

The moisture content may stay inside of olive-oil mill wastes in three different ways [26]:

**Table 2**  
Biomass features and annual consumption per type of biomass in Andalusia (Spain), 2013 [17].

Biomass	Energy (ktoe)	NCV (kJ kg <sup>-1</sup> dry basis)	Moisture content (% wet basis)
Dry de-oiled pomace	216.40	17,500	(8–12)
Olive stone	173.83	18,800	(10–15)
Forest wood	54.35	18,000	(40–60)
Pellets, wood chips	16.22	17,500	<12
Biogas	6	(14,200–30,000)	–

- On the surface (non-mixed with the solid), being the easiest part to evaporate.
- In the porous structure, the water needs to be carried to the surface to be evaporated.
- By capillarity, closely linked to the solid. It is the way that requires more effort to extract the moisture.

Different researchers have analyzed the drying kinetics of olive pomace (three-phase system) and two-phase olive mill waste in other drying systems like fluidized beds [27,28], convective dryers [29–37], microwave-convective dryers [38,39], infrared dryers [40] and solar dryers [41–43]. The experimental studies of the drying kinetics of olive-oil mill wastes are vital to understanding and improve the large-scale drying processes in rotary dryers. Drying kinetics allows to obtain the drying conditions, drying air temperature and moisture content, in which the release of volatiles and the combustion appear [44]. Furthermore, these phenomena are closely related to the emergence of polycyclic aromatic hydrocarbons (PAHs), mainly benzopyrene, in the olive pomace oil [45,46]. At present, in the European Union the maximum tolerable limit for benzopyrene in the group of oils and fats intended for direct human consumption or use as an ingredient in food has been set at the level of 2 µg kg<sup>-1</sup> by the Commission Regulation (EU) No. 835/2011 of August 19, 2011, from September 1, 2012 [47]. Arjona et al. demonstrated that the release of volatiles and the start of combustion occurred at temperatures above 200 °C and 250 °C, respectively, when the moisture content reached to equilibrium moisture content [37]. Similar conclusions were found in the drying of olive stone [48] and spent coffee grounds [49]. On the other hand, drying kinetics is able to identify the heat and mass transfer mechanisms, the drying times and the drying rates for each instant of time. With respect to olive-oil mill wastes, the vast majority of the research agreed that their drying occurs mainly by diffusion phenomenon in the falling rate period. In these studies, drying curves obtained, XR (dimensionless moisture ratio) versus *t* (time), are approximated with the main mathematical models in the drying of agricultural products (Table 3).

Dimensionless moisture ratio can be expressed as:

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

**Table 3**  
Semi-empirical and empirical mathematical models in the drying of olive-oil mill wastes.

Model name	Model type	Equation	Reference
Lewis	Empirical	$XR = \exp(-kt)$	[50]
Page	Empirical	$XR = \exp(-kt^n)$	[51]
Modified Page	Empirical	$XR = \exp(-(kt)^n)$	[52]
Henderson and Pabis	Empirical	$XR = a \cdot \exp(-kt)$	[53]
Logarithmic	Empirical	$XR = a \cdot \exp(-kt) + c$	[32]
Wang and Singh	Empirical	$XR = 1 + at + bt^2$	[54]
Two term	Semi-empirical	$XR = a \cdot \exp(-k_0t) + c \cdot \exp(-k_1t)$	[55]
Two term exponential	Semi-empirical	$XR = a \cdot \exp(-kt) + (1 - a) \cdot \exp(-kat)$	[56]
Approach of diffusion	Semi-empirical	$XR = a \cdot \exp(-kt) + (1 - a) \cdot \exp(-kbt)$	[57]
M. Henderson and Pabis	Semi-empirical	$XR = a \cdot \exp(-kt) + b \cdot \exp(-k_2t) + c \cdot \exp(-k_3t)$	[34]
Midilli et al.	Empirical	$XR = a \cdot \exp(-kt^n) + bt$	[58]
Yun et al.	Empirical	$XR = \frac{a+bt+ct^2}{1+dt+et^2}$	[59]
Two term Gaussian	Empirical	$XR = a \cdot \exp\left[-\left(\frac{t-b}{c}\right)^2\right] + d \cdot \exp\left[-\left(\frac{t-e}{f}\right)^2\right]$	[48]

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. Drying rate is calculated experimentally from the derivate of drying curves as:

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

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.

However, other research [35–37] showed that the convection phenomenon is important at the beginning of the drying process of olive-oil mill wastes. In fact, the analysis of the drying rate in these studies showed several stages during the drying. These stages were: warming-up period, first falling rate period, second falling rate period and volatiles period (only at temperatures > 200 °C). There was no constant rate period.

- In the first stage (warming-up period), a rapid heating is produced, being the capillary suction insufficient to carry the moisture to the surface. Drying rate increases quickly (higher vapor pressures) to a maximum value in this phase. This period is characteristic because the moisture of the surface is practically eliminated.
- Second stage (first falling rate period) appears at low drying temperatures where a *funicular* state exists. The surface is not saturated, but a continuous flow in contact with the gas exists. The drying rate begins to fall. At high temperatures, this stage overlaps the first.
- In the third stage (second falling rate period) the internal moisture is transported to the surface by diffusion when the temperature is low. Inside the mass, moisture and gas bubbles coexist. This stage is known as the *pendular* state [60]. At high temperatures, the liquid boils at 100 °C (atmospheric pressure), vapor rises to the surface with little resistance, leaving behind the hygroscopic range.
- The fourth phase (volatiles period) appears only at temperatures above 200 °C. This causes a loss of volatile material once the mass has been dried.

As mentioned above, only in the first stage appeared an increase in the drying rate. In all other stages decreased. To date, the mathematical model in the drying of olive-oil mill wastes that has the best results of fit is an empirical model: the Two Term Gaussian (Table 3). Furthermore, the derivate of this model faithfully reflects the different stages in the drying of olive-oil mill

wastes [36]. Both the drying time and the drying rate depend primarily on variables such as the drying temperature, the moisture content, the drying air velocity, the particles size and the sample thickness.

### 2.1. Drying temperature dependence and moisture content dependence

Drying air temperature is the most decisive factor in the drying process, regardless of the particle size [29]. The drying rate increases linearly regardless of moisture content, being even more sensitive to high moisture values. This is because the resistance of heat transfer is higher at low moisture contents. On the other hand, the drying time also decreases with the temperature. In fact, the higher the temperature is, the higher the drying rate is [37].

Another important factor that adequately explains the mass transfer process by diffusion (almost all drying process) during the drying of olive-oil mill wastes is the effective moisture diffusivity. This variable is associated with the diffusion of mass into the medium during changes of moisture content with the time. It can be experimentally calculated from Fick's second law of diffusion (Eq. (3)). The solution of this partial differential equation (Eq. (4)), was established by Crank [61] for the one-dimensional mass transport in infinite slab geometry, in spherical coordinates. This solution has the following limitations: constant temperature and diffusion coefficients and negligible shrinkage.

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

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

where,  $D_{eff}$  is the effective moisture diffusivity ( $m^2 s^{-1}$ ),  $t$  is the drying time (s) and  $L_c$  is the thickness of slab (m). If the equation is approximated to the first term of the series and taking the natural logarithm of both sides it yields:

$$\ln XR = \ln \frac{8}{\pi^2} - \frac{\pi^2 D_{eff} t}{L_c^2} \quad (5)$$

This equation represents the linear dependence between  $\ln XR$  and drying time. In this case, effective moisture diffusivity can be calculated from the slope of the linear function which approximates the experimental data.

Beginning with the experimental data, the activation energy can be obtained. The temperature dependence of the effective moisture diffusivity may be represented by an Arrhenius relationship:

$$D_{eff} = D_0 \exp\left(-\frac{E_a}{RT}\right) \quad (6)$$

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

## 2.2. Drying air velocity dependence

Experiments conducted so far showed that the drying air velocity intervenes considerably in the drying rate and the drying time. In fact, the effect of velocity becomes more decisive in the first stage of the drying process (increasing the drying rate) for both high and low temperature [35]. However, for lower moisture contents, the effect of velocity becomes negligible (the water migration to the surface is insignificant). Freire et al. [29] showed the existence of a critical velocity. For values above the critical velocity, the drying rate becomes negligible and the drying is controlled by internal mass transfer mechanism (migration of moisture through the internal structure of olive-oil mill wastes). On the other hand, for values below the critical velocity, the external resistance to moisture transfer could not be neglected. This indicates that the drying air temperature prevails on the drying air velocity.

## 2.3. Sample thickness dependence

Another way to analyze the drying rate and drying time is from the sample thickness. Research showed that the drying rate increases as the sample thickness decreases. The results were obtained through effective moisture diffusivity calculation for different thicknesses, in particular, 6 mm, 9 mm and 12 mm with constant drying air velocity of  $1.5 \text{ m s}^{-1}$  between  $60 \text{ }^\circ\text{C}$  and  $80 \text{ }^\circ\text{C}$  [33] and for the thicknesses 41 mm, 52 mm and 63 mm with constant drying air velocity of  $1 \text{ m s}^{-1}$  between  $50 \text{ }^\circ\text{C}$  and  $80 \text{ }^\circ\text{C}$  [28]. The natural logarithm of  $D_{eff}$  can be drawn as a function of the reciprocal of absolute temperature. The values of activation energy obtained by these researchers showed that they are larger when the sample thicknesses are greater. For the thicknesses 6 mm, 9 mm and 12 mm, the activation energies were  $25.4 \text{ kJ mol}^{-1}$ ,  $25.7 \text{ kJ mol}^{-1}$  and  $29.2 \text{ kJ mol}^{-1}$  respectively, and for the thicknesses 41 mm, 52 mm and 63 mm, the activation energies were  $34.05 \text{ kJ mol}^{-1}$ ,  $36.84 \text{ kJ mol}^{-1}$  and  $38.10 \text{ kJ mol}^{-1}$  respectively.

## 2.4. Particle size dependence

The influence of the particle size in the drying process was analyzed as well. The tests showed heterogeneity in particle size in a particular sample. The results [37] showed that the drying rate was decreased when the average particle size,  $d_p$ , was decreased. This is evident since the particle specific surface (external surface per unit sample volume) increases when  $d_p$  is smaller, and therefore, greater heat transfer exchange exists. However, there is a very important phenomenon to take into account, when the particle size ranges between values from 0 to approximately 2.5 mm [29]. It is noted that in this range the drying time increased when the particle size decreased. This may be because the composition of particles is quite heterogenous, formed by high moisture content and some oil content. The oil may have a barrier effect which prevents water leaks in the olive-oil mill wastes. The research conducted by Gögüs and Maskan [33] showed that the particles of a size greater than 2 mm had a porous structure that allowed the water leak. The opposite happened when the particle sizes were smaller, less than 1 mm, which were agglomerated building a non-porous structure that hinders the transport of moisture to the surface.

Lastly, Table 4 indicates a summary of the most important variables in the drying kinetics of olive-oil mill wastes from the different drying conditions in several types of dryers. The results obtained by all research indicate similar results in the values of effective moisture diffusivity and activation energy. Effective moisture diffusivity values depend mainly on the drying air temperature and the sample thickness. In fact, when the drying air temperature and the sample thickness increased, the values of this variable increased as well. Activation energy values depend on the sample thickness. The higher the sample thickness is, the higher the activation energy is.

## 3. Study models for trommel in olive-oil mill waste rotary dryers

Models of heat and mass transfer in the trommel of a rotary dryer have been studied from overall models and sectioned models. The first ones are mainly based on variables such as resident time and volumetric heat transfer coefficient [26,62–69]. On the other hand, overall models are analyzed both under steady-state [70–72] and transient [73,74] conditions. The second ones study the trommel from discretized models dividing the trommel length in sections [65,75,76]. Fig. 1 shows the trommel of an industrial olive-oil mill rotary dryer.

**Table 4**  
Summary of the main contributions in the drying kinetics of olive-oil mill wastes carried out in different types of dryers.

Type of dryer	Moisture content (w.b.) initial–final	Air temperature ( $^\circ\text{C}$ )	Air velocity ( $\text{m s}^{-1}$ )	Sample thickness (mm)	Activation energy ( $\text{kJ mol}^{-1}$ )	Effective moisture diffusivity ( $\text{m}^2 \text{s}^{-1}$ )	Reference
Solar dryer	55–20%	20, 30, 40, 50	5	30	38.64	$9.13 \cdot 10^{-11}$ – $1.40 \cdot 10^{-9}$	[43]
Solar dryer	66–7.5%	20, 40, 80	1	6.2	15.77	$2.22 \cdot 10^{-10}$ – $6.99 \cdot 10^{-10}$	[41]
Fluidized bed dryer	48.6–4.5%	50, 60, 70, 80	1	41	34.05	$0.68 \cdot 10^{-7}$ – $1.93 \cdot 10^{-7}$	[28]
				52	36.84	$0.82 \cdot 10^{-7}$ – $1.83 \cdot 10^{-7}$	
				63	38.10	$0.94 \cdot 10^{-7}$ – $2.15 \cdot 10^{-7}$	
				3	Not mentioned	$1.6 \cdot 10^{-9}$ – $3.1 \cdot 10^{-9}$	[29]
Convective dryer	50–1%	125, 150, 200	1.5	3	Not mentioned	$1.6 \cdot 10^{-9}$ – $3.1 \cdot 10^{-9}$	[29]
Convective dryer	45–5%	80, 90, 100, 110	1.2	12	26.71	$4.89 \cdot 10^{-8}$ – $9.99 \cdot 10^{-8}$	[31]
Convective dryer	45–5%	50, 60, 70, 80, 90, 100, 110	1.2	8	17.97	$0.3 \cdot 10^{-8}$ – $1.1 \cdot 10^{-8}$	[32]
Convective dryer	36.7–0%	60, 70, 80	1.5	6	24.4	$1.84 \cdot 10^{-7}$ – $3.42 \cdot 10^{-7}$	[33]
				9	25.7	$2.17 \cdot 10^{-7}$ – $3.67 \cdot 10^{-7}$	
				12	29.2	$2.18 \cdot 10^{-7}$ – $3.94 \cdot 10^{-7}$	
				13	12.43	$1.71 \cdot 10^{-9}$ – $2.03 \cdot 10^{-9}$	[34]
Convective dryer	65.6–27%	50, 60, 70, 80, 90	2	13	12.43	$1.71 \cdot 10^{-9}$ – $2.03 \cdot 10^{-9}$	[34]
Microwave-convective dryers	67.65–0%	40, 50, 60, 70	4	7	22.6	$8.95 \cdot 10^{-10}$ – $1.95 \cdot 10^{-9}$	[39]
				7	22.6	$8.95 \cdot 10^{-10}$ – $1.95 \cdot 10^{-9}$	
Infrared dryer	48–8%	80, 100, 120, 140	–	7	21.3	$5.958 \cdot 10^{-9}$ – $15.88 \cdot 10^{-9}$	[40]



Fig. 1. Trommel in an industrial olive-oil mill waste rotary dryer.

Even today, the design and modeling of an olive-oil mill waste rotary dryer is a very difficult task to perform. Although there are not many researchers in the field [21–23,25,30], they all performed a study on the trommel for co-current rotary dryer configuration. These models are based on differential equations which show the principles of the mass and energy conservation. The problem is to obtain a mathematical model that simulates the temperature conditions, drying air velocity and moisture content of olive-oil mill wastes within the trommel similar to Fig. 2. To do this, the researchers proposed the study of the trommel applying differential equations to the whole, or dividing it into finite sections. The controlled variable is the outlet drying air temperature, since the moisture measurement in situ is very complicated.

Fig. 3 exhibits the mass and heat transfer representation occurring into the trommel. The heat transfer is mainly carried out by the conduction-convection phenomenon. Heat losses are negligible with respect to others.

The heat in the combustion gases is ( $Q_G$ ) converted into:

- Raising the temperature of the product solid phase ( $Q_p$ ).
- Raising the temperature of the product liquid phase ( $Q_L$ ).
- Evaporating a part of the liquid contained in the product ( $Q_V$ ).
- Raising the temperature of the vapor in the gas ( $Q_H$ ).

3.1. Overall trommel model

3.1.1. Residence time and volumetric heat transfer coefficient

The volumetric heat transfer coefficient and the residence time are used as sub-models to describe the internal space of the trommel occupied by gas. These relations are mostly empirical by

nature. The residence time is defined as the mass retained (kg) divided by the solid matter flow ( $\text{kg s}^{-1}$ ). This parameter can be measured knowing the previous data. The mass could be calculated removing it from the dryer and weighing it. The mean residence time can be calculated as:

$$\bar{\tau} = \frac{m_p}{\dot{m}_p} \tag{7}$$

However, for this type of system, mathematical functions are used [26]. The equation commonly used in rotary dryers was proposed by Friedman and Marshall [62,63]:

$$\bar{\tau} = \frac{0.23L}{S \cdot N^{0.9} \cdot D} - 0.6 \frac{5d_p^{-0.5} \cdot L \cdot G_a}{G_p} \tag{8}$$

where  $L$  is the trommel length (m),  $D$  is the interior diameter (m),  $N$  is the cylinder rotational speed (rpm),  $S$  is the trommel slope ( $\text{m m}^{-1}$ ),  $G_a$  and  $G_p$  ( $\text{kg s}^{-1} \text{m}^{-2}$ ) are the mass velocities of gas and dry solid matter, respectively, and  $d_p$  ( $\mu\text{m}$ ) is the average weight of the particle size.

Other investigations use the Kelly correlation [66]:

$$\bar{\tau} = \frac{K' \cdot L}{S \cdot N \cdot D} \tag{9}$$

where  $K'$  is an empirical constant. Another correlation is provided by Saeman and Mitchell [68]:

$$\bar{\tau} = \frac{L}{A \cdot S \cdot N \cdot D} \tag{10}$$

where  $A$  is a constant which depends on the trommel design.

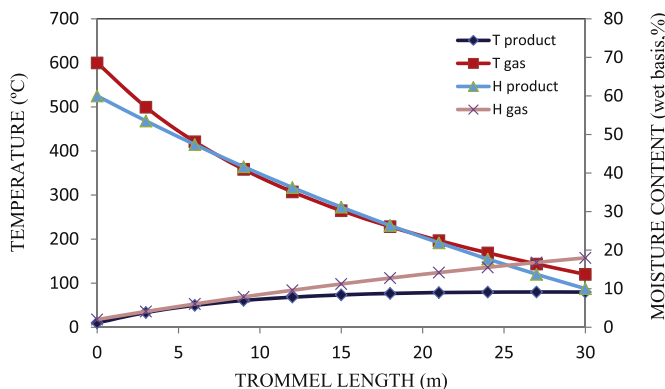


Fig. 2. Moisture and temperature evolution in olive-oil mill waste rotary dryer.

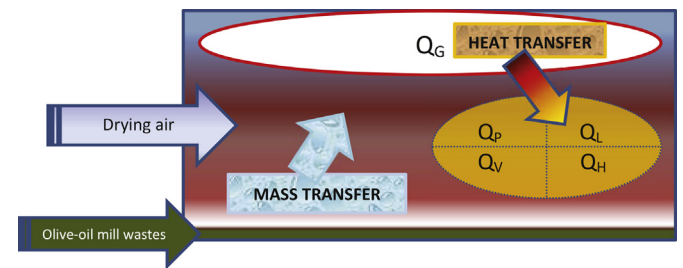


Fig. 3. Mass and heat transfer in the trommel.

The research realized by Casanova-Peláez [23] in an industrial trommel of olive-oil mill wastes presented an experimental residence time of 21.5 min and a design residence time of 20 min.

Volumetric heat transfer coefficient is a parameter to consider in the evolution of variables into the trommel. This parameter is defined as the rate of heat transfer transmitted to one unit volume of the trommel when the difference of temperature is the unit. All scholars [62,63,67,69] reached similar conclusions: volumetric heat transfer coefficient,  $U$ , depends on solid flow, dry air flow and solid physics features:

$$U = \frac{K \cdot G^v}{D} \quad (11)$$

where  $G$  is the dry gas flow ( $\text{kg h}^{-1} \text{m}^{-2}$ ),  $D$  is the trommel diameter (m) and  $K$  and  $v$  are empirical constants which depend on the material to dry. For olive-oil mill wastes the researchers obtained a volumetric heat transfer coefficient ranging from 0.11 to  $0.17 \text{ kW m}^{-3} \text{ K}^{-1}$  [23]. Castaño [21] obtained  $v$  and  $K$  empirical values of 0.5595 and 336 respectively, with  $U \approx 0.3 \text{ kW m}^{-3} \text{ K}^{-1}$ . For the steady-state study,  $U$  produces satisfactory results. Nevertheless, it does not reflect the dynamic behavior when a parameter varies.

### 3.1.2. Steady-state model

For the steady-state, conservation equations are applied to the gaseous and solid phase mass flows, having:

$$\dot{m}_{p,in} = \dot{m}_{p,out} + \dot{m}_{w,ev} \quad (12)$$

$$\dot{m}_{a,in} = \dot{m}_{a,out} - \dot{m}_{w,ev} \quad (13)$$

For the energy balances, the heat transferred can be quantified in a basic mode following a series of considerations. It is assumed that the inlet drying air is completely dry (in reality, it has a very low moisture content) and the air, water and olive-oil mill waste specific heat capacity are constant, as well as the latent heat of vaporization of water. Thus, the value of each one of them can be calculated:

- Heat transported by the drying gas (air) which corresponds to a decrease in the temperature:

$$\frac{\partial Q_G}{\partial t} = \dot{m}_{da} \cdot c_{p,da} \cdot (T_{a,in} - T_{a,out}) \quad (14)$$

- Heat received by the solid (dry product): this heat is invested in raising its temperature:

$$\frac{\partial Q_p}{\partial t} = \dot{m}_{dp} \cdot c_{p,dp} \cdot (T_{p,out} - T_{p,in}) \quad (15)$$

- Heat received by the liquid (water): this heat is invested in raising its temperature before vaporizing:

$$\frac{\partial Q_L}{\partial t} = \dot{m}_{dp} \cdot X \cdot c_{p,w} \cdot (T_{p,out} - T_{p,in}) \quad (16)$$

- Heat for evaporation: heat required to change the liquid water state which is contained in the solid to vapor:

$$\frac{\partial Q_V}{\partial t} = \dot{m}_{w,ev} \cdot \lambda \quad (17)$$

- Heat for reheating: this heat is invested in raising the vapor temperature:

$$\frac{\partial Q_H}{\partial t} = \dot{m}_{w,ev} \cdot c_{p,v} \cdot (T_{a,in} - T_{a,out}) \quad (18)$$

In these equations  $\dot{m}$  ( $\text{kg s}^{-1}$ ) is the mass flow,  $c_p$  ( $\text{J kg}^{-1} \text{K}^{-1}$ ) represents the specific heat capacity at constant pressure,  $T_a$  and  $T_p$  (K) are the temperature of the air and olive-oil mill wastes respectively,  $X$  is the olive-oil mill waste moisture content (dry basis) and  $\lambda$  ( $\text{J kg}^{-1}$ ) is the latent heat of vaporization of water.

If the heat transmission losses to the outside are neglected, the overall energy balance is:

$$\frac{\partial Q_G}{\partial t} = \frac{\partial Q_p}{\partial t} + \frac{\partial Q_L}{\partial t} + \frac{\partial Q_V}{\partial t} + \frac{\partial Q_H}{\partial t} \quad (19)$$

On the other hand, if the trommel is considered a heat exchanger, the amount of heat transferred per unit time can be expressed as:

$$\frac{\partial Q_G}{\partial t} = U \cdot V \cdot LMTD \quad (20)$$

where  $U$  is the volumetric heat transfer coefficient ( $\text{W m}^{-3} \text{K}^{-1}$ ), that depends on the drying gas flow per unit area transverse,  $G$  ( $\text{kg s}^{-1} \text{m}^{-2}$ ), and the trommel diameter,  $D$  (m);  $V$  is the volume of the trommel and  $LMTD$  (K) is the log mean temperature difference between the dry air temperature and dry product temperature. If these temperatures are unknown, the log mean temperature difference between drying air wet bulb depressions at the inlet and outlet of the trommel can be used.  $U$  must be determined experimentally.

### 3.1.3. Transient model

This model consists in applying the conservation equations of mass and energy to the trommel, for each instant of time. The balances of olive-oil mill waste mass and humid air are related by the evaporated water flow. Regarding mass balances:

In the dry product, the variation of dry mass retained in the trommel is the difference between the inlet and outlet of the dry product flows:

$$\frac{dm_{dp}}{dt} = \dot{m}_{dp,in} - \dot{m}_{dp,out} \quad (21)$$

In the moisture content of the product, the variation of moisture content inside of solid is:

$$\frac{d(m_{dp}X)}{dt} = \dot{m}_{dp,in}X_{in} - \dot{m}_{dp,out}X_{out} - x_v m_{dp} \quad (22)$$

The first and second term on the right side represent the difference of inlet and outlet water flow (contained in olive-oil mill wastes). The third term indicates the water flow which is evaporated in that instant of time.  $X$  and  $x_v$  indicate the olive-oil mill waste moisture content (dry basis) and drying rate, respectively.

In the moisture content of the dry air, this moisture variation with time is equal to:

$$\frac{d(m_{da}Y)}{dt} = \dot{m}_{da,in}Y_{in} - \dot{m}_{da,out}Y_{out} + x_v m_{dp} \quad (23)$$

The first and second term on the right side represent the difference of inlet and outlet water flow (contained into dry air) and the third term indicates the water flow which is evaporated in that instant of time.  $Y$  and  $x_v$  indicate the air moisture content (dry basis) and drying rate respectively.

These equations can be simplified as:

$$\begin{aligned} \frac{dX}{dt} &= \frac{1}{m_{dp}} \left[ \dot{m}_{dp,in} X_{in} - \dot{m}_{dp,out} X_{out} - X \frac{dm_{dp}}{dt} \right] \\ \frac{dY}{dt} &= \frac{1}{m_{da}} \left[ \dot{m}_{da,in} Y_{in} - \dot{m}_{da,out} Y_{out} - Y \frac{dm_{da}}{dt} \right] \end{aligned} \quad (24)$$

The conservation equations of energy are expressed as:

In the product, the enthalpy variation of the wet product contained into the trommel with respect to time is equal to:

$$\begin{aligned} \frac{d(m_{dp} c_{p,p} T_p)}{dt} &= \dot{m}_{dp,in} c_{p,p} T_{p,in} - \dot{m}_{dp,out} c_{p,p} T_{p,out} \\ &+ U \cdot V (T_a - T_p) - x_v m_p \lambda - x_v m_p c_{p,v} (T_a - T_p) \end{aligned} \quad (25)$$

where the first and second term on the right side indicate the enthalpy difference of the inlet and outlet of the moisture product flows. The third term is the heat transferred from the air to the product. The fourth terminus represents the heat required to vaporize the moisture content of the product and the fifth terminus is the heat required for heating the vapor to the air temperature.

In the dry air, the enthalpy variation of the humid air contained into the trommel with respect to time is equal to:

$$\begin{aligned} \frac{d(m_{da} c_{p,a} T_a)}{dt} &= \dot{m}_{da} c_{p,a} T_{a,in} - \dot{m}_{da} c_{p,a} T_{a,out} - U \cdot V \cdot LMTD \\ &+ x_v m_{dp} \lambda + x_v m_{dp} c_{p,v} (T_a - T_p) \end{aligned} \quad (26)$$

where the first and second term on the right side indicate the enthalpy difference of the inlet and outlet humid air flows. The third term is the heat transferred from the air to the product. The fourth terminus represents the heat required to evaporate the water and the fifth terminus is the enthalpy of evaporated water of the product up to the air temperature.

### 3.2. Sectioned trommel model

Because of the evaporation process is not identical along the trommel length; the study can be carried out by dividing it into small sections. Thus, the evaporation process is identical along a section and distinct between different sections. It is necessary to model it based on other parameters such as temperatures and moistures. The outlet flows and temperatures in a section are the inlet flows and temperatures in the next section. In this model, the valid equations are given above, considering that the evaporated water content in a section will become part of the gaseous stream of the following section (increasing its moisture content). The rest of sections are solved consecutively.

To perform the mass and energy balances, it is necessary to know the drying rate and volumetric heat transfer coefficient. These values can be determined from experimental data and empirical expressions. It is necessary to know or estimate the residence time of olive-oil mill wastes in the dryer, for determining the material advance from one section to another. The trommel is divided by a finite number of volumetric elements. The equations of conservation are applied for each element. Each volume element is bounded longitudinally by two sections, the inlet ( $i - 1$ ) and the outlet ( $i$ ). Knowing the inlet conditions, the model can be solved consecutively. General considerations for this model are:

- Air and solid specific heat capacity are changing, since its moisture varies.
- The evaporated water in a section passes to the gaseous flow, intervening in the following sections. The same occurs with the heat which is associated with this process.
- The air velocity changes, since its temperature decreases and its moisture content increases.
- The drying rate varies along the process (it depends on temperature, moisture, granulometry, etc.)

Applying the principle of mass conservation to dry air:

$$\frac{\partial(m_{da})^{(i)}}{\partial t} = \dot{m}_{da}^{(i-1)} - \dot{m}_{da}^{(i)} \quad (27)$$

In the mass of dry product:

$$\frac{\partial(m_{dp})^{(i)}}{\partial t} = \dot{m}_{dp}^{(i-1)} - \dot{m}_{dp}^{(i)} \quad (28)$$

In the water contained in the product:

$$\frac{\partial(m_w)^{(i)}}{\partial t} = \dot{m}_{w,p}^{(i-1)} - \dot{m}_{w,p}^{(i)} - \dot{m}_{w,ev}^{(i)} \quad (29)$$

In the vapor contained in the air:

$$\frac{\partial(m_v)^{(i)}}{\partial t} = \dot{m}_v^{(i-1)} - \dot{m}_v^{(i)} + \dot{m}_{w,ev}^{(i)} \quad (30)$$

Applying the conservation equation of energy in the olive-oil mill wastes:

$$\begin{aligned} \frac{\partial(c_{p,dp}^{(i)} \cdot m_{dp}^{(i)} + c_{p,w}^{(i)} \cdot m_w^{(i)}) \cdot T_p^{(i)}}{\partial t} &= U^{(i)} \cdot V \cdot (T_a^{(i)} - T_p^{(i)}) + (c_{p,dp}^{(i)} \cdot \dot{m}_{dp}^{(i)} \\ &+ c_{p,w}^{(i)} \cdot \dot{m}_w^{(i)}) T_p^{(i)} - (c_{p,dp}^{(i-1)} \cdot \dot{m}_{dp}^{(i-1)} \\ &+ c_{p,w}^{(i-1)} \cdot \dot{m}_w^{(i-1)}) T_p^{(i-1)} \\ &- \dot{m}_{w,ev}^{(i)} \cdot (c_{p,w}^{(i)} \cdot T_p^{(i)} + \lambda) \end{aligned} \quad (31)$$

And the dry air:

$$\begin{aligned} \frac{\partial(c_{p,v}^{(i)} \cdot m_v^{(i)} + c_{p,da}^{(i)} \cdot m_{da}^{(i)}) T_p^{(i)}}{\partial t} &= -U^{(i)} \cdot V \cdot (T_a^{(i)} - T_p^{(i)}) + (c_{p,da}^{(i)} \cdot \dot{m}_{da}^{(i)} \\ &+ c_{p,v}^{(i)} \cdot \dot{m}_v^{(i)}) T_p^{(i)} - (c_{p,da}^{(i-1)} \cdot \dot{m}_{da}^{(i-1)} \\ &+ c_{p,v}^{(i-1)} \cdot \dot{m}_v^{(i-1)}) T_p^{(i-1)} \\ &- \dot{m}_{w,ev}^{(i)} \cdot c_{p,v}^{(i)} (T_p^{(i)} - T_a^{(i)}) \end{aligned} \quad (32)$$

In Eqs. (31) and (32), the first term of the right side is positive and negative respectively, because the hot air transfers heat at olive-oil mill wastes.

## 4. Study models for furnace in olive-oil mill waste rotary dryers

Another important process within the olive-oil mill waste drying is produced in the combustion chamber. The inlet hot gas temperature in the trommel depends on the control of the fuel combustion. Generally, fuel used is dry de-oiled pomace which is extracted from the drying plant. Its combustion is not carried out

regularly, unlike liquids and gases. Therefore, the temperatures reached present important oscillations. As it is known, dry de-oiled pomace has a heterogenous distribution in its composition, granulometry and moisture content. Furthermore, This biofuel is accumulated in grills when it enters into the furnace. This implies a complete combustion outside the mound and an incomplete combustion inside the mound. This drawback means that its control is more complicated due to the delay produced in the combustion.

The mathematical models set out so far are based on the application of the principles of mass and energy conservation to the furnace. Figs. 4 and 5 show the furnace in an industrial olive-oil mill rotary dryer and the diagram of the thermal processes in it. In its interior, the conditions are considered the same in any position.

Castañó et al. [19,25] took into account the characteristics of the fuel used, as well as the combustion yield. Applying the principles of mass and energy conservation, the equations are:

$$\frac{\partial m_g}{\partial t} = \dot{m}_{da} - \dot{m}_g + \dot{m}_f \cdot (1 - z) \cdot \eta \cdot (1 - B) \quad (33)$$

$$c_{Pg} \left( T_g \frac{\partial m_g}{\partial t} + m_g \frac{\partial T_g}{\partial t} \right) = \dot{m}_f \cdot NCV \cdot \eta + \dot{m}_{da} (c_{Pa} + Y_a c_{Pva}) T_a - \dot{m}_g (c_{Pg} + Y_g c_{Pvg}) \quad (34)$$

where in Eq. (33):  $\dot{m}_{da}$ ,  $\dot{m}_g$  and  $\dot{m}_f$  ( $\text{kg s}^{-1}$ ) are the mass flow of dry air, combustion gas and fuel respectively;  $\eta$  is the combustion yield,  $B$  is the fuel wet fraction and  $z$  is amount of ash. Masghouni and Hassairi [77] estimated the ash content about 6.6%. In Eq. (34)  $NCV$  ( $\text{J kg}^{-1}$ ) is the net calorific value of the fuel.

The gas mass in the furnace,  $m_g$ , and its dry fraction of the gas humidity,  $Y_g$ , can be calculated knowing that 2 kg of hydrogen originate 18 kg of water.

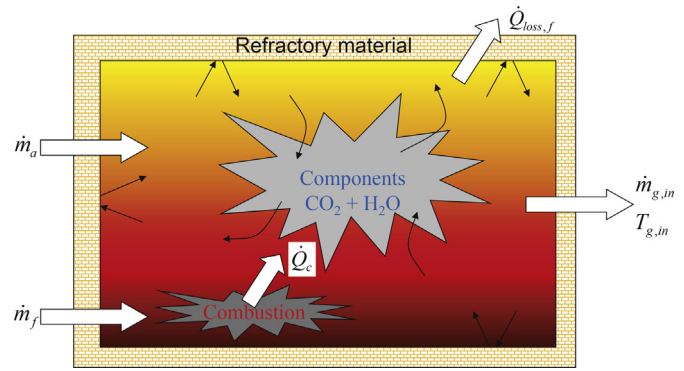


Fig. 5. Heat and mass transfer in the furnace.

$$m_g = V \cdot \frac{29 \cdot 18 \cdot (1 + Y_g)}{18 + 28 \cdot Y_g} \frac{P}{R \cdot T} \quad (35)$$

$$Y_g = \frac{Y_a \cdot \dot{m}_{da} + \dot{m}_f \cdot B + \dot{m}_g \cdot Hi \cdot \eta \cdot (1 - B) \cdot 18/2}{\dot{m}_g} \quad (36)$$

where  $P$  ( $\text{J m}^{-3}$ ),  $T$  (K) and  $V$  ( $\text{m}^3$ ) indicate the pressure, the temperature and the volume of the combustion chamber.  $R$  ( $\text{J kg}^{-1} \text{K}^{-1}$ ) is the universal gas constant and  $Hi$  is the mass fraction Hydrogen/Fuel.

The process control requires the inspection of different variables. The outlet temperature in the furnace is usually the controlled variable and the fuel mass is the control variable. In the trommel, the outlet gas temperatures can be controlled varying the inlet gas temperatures, simply by regulating the dry de-oiled pomace flow. Dry de-oiled pomace feeding is usually carried out by an Archimedes screw which can supply the biofuel by



Fig. 4. Furnace in an industrial olive-oil mill waste rotary dryer.

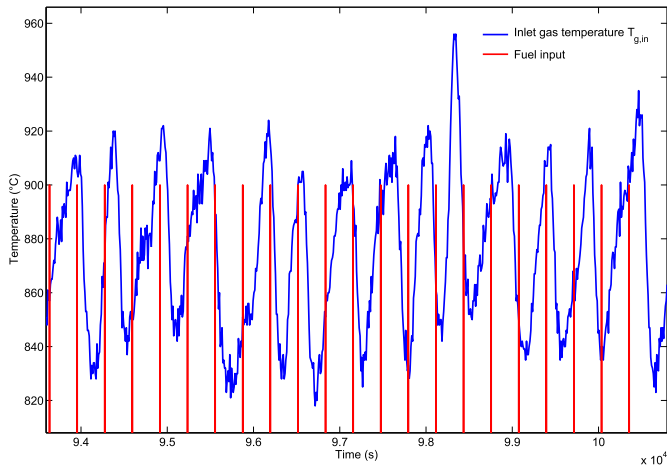


Fig. 6. Inlet drying gas temperature variation versus inlet fuel. Discontinuous feeding by PWM.

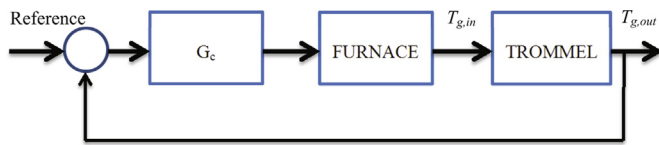


Fig. 7. Output gas temperature control by PID.

discontinuous feeding. Biofuel is introduced via package, starting off the Archimedes' screw motor periodically, following a “pulse-width modulation” (PWM) strategy. Fig. 6 depicts the variation of the inlet gas temperatures to the trommel and the inlet fuel in the furnace at steady-state [23,24]. In both cases, the control is usually performed by applying a PID [22,23] (Fig. 7).

Nevertheless, the dry de-oiled pomace furnace features involve a delay difficult to calculate, as well as large oscillations for outlet

temperature due to solid combustion. However, Casanova-Peláez et al. [24] performed an industrial furnace model based on *auto-regression method with exogenous variables (ARXs)*. This method allows to simulate a system quickly. The process defines the ARX experimental relation between the amounts of dry de-oiled pomace used as fuel and outlet gas temperatures in the trommel. They proposed that the furnace can be considered an open system where the inlet air and the biofuel are inlet variables and the hot gases and the heat losses through the walls are the outlet variables. Applying the conservation equations of mass and energy in a control volume,  $V$ , they propounded the following expression:

$$\frac{dQ}{dt}(1 - F) \approx V \frac{d(\rho_g T_g)}{dt} - \dot{m}_g c_{p,g} (T_g - T_a) \tag{37}$$

In this equation  $\dot{m}(\text{kg s}^{-1})$  is the mass flow,  $\rho$  ( $\text{kg m}^{-3}$ ) is the density,  $T$  (K) is the temperature and  $c_p$  ( $\text{J kg}^{-1} \text{K}^{-1}$ ) is the specific heat capacity at constant pressure. The  $F$  value represents the fuel fraction. The first term of the equation indicates a continuous heat loss that depends on this value. The model proposed above is not linear. Nevertheless, the process can be linearized near to the operating point simply by obtaining the values of the different constants and variables around this point. On the other hand, the proposed model by Eq. (37) can be studied from the combination of ARXs model with the approximation tools of neuronal network with radial basic functions, RBFs. The obtained results achieved good approximations in behavior of non-linear systems. Fig. 8 shows the prediction and the subsequent simulation of the inlet gas temperature versus time with the ARX–RBF non-linear model [23,24]. As it can be seen, the non-linear approximation presents better results in the prediction than in the simulation. This is due to an overfitting in the approximation functions of the experimental data when the function is identified.

### 5. Conclusions

The study of the olive-oil mill waste rotary dryers is mainly based on the furnace and trommel mathematical models. An

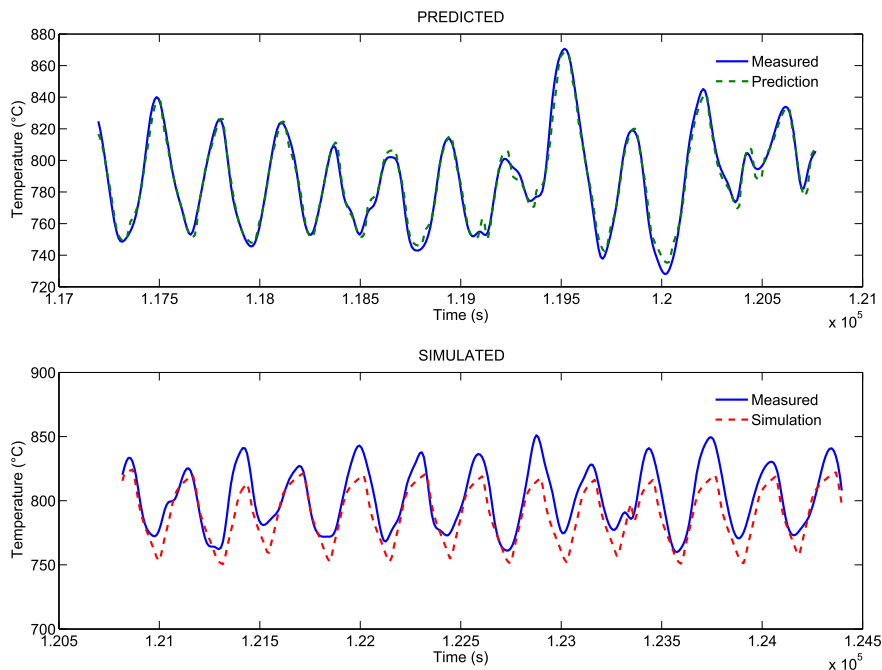


Fig. 8. Predicted-simulated inlet gas temperature versus time with the ARX–RBF model.

exhaustive control of the different characteristic parameters of both systems implies an improvement in the performance and an optimization of resources used and products obtained. Special emphasis is placed on obtaining the drying rate and the control of the gas temperature with a view to a design strategy, optimization and process control. Controlling the gas temperatures in the furnace and drying rate in each of the different sections of the trommel means an important saving of the dry de-oiled pomace that feeds the furnace and an olive-oil mill waste optimum drying. Furthermore, the control of temperature in the drying gases and in the olive-oil mill waste is vital to prevent the occurrence of PAHs. This implies high quality oil and appropriate for human consumption.

The mathematical models of the trommel and the furnace are very complicated. These models are based on principles of the mass and energy conservation. However, in these models the equations of momentum are not present. This can generate a problem even more complex to solve. For future research, Computational Fluid Dynamics techniques would need to be employed. These techniques can simulate the physical and chemical phenomena which occur in olive-oil mill waste rotary dryers.

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## Experimental determination of effective moisture diffusivity during the drying of clean olive stone: Dependence of temperature, moisture content and sample thickness

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### ABSTRACT

Drying of clean olive stone (free of olive pulp) means a revaluation as biofuel especially used for thermal processes. This work has studied the time-dependent effective moisture diffusivity from isothermal drying experiments in a convective dryer. A new method, based on a modification of the *simplified method*, has been established to calculate this coefficient. A comparison between this method and the *slope method* has been carried out and similar results have been obtained. *Modified simplified method* requires fewer calculations than the *slope method*. The dependence between the effective moisture diffusivity, the temperature and the moisture ratio was analyzed by multiple regression analysis from a second order multivariate polynomial model and the mechanisms of moisture transport were exposed. Finally, the activation energy was analyzed and its values were shown with respect to the moisture ratio and the sample thickness.

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### 1. Introduction

Biomass is one of the most important renewable energy sources in the production of thermal energy. In the last years, the price of fossil fuels has made that olive stone is emerging as one of the biomass products most highly valued in the production of thermal energy, especially in the European Union which produces more than 75% of the total of olive oil in the world [1]. Olive stone is particularly used for space heating in the industry, residential buildings and homes [2]. The excellent combustion characteristics due to low ash content, low sulfur content and a net calorific value of 19,200 kJ/kg are very well suited for biomass boilers [3].

Olive stone is obtained in the olive oil extraction process. It is contained in the main by-product of the olive oil, the *alpeorujo*. Approximately 80% of olive is a thick sludge formed by pulp, skin, olive stones and vegetation water with organic compounds. The *alpeorujo*, with a moisture content of 60–70%, is a serious environmental problem due to its high biochemical oxygen demand (BOD) and should be dried in industrial rotary dryers of the extracting plants [4]. After drying and subsequently treatments with solvents, olive pomace oil and “*orujillo*” (another biomass product) are obtained. However, olive stone is

separated from the *alpeorujo*, before drying, in the olive oil mills by means of mechanical procedures [5]. Its moisture content ranges between 20% and 30% (wet basis), which depends on several factors such as particle sizes, quantity of pulp and the conditions in the stages of the olive oil extraction process (washing, milling, crushing and kneading) [6]. However, recently new techniques in the olive stone separation process are allowing the obtainment of a by-product free of olive pulp, which implies moisture contents less than 20% (wet basis) and avoids possible problems in boilers.

Nevertheless, drying of olive stone up to equilibrium moisture content is an important stage for its revaluation as biofuel. The combustion yield in the biomass boilers increases when the olive stone moisture content decreases and high moisture contents cause water vapor condensations which are unfavorable in the biomass boilers [7]. Furthermore, drying process contributes to a cleaner production with a significant reduction in the cost of transport [8].

The porous nature of olive stone is similar to olive cake (three-phase system) and *alpeorujo* (two-phase system). The drying of olive cake [9–13], *alpeorujo* [14–19] and olive stone [20] has been studied by different researchers. All studies agree that the vast majority of the drying of these by-products is mainly produced by diffusion, capillarity and evaporation-condensation phenomena, in the falling rate period. In this sense, researchers usually obtain the average values of the effective moisture diffusivity in all drying process. However, effective moisture diffusivity can be studied as a time-dependent variable which represents

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### Nomenclature

$a, b$	Coefficients of the quadratic polynomial fit.
$a_0, a_1, a_2, a_3, a_4, a_5$	Coefficients of the second order multivariate polynomial model.
$D_{eff}$	Effective moisture diffusivity ( $m^2 \cdot s^{-1}$ )
$D_0$	Pre-exponential factor of the Arrhenius equation ( $m^2 \cdot s^{-1}$ )
$E_a$	Activation energy ( $J \cdot mol^{-1}$ )
$F_0$	Fourier diffusion number
$L$	Thickness of the slab (m)
$m$	Slope
$R$	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 ( $^{\circ}C, K$ )
$x$	Spatial dimension of mass transport (m)
$X_0$	Initial moisture content (kg moisture/kg dry matter)
$X_t$	Moisture content at time $t$ (kg moisture/kg dry matter)
$XR$	Dimensionless moisture ratio

an overall moisture transport property [21]. This variable involves not only diffusion phenomenon, but also other phenomena such as capillary movements, vaporization-condensation sequence flow, Knudsen diffusion, non-Fickian or stress-driven diffusion [22].

Commonly, in the field of numerical modeling of heat and mass transfer, the values of effective moisture diffusivity are constant [23] or are based on an Arrhenius type relationship which depend on the temperature [24,25]. In this sense, effective moisture diffusivity of olive stone can be estimated experimentally as an explicit function of moisture content, temperature and shrinkage. The objective of this work was to determine the effective moisture diffusivity of clean olive stone (free of olive pulp) in a convective dryer and its relationship with other variables such as drying air temperature and moisture ratio, for each sample thickness studied. A new method to calculate the time-dependent effective moisture diffusivity has been employed, the *modified simplified method*. The results obtained have been compared to the *slope method* and similar outcomes have been found. Finally, an experimental relationship for the activation energy with respect to moisture ratio and sample thickness was calculated.

## 2. Materials and methods

### 2.1. Materials

Clean olive stone was supplied by several olive oil mills in the province of Jaén (Spain). Samples were obtained during the olive harvesting period, at the beginning of November 2014. This ensured the original moisture content in all samples studied. Olive stone, free of olive pulp, presented different particle sizes. To obtain the distribution of particle sizes, a quantity of solids was screened using a vibratory screen (Restch, Mod. Vibro). The results showed that clean olive stones had a particle size between 7 and 5 mm (9.7%), 5–3 mm (43.1%), 3–1 mm (36.9%), and <1 mm (10.3%). Approximately, an average particle size of 3.1 mm was found. Similar results have been obtained by other authors [26]. Regarding the porosimetric data of olive stone, Cuevas et al. [27] have found a total cumulative volume (TCV) of  $0.172 \text{ cm}^3 \cdot \text{g}^{-1}$ , a specific surface area (SSA) of  $15.8 \text{ m}^2 \cdot \text{g}^{-1}$  and an average pore diameter (APD) of 6.92 nm using the mercury porosimetry method. Fig. 1 shows the SEM images in the external and internal surface of a particle of olive stone which were performed with a Merlin—Carl Zeiss instrument operating at 15 kV. The moisture content of clean olive stone

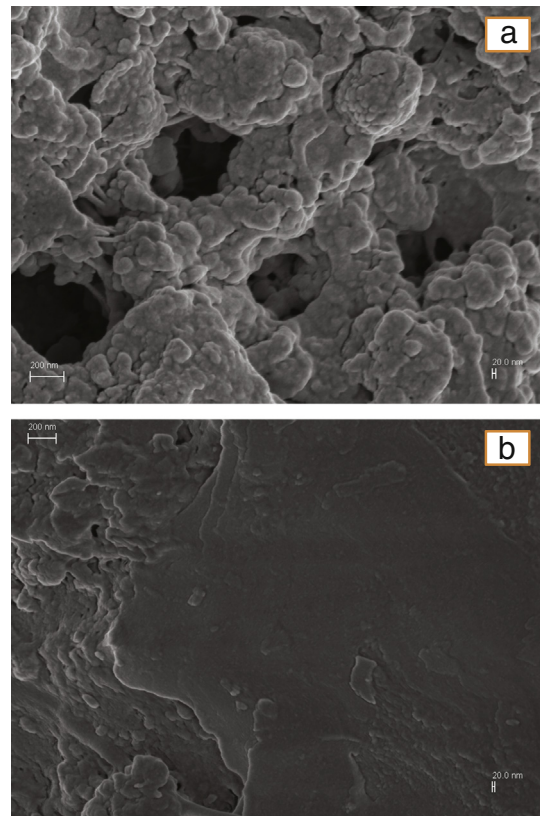


Fig. 1. SEM images of crushed olive stone, (a) external surface and (b) internal surface.

samples was obtained by drying in an oven (Mettmert GmbH + Co. KG, SNB 167 Model 100, Germany) at  $105^{\circ}C$  for 24 h. Drying of samples was carried out in triplicate. An average initial moisture content of  $19 \pm 0.3\%$  (wet basis) was found. A value of equilibrium moisture content of  $7 \pm 0.5\%$  was obtained. Surrounding air presented a relative humidity of 50% and a temperature of  $20^{\circ}C$ .

### 2.2. Experimental procedure

Experiments design was based on isothermal tests at four drying air temperature (100, 150, 200 and  $250^{\circ}C$ ) and three sample thickness (10, 20 and 30 mm), with a drying air velocity of  $1 \pm 0.1\%$  m/s. The

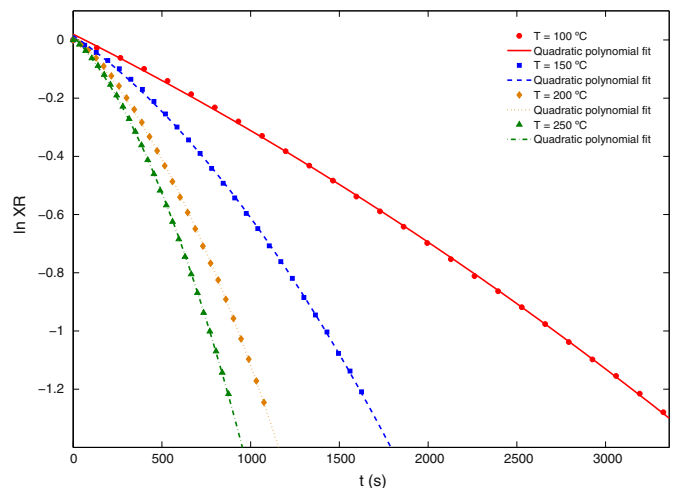


Fig. 2. Logarithmic drying curves at different temperatures and their fit by quadratic polynomial for  $L = 10$  mm.

**Table 1**  
Slope, R<sup>2</sup> and RMSE values in the approach by second order polynomial of the logarithmic drying curves.

Sample thickness (mm)	Temperature (°C)	$\frac{d(\ln(XR))}{dt}$	R <sup>2</sup>	RMSE
10	100	$2(-2.64 \cdot 10^{-8}) \cdot t - 3.059 \cdot 10^{-4}$	0.9998	0.005195
	150	$2(-2.033 \cdot 10^{-7}) \cdot t - 4.27 \cdot 10^{-4}$	0.9998	0.005238
	200	$2(-5.48 \cdot 10^{-7}) \cdot t - 5.889 \cdot 10^{-4}$	0.9998	0.005661
	250	$2(-8.797 \cdot 10^{-7}) \cdot t - 6.408 \cdot 10^{-4}$	0.9999	0.004508
20	100	$2(-1.345 \cdot 10^{-9}) \cdot t - 2.16 \cdot 10^{-4}$	0.9999	0.002951
	150	$2(-3.15 \cdot 10^{-8}) \cdot t - 3.156 \cdot 10^{-4}$	0.9997	0.006567
	200	$2(-1.017 \cdot 10^{-7}) \cdot t - 4.479 \cdot 10^{-4}$	0.9999	0.004074
	250	$2(-2.969 \cdot 10^{-7}) \cdot t - 5.112 \cdot 10^{-4}$	0.9999	0.002742
30	100	$2(-2.227 \cdot 10^{-14}) \cdot t - 1.802 \cdot 10^{-4}$	1	0.002616
	150	$2(-8.185 \cdot 10^{-9}) \cdot t - 3.118 \cdot 10^{-4}$	0.9997	0.006493
	200	$2(-4.707 \cdot 10^{-8}) \cdot t - 3.697 \cdot 10^{-4}$	0.9997	0.004657
	250	$2(-1.033 \cdot 10^{-7}) \cdot t - 4.406 \cdot 10^{-4}$	0.9999	0.004193

drying process was performed in a drying tunnel designed by the Department of Mechanical and Mining Engineering of University of Jaén, described previously by Gómez-de la Cruz et al. (2015) [28]. The dryer mainly consists of a centrifugal fan to supply the air, a tunnel of 2 m length with thermal insulation and a square section of 0.15 m, electrical resistances (up to 45 kW) and a PID (Proportional-Integral-Differential) controller which controls the drying air temperature into the sample studied. Clean crushed olive stones (endocarps in fragments) were uniformly distributed and dried in a rectangular tray of 100 mm of width and 150 mm of length for different heights (thicknesses of 10, 20 and 30 mm). The slab-shaped tray was placed over a precision balance (Blauscal AH1200) with an error of ±0.01 g. The balance was connected to personal computer. Moisture loss was recorded during drying in computer files. The experiments were continued approximately till the equilibrium moisture content was reached. After testing, to obtain the exact final moisture content, samples were dried up to 0% moisture content in the oven at 105 °C for 24 h.

**3. Results and discussion**

The mechanisms of moisture transport during drying can be modeled mathematically from the Fick's second law diffusion (Eq. (1)):

$$\frac{\partial(XR)}{\partial t} = D_{eff} \frac{\partial^2(XR)}{\partial x^2} \tag{1}$$

where  $D_{eff}$  is the effective moisture diffusivity ( $m^2 \cdot s^{-1}$ ),  $t$  is the drying time (s),  $x$  is the spatial dimension of mass transport (m), and  $XR$  is the moisture ratio and can be expressed as the moisture content at time  $t$  divided by initial moisture content,  $X_t / X_0$  (dry basis).

One of the solutions of this partial differential equation for one dimensional mass transport in infinite slab geometry has been proposed by Crank (1975) (Eq. (2)) [29]. The solution should be applied taking into account suppositions such as constant drying temperature, migration by diffusion and negligible shrinkage.

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

In this equation  $L$  is the sample thickness (m). Effective moisture diffusivity is usually used as a measure of the variation of moisture content, independently of the mechanism actually involved. Eq. (2) can be simplified utilizing the first term of the series when the drying times are considerable as follows:

$$XR = \frac{8}{\pi^2} \exp\left(-\frac{\pi^2 D_{eff} t}{L^2}\right) \tag{3}$$

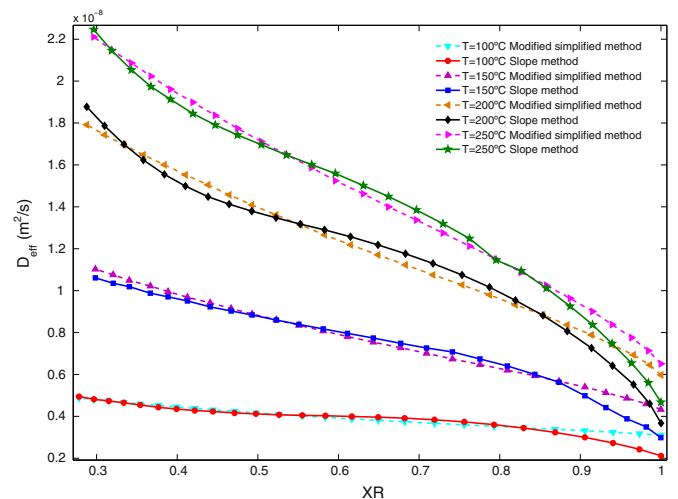
Effective moisture diffusivity is calculated from experimental data obtained in the drying process. The experimental representation  $\ln(XR)-t$  can obtain this variable from Eq. (4) as follows:

$$D_{eff} = -\frac{m \cdot L^2}{\pi^2} \tag{4}$$

where  $m$  is the slope of the straight line which fits the experimental data. This method is considered for many researchers as *simplified method* and it allows calculating a constant value for the whole process. However, during the drying process of clean olive stone, effective moisture diffusivity was found to be a dependence function of temperature and moisture content. In this work, a new method based on a modification of the *simplified method* has been used for the determination of a time-dependent effective diffusion coefficient. *Modified simplified method* consists of fitting the experimental data,  $\ln(XR)-t$ , replacing a linear equation by a quadratic equation. Fig. 2 shows the plot  $\ln(XR)-t$  for tests carried out for a sample thickness of 10 mm and the second order polynomial fit proposed for each experiment.

Therefore, in general case of nonlinear drying curves, *modified simplified method* can be applied to estimate the time-dependent effective moisture diffusivity from the derivate of the quadratic equations as follows:

$$D_{eff}(t) = -\frac{d(\ln(XR))}{dt} \cdot \frac{L^2}{\pi^2} = -(2a \cdot t + b) \cdot \frac{L^2}{\pi^2} \tag{5}$$



**Fig. 3.** Comparison between the *slope method* and the *modified simplified method* in the calculation of moisture ratio-dependent effective moisture diffusivity.  $L = 10$  mm.

**Table 2**  
Parameters for the second order multivariate polynomial model, Eq. (10), and statistical criteria in each sample thickness.

Sample thickness (mm)	Type of method	$a_0$	$a_1$	$a_2$	$a_3$	$a_4$	$a_5$	$R^2$	RMSE
10	Modified simplified method	$-1.231 \cdot 10^{-8}$	$1.96 \cdot 10^{-10}$	$7.833 \cdot 10^{-9}$	$-1.252 \cdot 10^{-13}$	$-1.244 \cdot 10^{-10}$	$1.472 \cdot 10^{-9}$	0.9963	$3.144 \cdot 10^{-10}$
10	Slope method	$-1.482 \cdot 10^{-8}$	$1.946 \cdot 10^{-10}$	$1.779 \cdot 10^{-8}$	$-1.183 \cdot 10^{-13}$	$-1.273 \cdot 10^{-10}$	$-6.626 \cdot 10^{-9}$	0.9884	$5.73 \cdot 10^{-10}$
20	Modified simplified method	$-1.519 \cdot 10^{-8}$	$2.061 \cdot 10^{-10}$	$2.33 \cdot 10^{-8}$	$4.634 \cdot 10^{-13}$	$-2.882 \cdot 10^{-10}$	$5.985 \cdot 10^{-9}$	0.9958	$8.08 \cdot 10^{-10}$
20	Slope method	$-1.904 \cdot 10^{-8}$	$1.962 \cdot 10^{-10}$	$4.156 \cdot 10^{-8}$	$4.944 \cdot 10^{-13}$	$-2.92 \cdot 10^{-10}$	$-9.609 \cdot 10^{-9}$	0.9962	$7.795 \cdot 10^{-10}$
30	Modified simplified method	$-3.198 \cdot 10^{-8}$	$4.972 \cdot 10^{-10}$	$2.586 \cdot 10^{-8}$	$3.181 \cdot 10^{-15}$	$-3.532 \cdot 10^{-10}$	$1.104 \cdot 10^{-8}$	0.9971	$9.626 \cdot 10^{-10}$
30	Slope method	$-4.199 \cdot 10^{-8}$	$4.689 \cdot 10^{-10}$	$7.212 \cdot 10^{-8}$	$7.193 \cdot 10^{-14}$	$-3.554 \cdot 10^{-10}$	$-2.753 \cdot 10^{-9}$	0.9966	$1.032 \cdot 10^{-9}$

where  $a$  and  $b$  are the coefficient of quadratic equation. Statistical criteria such as coefficient of determination,  $R^2$ , and root mean square error,  $RSME$ , were utilized to obtain the quality of fit. These statistical criteria can be calculated as:

$$R^2 = \frac{\sum_{i=1}^N (XR_{cal,i} - \overline{XR_{exp,i}})^2}{\sum_{i=1}^N (XR_{exp,i} - \overline{XR_{exp,i}})^2} \tag{6}$$

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^N (XR_{exp,i} - XR_{cal,i})^2} \tag{7}$$

where  $N$  is the number of data and the subscripts  $exp$  and  $cal$  mean experimental and calculated, respectively. Table 1 indicates the values of the derivate and their values of  $R^2$  and  $RSME$  in each experiment.

The results obtained by the *modified simplified method* have been compared to those calculated by the *slope method*, which has been widely used for a large variety of agricultural products and foods [30–32]. *Slope method* has been applied to estimate the effective moisture diffusivity in experimental nonlinear logarithmic drying curves as a function of moisture ratio. The slope of the experimental drying curves  $(dXR / dt)_{exp}$ , the experimental drying rate, is determined from experimental data. And the slope of the theoretical curves  $(dXR / dFo)_{the}$  is calculated by differentiating the moisture ratio, Eq. (3), with respect to

the Fourier diffusion number (Eq. (8)). Effective moisture diffusivity can be obtained at a given moisture ratio from Eq. (9).

$$Fo = \frac{D_{eff} \cdot t}{L^2} \tag{8}$$

$$D_{eff} = \frac{(dXR/dt)_{exp}}{(dXR/dFo)_{the}} \cdot L^2 \tag{9}$$

Both *modified simplified method* and *slope method* can be utilized in materials with a high degree of shrinkage substituting the constant value of sample thickness,  $L$ , by a time-dependent function,  $L(t)$ , which indicates the variation of sample thickness in the drying experiment. However, the results obtained in the drying experiment of clean olive stone did not show this behavior due to its granular nature. Since each value of drying time is linked to a value of moisture ratio, effective moisture diffusivity can be expressed in a form  $D_{eff} = D_{eff}(XR)$  for each experiment. Fig. 3 depicts the moisture ratio-dependence effective moisture diffusivity, for each drying temperature and a sample thickness of 10 mm, calculated from these methods. As can be seen, the results obtained were very similar between both methods.

However, numerical techniques related to heat and mass transfer in porous media require a calculation more exhaustive of the properties on which these phenomena depend. The experimental design from isothermal drying tests carried out in this research allowed estimating the effective moisture diffusivity as a function of moisture ratio and

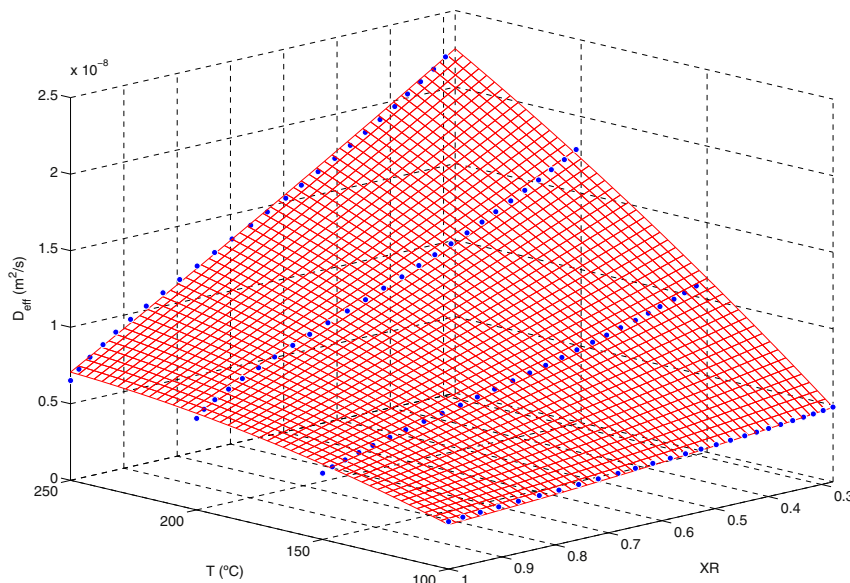


Fig. 4. Mathematical surface plot of effective moisture diffusivity in terms of moisture ratio and temperature by Eq. (10).  $L = 10$  mm.

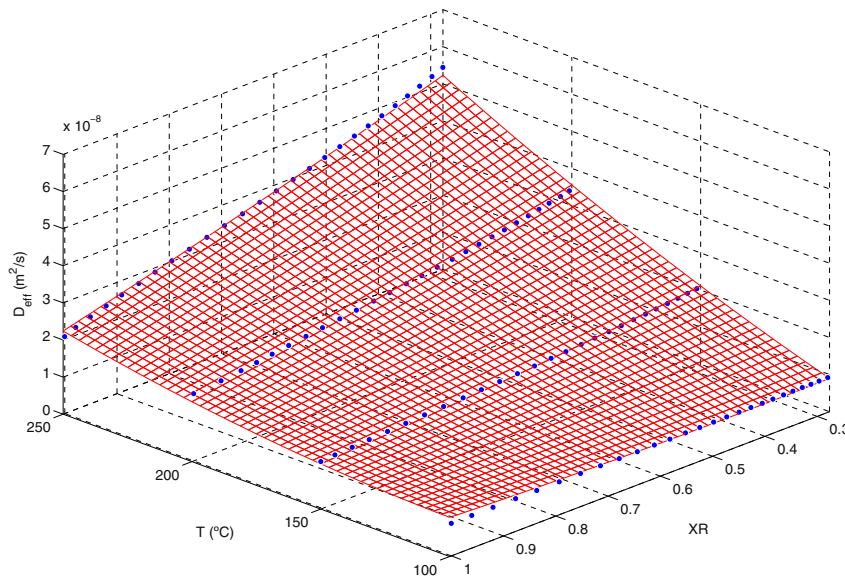


Fig. 5. Mathematical surface plot of effective moisture diffusivity in terms of moisture ratio and temperature by Eq. (10).  $L = 20$  mm.

temperature for each sample thickness,  $D_{eff} = D_{eff}(T, XR)$ , by means of multiple regression analysis. Furthermore, at high temperatures, the sample temperature is quickly reached, and the difference of temperature between the surface and the interior of the sample is hardly appreciated as the drying experiment is performed [33]. A second order multivariate polynomial model at a confidence level of 95% was performed (Eq. (10)). This model presented excellent results of fit for the experimental function  $D_{eff} = D_{eff}(T, XR)$ , eliminating the use of high number of coefficients (Table 2).

$$D_{eff}(T, XR) = a_0 + a_1 \cdot T + a_2 \cdot XR + a_3 \cdot T^2 + a_4 \cdot T \cdot XR + a_5 \cdot XR^2 \quad (10)$$

Figs. 4–6 show the experimental function  $D_{eff} = D_{eff}(T, XR)$  for each sample thickness obtained from the *modified simplified method*. These mathematical surfaces allowed obtaining the effective moisture

diffusivity values in the range of the actual operation conditions in the drying of clean olive stone.

Similar behavior has been obtained in the drying of chitosan for a temperature of 60 °C, a drying air velocity of 1.5 m·s<sup>-1</sup> and a sample thickness of 4 mm [34], and during baking of white cake for temperatures between 50 and 160 °C, an air velocity of 0.56 m·s<sup>-1</sup> and a sample thickness of 3 mm [35].

The mechanisms of mass transport during drying in the warming-up, constant and falling rate periods are rather complex and it is often very difficult to identify them. The mechanisms of moisture transport have been detailed for olive stone with olive pulp and *alpeorujo* from drying rates curves [17,20] and it has been demonstrated that there is no constant rate period. Drying phenomena of clean olive stone can be detailed from the behavior of effective moisture diffusivity.

Figs. 4–6 indicate that the effective moisture diffusivity values increased as the moisture ratio decreased and drying temperature

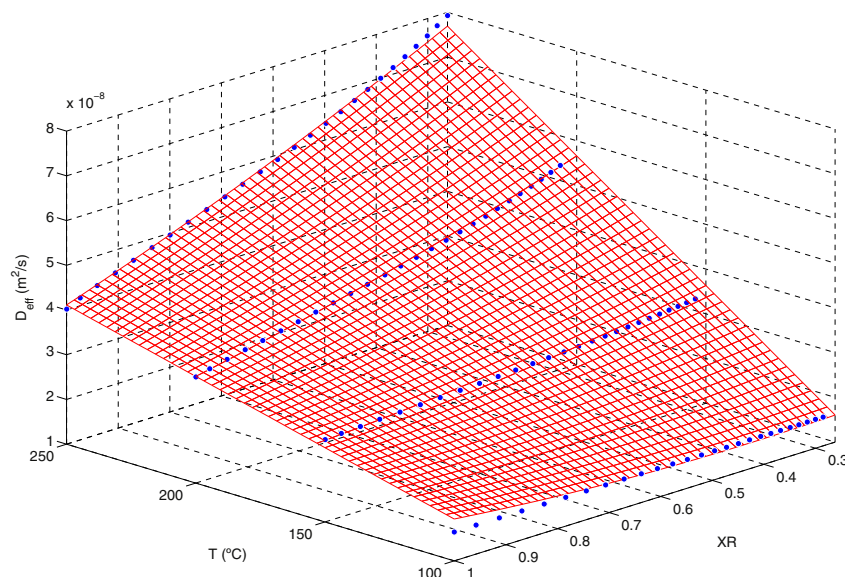


Fig. 6. Mathematical surface plot of effective moisture diffusivity in terms of moisture ratio and temperature by Eq. (10).  $L = 30$  mm.

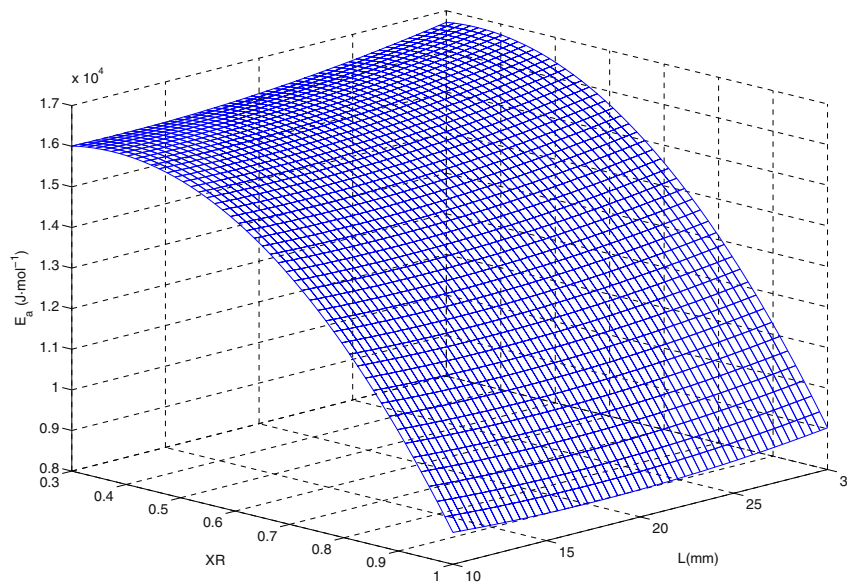


Fig. 7. Representation of activation energy as a function of moisture ratio and sample thickness.

increased. At the beginning of the drying process of each test, a short time was needed to establish the stationary experimental drying conditions. This stage is known as *the warming-up period*. The drying air is blown over the sample and the heat is transferred to the surface. The latent heat of vaporization provokes the water evaporation. As the moisture content decreased in the surface, a region of lower water vapor pressure was originated. A water pressure gradient is manifested from the water interior to the drying air, which provides the drying force for the removal of water. Thus, this stage is governed by diffusion through the boundary layer because of the difference in moisture content concentrations between the sample surface and the drying air. Fig. 3 shows *the warming-up period* between  $XR = 1$  and  $XR = 0.85$  where the effective moisture diffusivity is low.

The *first falling rate period* is characterized by a moderate increase of effective moisture diffusivity due to the moisture transport by *capillarity diffusion*. In this stage, as evaporation occurred, the surface of the sample was practically saturated due to constant flow of water from the interior of the sample. Thus, the main mechanism of mass transport in this period is originated by the gradient of the capillary potential. On the other hand, a liquid flow in pores, which is created due to the difference of the pressure caused by friction, appeared. As these two mechanisms acted, the surface of the sample abandoned the saturated state and there was only a continuous liquid state within the porous material (*funicular state* [36]). At high temperatures, higher than 100 °C, first falling rate period can be included in the *warming-up period* [17]. It is shown for effective moisture diffusivity values between  $XR = 1$  and  $XR = 0.4$ , while at low temperature this stage is manifested for values between  $XR = 0.9$  and  $XR = 0.4$ .

Finally, in the *second falling rate*, the surface is totally dry and the continuous channels of water within of porous medium have disappeared (*pendular state* [36]). This stage can be observed when the effective moisture diffusivity reached a moisture ratio value of 0.4 (Fig. 3). As the moisture ratio decreased below this value, effective moisture diffusivity was slightly increased. At low temperatures, liquid diffusion was produced in the pores due to the concentration gradient, and, at high temperatures, vapor was transported by the differences in total pressure (*hydrodynamic flow*). Due to high drying temperatures, in the experiments carried out at 250 °C when the moisture content was close to equilibrium moisture content, the samples of clean olive stone were darkened. This phenomenon caused release of volatile materials, but there was no combustion. Experiments were stopped when the volatile matters appeared. This indicated that the possible combustion risk

depends on the quantity of olive pulp in the drying samples for these same temperatures, as other researchers have demonstrated [20].

Activation energy can be expressed as a function of moisture ratio and sample thickness from the study of the mathematical surfaces proposed for the effective moisture diffusivity. This value can be obtained for each moisture ratio taking into account the drying temperatures (100, 150, 200 and 250 °C) by an Arrhenius type relationship as follows:

$$D_{eff} = D_0 \cdot \exp\left(-\frac{E_a}{RT}\right) \quad (11)$$

where  $D_0$  is the pre-exponential factor ( $\text{m}^2 \cdot \text{s}^{-1}$ ),  $R$  is the universal gas constant ( $\text{J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$ ),  $T$  is the absolute temperature (K) and  $E_a$  is the activation energy ( $\text{J} \cdot \text{mol}^{-1}$ ). The activation energy was found out for each moisture ratio and sample thickness. The function  $\ln D_{eff}$  versus  $1/T$  was obtained for the values of moisture ratio of 1, 0.9, 0.8, 0.7, 0.6, 0.5, 0.4 and 0.3 for each sample thickness. Fig. 7 plots the activation energy in function of these variables.

As can be observed, activation energy has a strongly dependence to moisture ratio. These values decreased as the moisture ratio increased. This indicates that the energy to activate the mechanisms of mass transport was small at the beginning of the experiments and it was increased as the drying process was carried out. However, activation energy values rose slightly when the sample thickness grew as well.

#### 4. Conclusion

The experimental design in the drying of clean olive stone allowed studying the effective moisture diffusivity as overall mass transport property. The new method developed in this work, *modified simplified method*, obtained similar results to those achieved in the *slope method*. This method estimated this variable in terms of moisture ratio analyzing only the experimental data  $\ln(XR)-t$ . A multiple regression analysis was proposed to obtain the experimental function  $D_{eff} = D_{eff}(T, XR)$  for each sample thickness. Results obtained in this work will serve as a starting point in future research from numerical techniques and simulations in the drying of clean olive stone.

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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 °C and 120 °C, while the inlet temperature may fluctuate between 400 °C and 500 °C. 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.

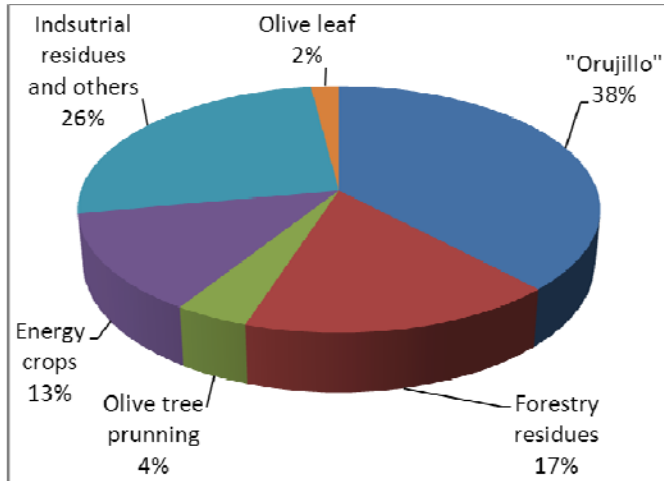


Fig. 1. Biofuels utilized in the generation of electrical power in Andalusia (Spain) [4].

## 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 °C for 24 hours. Drying samples were performed in triplicate. An average moisture content of  $60 \pm 5\%$  (wet basis) was found. The same procedure was applied to obtain the equilibrium moisture content which was estimated at  $8 \pm 1\%$  (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  $\pm 0.01$  g. The precision balance was connected to personal computer by USB port. The information was stored in files and finally, was analyzed.

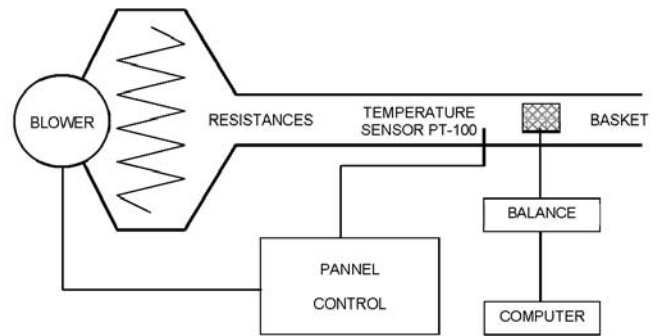


Fig. 2. Drying tunnel scheme.

### 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 100°C and 425°C.
- 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.

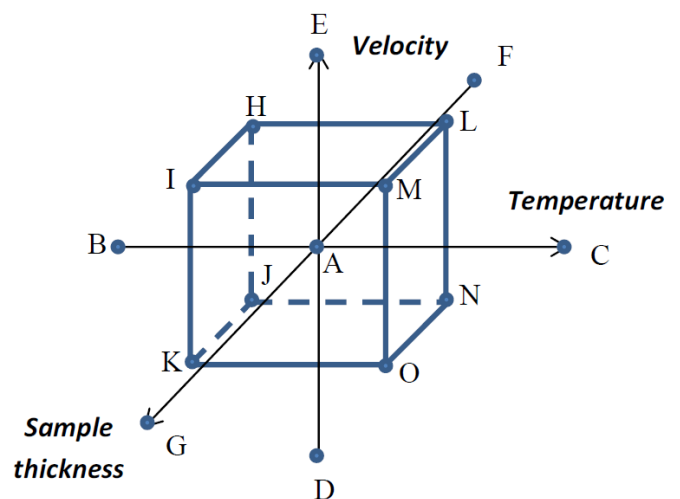


Fig. 3. Central composite design for three variables.

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

TEST	T (°C)	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):

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

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 high 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.

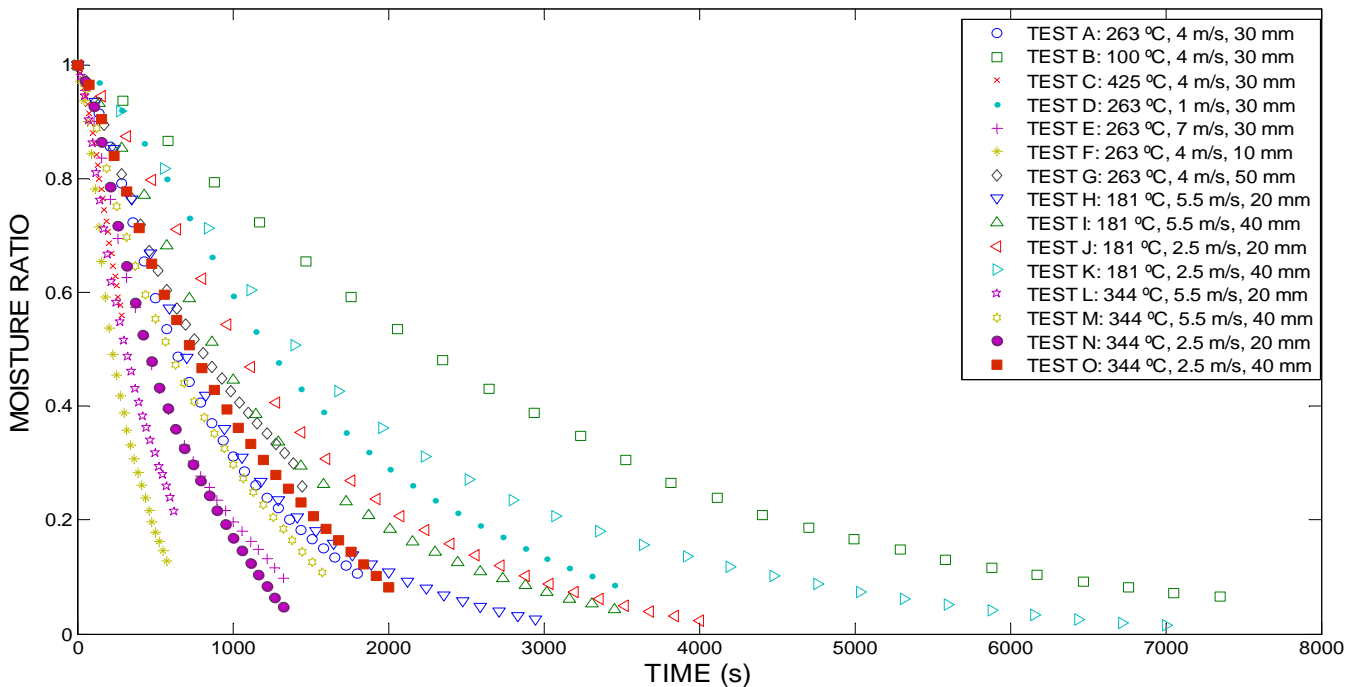


Fig. 4. Drying curves of the alpeorujo for each test.

TABLE II. MATHEMATICAL MODELS IN THE DRYING OF ALPEORUJO

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

TABLE III. STATISTICAL RESULTS OBTAINED, R<sup>2</sup> Y RMSE, FROM DIFFERENT THIN-LAYER DRYING MODELS

TEST	MATHEMATICAL MODEL									
	Lewis		Page		Modified Page		Henderson and Pabis		Logarithmic	
	R <sup>2</sup>	RMSE	R <sup>2</sup>	RMSE	R <sup>2</sup>	RMSE	R <sup>2</sup>	RMSE	R <sup>2</sup>	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
TEST	MATHEMATICAL MODEL									
	Wang and Singh		Two Term		Approach of Diffusion		Midilli et al.		Two Term Gaussian	
	R <sup>2</sup>	RMSE	R <sup>2</sup>	RMSE	R <sup>2</sup>	RMSE	R <sup>2</sup>	RMSE	R <sup>2</sup>	RMSE
A	0,9975	0,0135	0,9975	0,0135	0,9996	0,0054	0,9991	0,0083	<b>1,0000</b>	<b>0,0011</b>
B	0,9993	0,0073	0,9999	0,0024	0,9999	0,0025	0,9999	0,0023	<b>0,9999</b>	<b>0,0022</b>
C	0,9983	0,0056	0,9999	0,0014	0,9999	0,0014	0,9999	0,0014	<b>0,9999</b>	<b>0,0015</b>
D	0,9965	0,0168	0,9968	0,0161	0,9996	0,0054	0,9992	0,0082	<b>1,0000</b>	<b>0,0012</b>
E	0,9972	0,0142	0,9985	0,0103	0,9996	0,0054	0,9991	0,0078	<b>0,9999</b>	<b>0,0024</b>
F	0,9963	0,0167	0,9955	0,0184	0,9991	0,0084	0,9981	0,0119	<b>0,9999</b>	<b>0,0026</b>
G	0,9982	0,0092	0,9981	0,0094	0,9988	0,0075	0,9988	0,0076	<b>0,9998</b>	<b>0,0032</b>
H	0,9904	0,0278	0,9973	0,0148	0,9995	0,0064	0,9989	0,0096	<b>1,0000</b>	<b>0,0017</b>
I	0,9925	0,0242	0,9977	0,0133	0,9995	0,0064	0,9990	0,0090	<b>1,0000</b>	<b>0,0014</b>
J	0,9951	0,0205	0,9971	0,0157	0,9996	0,0057	0,9992	0,0084	<b>1,0000</b>	<b>0,0010</b>
K	0,9837	0,0352	0,9982	0,0118	0,9993	0,0075	0,9986	0,0103	<b>1,0000</b>	<b>0,0012</b>
L	0,9979	0,0109	0,9809	0,0333	0,9993	0,0062	0,9988	0,0084	<b>0,9999</b>	<b>0,0027</b>
M	0,9975	0,0128	0,9998	0,0038	0,9987	0,0094	0,9987	0,0091	<b>0,9999</b>	<b>0,0023</b>
N	0,9969	0,0160	0,9977	0,0137	0,9979	0,0132	0,9982	0,0123	<b>0,9999</b>	<b>0,0026</b>
O	0,9981	0,0116	0,9993	0,0070	0,9985	0,0104	0,9989	0,0088	<b>0,9999</b>	<b>0,0022</b>

### 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):

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

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 mathematical 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.

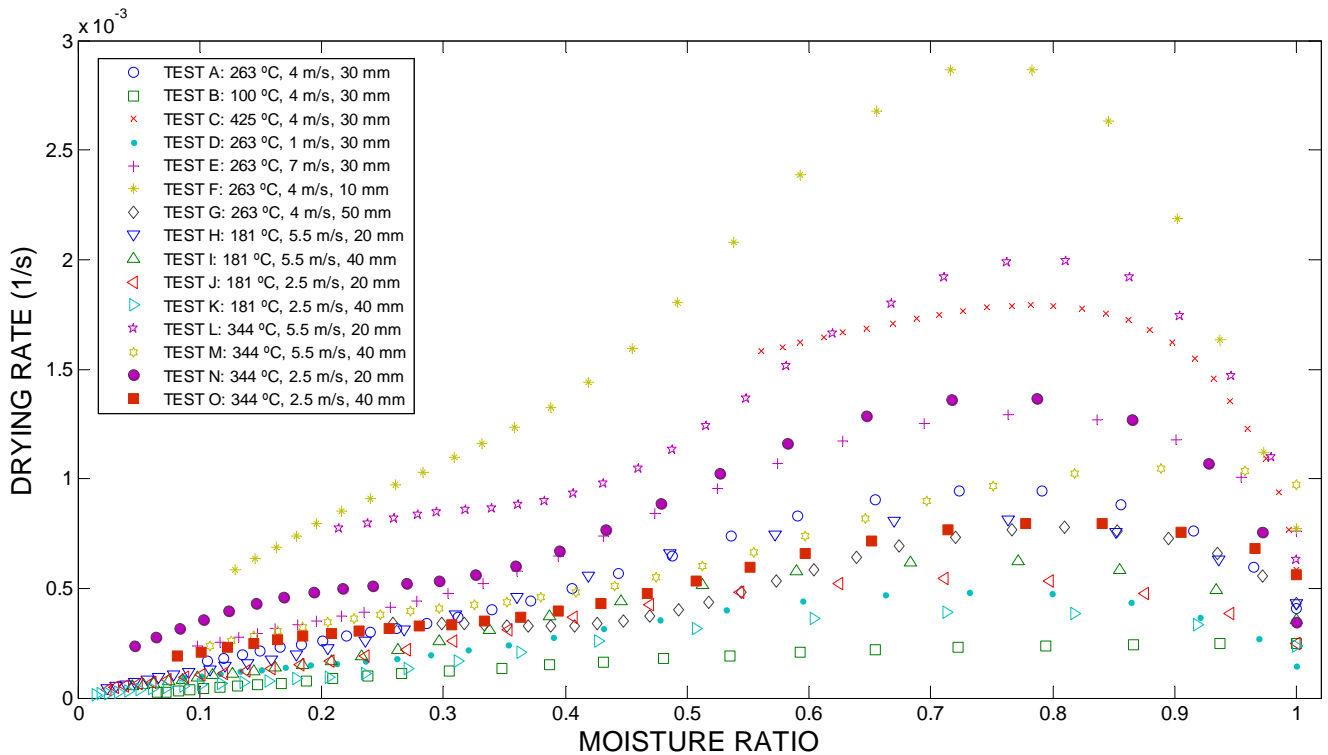


Fig. 5. Drying rate versus moisture ratio of the alpeorujo for each test.

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

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

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):

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

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):

$$D_{eff} = \frac{slope \cdot L^2}{\pi^2} \quad (6)$$

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	$1.29 \cdot 10^{-7}$	0.9956
B	$4.47 \cdot 10^{-8}$	0.9823
C	$2.05 \cdot 10^{-7}$	0.9841
D	$7.68 \cdot 10^{-7}$	0.9828
E	$1.79 \cdot 10^{-7}$	0.9923
F	$4.09 \cdot 10^{-8}$	0.9926
G	$2.44 \cdot 10^{-7}$	0.9974
H	$6.91 \cdot 10^{-8}$	0.9368
I	$1.97 \cdot 10^{-7}$	0.9574
J	$4.83 \cdot 10^{-8}$	0.9271
K	$1.23 \cdot 10^{-7}$	0.9476
L	$1.10 \cdot 10^{-7}$	0.9949
M	$2.44 \cdot 10^{-7}$	0.9844
N	$1.06 \cdot 10^{-7}$	0.9178
O	$2.15 \cdot 10^{-7}$	0.9737

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

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

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.

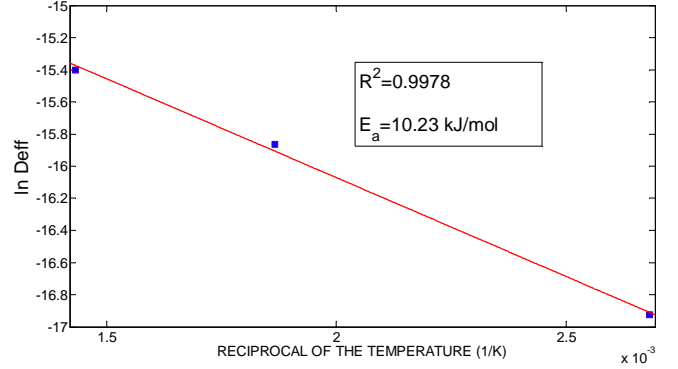


Fig. 6. Arrhenius-type relationship between effective diffusivity logarithmic and the reciprocal of absolute temperature for tests A, B and C.

Similar results can be found in the literature for olive cake. Meziante [18], with a drying air velocity in his experiments of 1 m/s, obtained values of effective diffusivity between  $0.68 \cdot 10^{-7}$  to  $2.15 \cdot 10^{-7} m^2/s$  for tests carried out at temperatures and sample thicknesses between 50 °C to 80 °C and 41, 52 and 63 mm, respectively, and activation energy values of 34.05, 36.84, 38.1 kJ/mol for each sample thickness. Göğüs and Maskan [19] calculated, for temperature between 60 °C to 80 °C, sample thicknesses of 6, 9 and 12 mm and velocity of 1.5 m/s, effective diffusivity values between  $1.84 \cdot 10^{-7}$  to  $3.94 \cdot 10^{-7} m^2/s$  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  $4.95 \cdot 10^{-10}$  to  $1.42 \cdot 10^{-9} m^2/s$  for temperatures ranging from 50-110 °C, 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 <sub>0</sub> ,k <sub>1</sub>	Constants of the mathematical models (s <sup>-1</sup> )
D <sub>eff</sub>	Effective diffusivity (m <sup>2</sup> /s)
D <sub>0</sub>	Pre-exponential factor of the Arrhenius equation (m <sup>2</sup> /s)
E <sub>a</sub>	Activation energy (kJ/mol)
L	Thickness of the slab (m)
R	Universal gas constant (kJ·mol <sup>-1</sup> ·K <sup>-1</sup> )
R <sup>2</sup>	Coefficient of determination
RMSE	Root mean square error
t	Time (s)
T	Temperature (°C, K)
v	Velocity (m·s <sup>-1</sup> )
X <sub>e</sub>	Equilibrium moisture content (kg moisture/kg dry matter)
X <sub>0</sub>	Initial moisture content (kg moisture/kg dry matter)
X <sub>t</sub>	Moisture content at time t (kg moisture/kg dry matter)
XR	Dimensionless moisture ratio
x <sub>v</sub>	Drying rate (kg moisture/(kg dry matter · s))

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