PAPER

DRYING KINETICS OF SAFFRON FLORAL BIO-RESIDUES

A. ALVARRUIZ*, C. LORENZO, G.L. ALONSO and J. SERRANO-DÍAZ

^aEscuela Técnica Superior de Ingenieros Agrónomos, Universidad de Castilla-La Mancha, Campus Universitario, 02071 Albacete, Spain

^bDepartment of Food Technology, Nutrition and Food Science, Regional Campus of International Excellence Campus Mare Nostrum, Murcia University, 30100 Murcia, Spain *Corresponding author. Tel.: +39 967599200; fax: +39 967599238 E-mail address: andres.alvarruiz@uclm.es

ABSTRACT

The kinetics of hot-air drying of saffron floral bio-residues was studied at two air-drying temperatures (70 and 90°C) and four air-flow rates (2, 4, 6 and 8 m·s¹). No constant-rate drying period was observed during drying. Ten thin-layer drying models and the theoretical Fick's diffusion model were fit by non-linear regression to drying data. Three statistical parameters, Chi-squared, correlation coefficient and relative percentage deviation were used to compare the models. Effective moisture diffusivity, calculated using Fick's diffusion model, was in the range of 0.78-1.86 x 10¹¹¹ m²¹·s¹¹. According to the statistical parameters, three drying models (the logarithmic, two-term and Midilli-Kucuk models) were equally good to describe the drying curve and fit the data better than the other models. The model constants were independent of air-flow rate. The use of air at 90 °C decreased drying time in half compared with drying at 70°C.

Keywords: saffron, floral waste, drying, thin-layer models

1. INTRODUCTION

Saffron (*Crocus sativus* L.) spice is the dehydrated stigma of the flowers of this plant of the *Iridiaceae* family. Saffron spice production worldwide is about 250 tons. According to the Ministry of Agriculture of Iran (GHORBANI, 2008), the main producer exporter of saffron spice is Iran (93.7% of world production in 2005), with an export value of \$100 million. Other countries, such as India, Greece, Spain, Morocco and Italy, are also producers and marketers of saffron spice. Spain is noted for producing saffron spice with the highest quality recognized worldwide. Moreover, Spain is also the leader in its trade, because it processes and re-exports saffron spice produced in other countries (i).

Stigma is only 7.4% of the fresh weight of a flower (SERRANO-DÍAZ et al., 2013a). Tepals, stamens and styles are also part of the flowers of saffron, but they have traditionally been thrown away. About 173,250 flowers weighing over 68 kg were used in Castilla-La Mancha (Spain) in 2009 to obtain 1 kg of saffron spice. As a result, 63 kg of these floral bioresidues (53 kg of tepals, 9 kg of stamens and 0.5 kg of styles) were generated (SERRANO-DÍAZ et al. 2013b). The introduction of the forced cultivation and mechanization of saffron production (GARVI PALAZÓN, 1987; GRACIA et al., 2008) will cause an increase in the production capacity and the concentration of these bio-residues in companies producing saffron spice, as stated in the white book of saffron (ii). This new situation is raising interest in the exploitation of saffron floral bio-residues. Many studies have demonstrated the biomedical properties of saffron tepal extracts. MOSHIRI et al. (2006) demonstrated the efficacy of the extracts in the treatment of mild-to-moderate depression. FATEHI et al. (2003) showed that they lower blood pressure and reduce the contractions induced by electrical field stimulation. HOSSEINZADEH and YOUNESI (2002) concluded that they have antinociceptive and anti-inflammatory effects. ZHENG et al. (2011) found that the saffron stamen and perianth possess significant antifungal, cytotoxic and antioxidant activity. BERGOIN (2005) extracted and characterized the volatile fraction and colorant compounds from fresh flowers and explored their use for the cosmetic, perfume and fragrance industries. The high phenolic content of the saffron floral bio-residues (SERRANO-DÍAZ et al., 2014b; NØRBÆK et al., 2002), their antioxidant properties (SERRANO-DÍAZ et al., 2012) their adequate nutritional composition (SERRANO-DÍAZ et al., 2013b) and the absence of cytotoxicity (SERRANO-DÍAZ et al., 2014a) show that these products could be used as food ingredients with high added value.

Traditionally, the remains of flowers that are generated in the production of saffron spice have been thrown near the saffron field; deterioration within hours has been observed, even though they were exposed to the sun. This spoilage could be due to their high moisture (SERRANO-DÍAZ et al., 2013b), which favors microbial attack. As in saffron stigmas, there is a need to dewater the saffron floral bio-residues the same day as the flowers are harvested. The technique used for dehydration of the stigma to produce saffron spice differs by country: sun drying, drying at room temperature in air-ventilated conditions (India, Iran and Morocco), drying at moderate temperatures (Greece and Italy) and drying at high temperatures (Spain). CARMONA et al. (2005) characterized the time-temperature profile during the traditional dehydration process in Castilla-La Mancha (Spain) compared with other dehydration processes. DEL CAMPO et al. (2010) studied the effect of mild temperature during dehydration in the main components responsible for the quality of saffron spice.

Drying is the most common way to preserve the quality of aromatic and medicinal plants (ROCHA *et al.*, 2011). Hot air dehydration, by itself or combined with infrared radiation, has been successfully used to dry a number of flower commodities, such as marigold flower (SIRIAMORNPUN *et al.* 2012), torch ginger (JUHARI *et al.*, 2012), chrysanthemums, roses (CASTRO *et al.*, 2003), chamomile (BORSATO *et al.*, 2009), daylily (MAO *et al.*, 2006)

and oregano (CESARE *et al.*, 2004) among others, while preserving their color, antioxidant properties and/or bioactive compounds. SERRANO-DÍAZ *et al.* (2013a) studied the conditions of hot-air drying of saffron floral residues to achieve minimal deterioration of the physicochemical quality of these products and concluded that the best quality was achieved with air at 90 °C combined with a flow rate of 2, 4 and 6 m s¹, but the kinetics of the drying process of these products have never been studied.

The aim of this study was to select and test the best drying model for hot-air dehydration of saffron floral bio-residues and to determine the influence of temperature and air-flow rate on dehydration kinetics.

2. MATERIALS AND METHODS

2.1. Plant material

The floral bio-residues generated by saffron spice production were from the Agrícola Técnica de Manipulación y Comercialización S.L. company (Minaya, Spain) during the 2010-2011 harvest season. The floral bio-residues were collected after separating the stigma from flowers using traditional procedures for the Protected Designation of Origin Azafrán de La Mancha. The thickness of the different floral tissues was measured with calipers. Fresh floral bio-residues were stored at -20°C.

2.2. Hot-air drying

Hot-air drying was performed in a laboratory-scale hot-air dryer (Fig. 1). The dryer was equipped with four 500-W electric resistors, coupled to an automatic temperature (\pm 0.1°C) controller. The air was impelled trough the drying bed by a 0.5-CV fan equipped with an automatic air velocity controller (\pm 0.1 m·s³). The evolution of the product was monitored by weighing the sample periodically with a Mettler (Switzerland) PM2000 balance (\pm 0.01 g) linked to a computer.

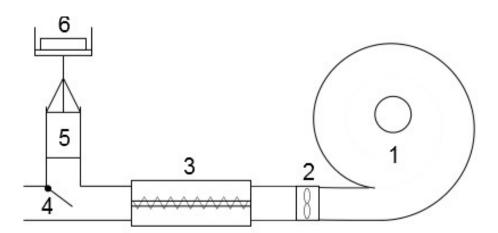


Figure 1: Hot air dryer set-up. 1: Fan, 2: Air velocity meter, 3: Electric heater, 4: Diversion valve, 5: Sample holder, 6: Scales.

Fresh floral tissues were placed on a cylindrical sample holder (9.2-cm diameter) with a perforated bottom. Sample size was kept constant (50 ± 2 g) for each experiment. Weight loss was recorded at 5-min intervals, and drying was continued until the moisture difference was lower than 5% (w/w). Dry runs were performed at temperatures of 70°C and 90 °C, with air-flow of 2 m·s¹, 4 m·s¹, 6 m·s¹ and 8 m·s¹. Reynolds numbers, calculated using the slab half-thickness as the characteristic dimension (about 5·10⁴ m), were on the order of 50, 100, 150 and 200, respectively. The sample temperature during the drying process was determined with an infrared laser thermometer (range: -33 to +250°C, accuracy: \pm 2°C).

Sample moisture level was determined with a halogen lamp moisture balance, model XM-120T (Cobos, Barcelona, Spain) at 105 °C, in triplicate. When moisture loss was less than 0.1% in 180 s, samples were considered to have reached constant mass. Nine measurements were obtained for each combination of temperature-air flow.

2.3. Mathematical models

The moisture ratio (MR) was defined as:

$$MR = \frac{M - M_e}{M_0 - M_e}$$
 Eq. 1

where M is sample moisture at time t, Me is equilibrium moisture content, and M_o is initial moisture content. All moisture content was determined on a dry basis. Because drying experiments were carried out using hot air, with very low relative humidity, the moisture ratio was simplified to M/M_0 .

The experimental data were fit to ten thin-layer drying models and to the theoretical Fick's diffusion model for a slab (Table 1). References for the model equations can be found elsewhere (AKPINAR, 2006).

Table 1: Mathematical drying models.

Model name	Model equation
Exponential	$MR = \exp(-kt)$
Page	$MR = \exp\left(-kt^n\right)$
Modified Page	$MR = \exp\left(-kt\right)^n$
Henderson and Pabis	$MR = a \exp(-kt)$
Logarithmic	$MR = a \exp(-kt) + c$
Two term	$MR = a \exp(-k_0 t) + b \exp(-k_1 t)$
Two term exponential	$MR = a \exp(-kt) + (1-a) \exp(-kat)$
Wang and Singh	$MR = 1 + at + bt^2$
Verma	$MR = a \exp(-kt) + (1-a) \exp(-gt)$
Midilli-Kucuk	$MR = \exp(-kt^n) + bt$
Fick's diffusion	Eq (2)

The thin-layer drying models are simple empirical models that give good results when the assumptions needed for developing the analytical solutions to Fick's second law, namely the surface resistance or the geometry, are not truly met. Their main drawback is that their parameters lack physical meaning. Conversely, rigorous or phenomenological models can give a hint to the mechanism of the underlying process. An innovative approach to mathematical modelling of the drying of eggplant slabs considering the shrinkage effect can be found elsewhere (BRASIELLO *et al.*, 2013; RUSSO *et al.*, 2013).

The analytical solution to Fick's second law for a slab, in the case of negligible surface resistance, is (CRANK, 1975):

$$MR = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left(-\frac{(2n+1)^2 \pi^2 Dt}{4L^2}\right)$$
 Eq. 2

where D is the effective water diffusivity ($m^2 \cdot s^4$), and L is the half-thickness of the slab. For long drying times, Eq. 2 can be simplified by taking only the first term in the summation, leading to the Henderson and Pabis equation with a theoretical value for the constant a of $8/\pi^2$.

The data were also fit to Eq. 2, and the effective diffusivity was calculated. The number of summation terms was adjusted to ensure that the error in MR was less than 0.1%.

2.4. Statistical analysis

All non-linear regressions were performed using the SOLVER optimization tool (GRG nonlinear method) included in the Microsoft Excel $2010^{\text{\tiny IM}}$ spreadsheet, by minimizing the sum of the square differences between the experimental and calculated moisture ratios. Comparison of the goodness of fit for each equation was determined by means of the following parameters: correlation coefficient (R), reduced chi-square (χ) and mean relative percentage deviation (P):

$$\chi^2 = \frac{1}{N-n} \sum_{i=1}^{n} (MR_{ei} - MR_{ci})$$
 Eq. 3

$$P(\%) = \frac{100}{N} \sum_{i=1}^{n} \frac{|MR_{ei} - MR_{ci}|}{MR_{ei}}$$
 Eq. 4

where MR_a and MR_a are experimental and predicted moisture ratios, respectively; N is the number of experimental data-points; n is the number of model parameters. Multiple linear regressions were performed to determine the influence of temperature and air-flow rate using SPSS 19.0 for Windows (SPSS Inc., Chicago, IL, USA).

3. RESULTS AND DISCUSSIONS

Figure 2 shows that the drying rate was decreasing from the beginning of the drying process, and there was no constant rate period. This pattern suggests that the drying resistance would be inside the product rather than in the outside air layer and is in agreement with the results reported for thin-layer drying of similar products, such as

saffron (AKHONDI *et al.*, 2011), betel leaves (PIN *et al.*, 2009), mint leaves (DOYMAZ, 2006) and spinach leaves (DOYMAZ, 2009).

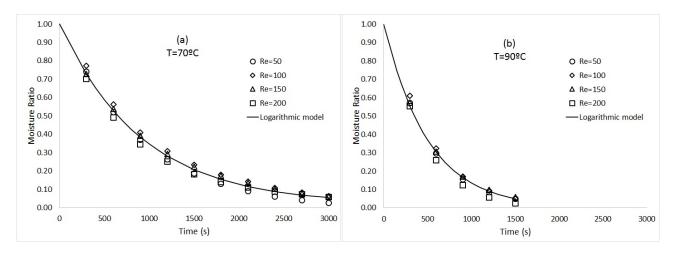


Figure 2: Drying rate of saffron flowers versus moisture ratio.

Table 2 shows the results of the statistical parameters obtained for each model. The best model was the one with the highest R-values, the lowest χ^2 values and the lowest P values. The two-term model was the best with regard to the R and χ^2 values, with all R values higher than 0.99987 and χ^2 values lower than $1.15\cdot10^4$, but the logarithmic model also gave very good agreement, with R values higher than 0.99986 and χ^2 values lower than $1.56\cdot10^4$. These two models also had very low P values (<4.8%). The Midilli-Kucuk model had the lowest P values, all lower than 4.2%, very high R values (>0.99981) and low χ^2 values ($<1.89\cdot10^4$). Therefore, the three best models were the two-term, logarithmic and Midilli-Kucuk models. The Verma model also gave a good fit, with R >0.99924, $\chi^2 < 9.40\cdot10^4$ and P <5.4%. In contrast, the theoretical Fick's diffusion model (Eq. 2) gave one of the worst fits, with R >0.98751, $\chi^2 < 3.30\cdot10^4$ and P in the range of 9.7-41.3%, while the Henderson and Pabis model gave better results than the theoretical model (R >0.99810, $\chi^2 < 2.09\cdot10^4$ and P <10.8%).

The logarithmic model has been reported by several authors as the one that gave the best fit for thin-layer drying of a number of products, such as finger millet (RADHIKA *et al.*, 2011), olive cake (AKGUN and DOYMAZ, 2005), mint leaves (DOYMAZ, 2006), spinach leaves (DOYMAZ, 2009) and apricots (TOGRUL and PEHLIVAN, 2002). PIN *et al.* (2009) found that the logarithmic model gave the best results for drying betel leaves at 40-60°C, while the Midilli and Kucuk model was better for drying at 70°C. In some instances, the two-term drying model proved to be better than the logarithmic model for drying sultana grapes (YALDIZ *et al.*, 2001). Other authors have claimed that the Midilli and Kucuk model was the best thin-layer drying model for drying potato, apple and pumpkin slices (AKPINAR, 2006) or saffron (AKHONDI *et al.*, 2011). Our results agree with those of previous works and confirm that the two-term, the logarithmic and the Midilli-Kucuk model are the three best models for drying saffron floral bioresidues. This suggests that the Verma model could also be acceptable.

Table 2: Statistical parameters of the drying models.

Model	Temperature	Re	χ^2	R	P (%)
Exponential	70	50	1.15E-04	0.99995	10.0
		100	1.10E-04	0.99903	4.4
		150	1.30E-04	0.99974	7.2
		200	3.14E-04	0.99905	13.9
	90	50	1.38E-04	0.99935	3.7
		100	5.32E-04	0.99920	11.2
		150	7.52E-05	0.99935	3.1
		200	4.83E-04	0.99917	19.0
Page	70	50	1.24E-05	0.99989	3.0
3		100	1.10E-04	0.99903	4.6
		150	2.44E-05	0.99976	2.3
		200	1.17E-04	0.99876	7.4
	90	50	3.86E-05	0.99960	5.0
		100	4.00E-05	0.99964	4.7
		150	4.90E-05	0.99946	4.7
		200	1.81E-05	0.99982	4.5
Modified Page	70	50	1.24E-05	0.99989	3.0
woulder age	70	100	1.10E-04	0.99903	4.6
		150	2.44E-05	0.99976	2.3
		200	1.17E-04	0.99876	7.4
	90	50	3.86E-05	0.99960	5.0
	50	100	4.00E-05	0.99964	4.7
		150	4.90E-05	0.99946	4.7
		200	1.81E-05	0.99982	4.5
Henderson and Pabis	70	50	1.15E-05	0.99991	4.8
Henderson and Fabis	70	100	9.40E-05	0.99924	5.4
		150		0.99924	4.5
			6.04E-05		
	00	200	2.09E-04	0.99810	10.8
	90	50	1.62E-05	0.99985	3.7
		100	2.00E-06	0.99998	1.2
		150	3.28E-05	0.99968	4.2
		200	4.03E-07	1.00000	1.1
Logarithmic	70	50	1.15E-05	0.99991	4.8
		100	1.56E-05	0.99986	2.2
		150	1.21E-05	0.99987	2.1
	00	200	3.93E-06	0.99996	1.4
	90	50	4.07E-07	1.00000	0.3
		100	8.22E-07	0.99999	0.4
		150	8.63E-07	0.99999	0.7
		200	4.03E-07	1.00000	1.1
Two term	70	50	1.15E-05	0.99991	4.8
		100	2.84E-06	0.99997	0.7
		150	5.95E-06	0.99994	1.0
		200	2.31E-06	0.99997	0.9
	90	50	1.15E-05	0.99987	2.1
		100	1.37E-06	0.99999	0.5
		150	8.63E-07	0.99999	0.7
		200	4.96E-07	1.00000	1.2

Two term exponential	70	50	1.15E-04	0.99995	10.0
		100	1.10E-04	0.99903	4.4
		150	1.30E-04	0.99974	7.2
		200	3.42E-04	0.99876	12.9
	90	50	1.38E-04	0.99935	3.7
		100	5.32E-04	0.99920	11.2
		150	7.52E-05	0.99935	3.1
		200	4.83E-04	0.99917	19.0
Wang and Singh	70	50	1.60E-03	0.99235	38.7
		100	1.23E-03	0.99270	18.4
		150	1.99E-03	0.99091	22.7
		200	2.94E-03	0.98678	31.0
	90	50	1.05E-03	0.99390	23.7
		100	4.54E-04	0.99680	16.1
		150	1.12E-03	0.99341	21.5
		200	1.19E-03	0.99288	42.1
Verma	70	50	1.13E-05	0.99991	4.6
		100	9.40E-05	0.99924	5.4
		150	6.64E-06	0.99993	1.2
		200	5.64E-06	0.99994	1.4
	90	50	1.62E-05	0.99985	3.6
		100	2.53E-06	0.99998	1.4
		150	3.28E-05	0.99968	4.2
		200	1.42E-06	0.99999	0.8
Midilli-Kucuk	70	50	1.09E-05	0.99991	4.2
		100	1.12E-05	0.99990	1.4
		150	1.89E-05	0.99981	2.0
		200	8.33E-06	0.99991	2.0
	90	50	5.10E-06	0.99995	1.2
		100	3.43E-06	0.99997	1.3
		150	1.03E-05	0.99988	1.7
		200	6.33E-06	0.99993	2.3
Fick's diffusion	70	50	2.55E-03	0.99768	33.2
		100	2.43E-03	0.99488	13.0
		150	1.26E-03	0.99682	9.7
		200	1.41E-03	0.98751	16.6
	90	50	1.87E-03	0.99625	20.0
		100	3.30E-03	0.99609	27.4
		150	1.59E-03	0.99628	15.8

Table 3 shows the constants for the four best drying models, together with the values of the effective diffusivity obtained with the solution to Fick's equation (Eq. 2). AKGUN and DOYMAZ (2005), DOYMAZ (2006) and DOYMAZ (2009) calculated the effective diffusivities for the simplified Fick's equation for drying olive cake. Note that they calculated D_{eff} using the traditional method of computing the slope of ln (MR) versus time by linear regression, while we obtained D_{eff} by non-linear regression. Our results for D_{eff} were in the range of 0.78-0.93 x 10^{10} m²·s¹ at 70°C and 1.55-1.86 x 10^{10} m²·s¹ at 90 °C, which is lower than the results for olive cake at the same temperatures (6.252 x 10^{9} m²·s¹ and 7.887 x 10^{9} m²·s¹, respectively) or spinach leaves at 70^{9} C (1.5×10^{9} m²·s¹).

Table 3: Constants of selected drying models.

Model	Temperature	Re				
Logarithmic			а	k	С	
	70	50	1.0527	0.0012	0.0000	
		100	1.0238	0.0011	0.0311	
		150	0.9638	0.0011	0.0252	
		200	0.9676	0.0013	0.0417	
	90	50	1.0948	0.0023	0.0130	
		100	1.1547	0.0021	0.0037	
		150	1.0677	0.0022	0.0198	
		200	1.1786	0.0025	0.0000	
Two term			а	k_0	b	<i>k</i> ₁
	70	50	0.0059	0.0012	1.0468	0.0012
		100	0.4423	0.0018	0.6488	0.0008
		150	0.4598	0.0016	0.5523	0.0007
		200	0.9275	0.0014	0.0915	0.0002
	90	50	0.0072	0.0003	1.0666	0.0021
		100	0.0068	0.0005	1.1454	0.0021
		150	0.0198	0.0000	1.0678	0.0022
		200	0.0051	0.0016	1.1737	0.0025
Verma			а	k	g	
	70	50	1.0587	0.0012	0.0089	
		100	1.0186	0.0010	0.0380	
		150	0.3303	0.0006	0.0013	
		200	0.0399	0.0000	0.0013	
	90	50	1.0747	0.0021	0.0276	
		100	1.1576	0.0021	0.0135	
		150	1.0426	0.0020	0.0283	
		200	1.2324	0.0026	0.0090	
Midilli-Kucuk			а	k	n	b
	70	50	1.0229	0.00087	1.0390	5.62 x 10 ⁻⁷
		100	1.0871	0.00167	0.9352	3.73 x 10 ⁻⁶
		150	0.9729	0.00093	1.0107	6.06 x 10 ⁻⁶
		200	1.0076	0.00135	0.9838	1.12 x 10 ⁻⁵
	90	50	0.9820	0.00087	1.1307	1.22 x 10 ⁻⁵
		100	1.0437	0.00087	1.1274	9.03 x 10 ⁻⁶
		150	0.9701	0.00089	1.1232	1.55 x 10 ⁻⁵
		200	1.0173	0.00089	1.1472	4.28 x 10 ⁻⁶
Fick's diffusion			$D (m^2/s)$			
	70	50	0.92 x 10 ⁻¹⁰			
		100	0.78 x 10 ⁻¹⁰			
		150	0.82 x 10 ⁻¹⁰			
		200	0.93 x 10 ⁻¹⁰			
	90	50	1.66 x 10 ⁻¹⁰			
		100	1.55 x 10 ⁻¹⁰			
		150	1.61 x 10 ⁻¹⁰			
		200	1.86 x 10 ⁻¹⁰			

The model constants were regressed against the drying air temperature and flow rate to determine the influence of these variables (Table 4). The model constants that gave non-

significant regressions (p > 0.05) were omitted from the table. Note that the flow rate variable was non-significant for all model constants and does not appear in the equations.

Table 4: Influence of drying temperature on model constants. T: absolute temperature (K).

Model	Constant	Regression equation	R ²
Logarithmic	а	-1.044+0.006T	0.699
	k	-1.240+5.64·10 ⁻⁵ T	0.969
Two term	а	7.688-0.022T	0.722
	b	-8.023+0.026T	0.722
	<i>k</i> ₁	-0.025+7.585·10 ⁻⁵ T	0.907
Verma	k	-0.025+7.565·10 ⁻⁵ T	0.853
Midilli-Kucuk	n	-1.403+0.007T	0.865
Fick's diffusion	D	-8.985·10 ⁻¹⁰ +4.044·10 ⁻¹² T	0.959

RADHIKA *et al.* (2011) developed the relation equations between the constants of the logarithmic model and the drying temperature for the drying of finger millet. Their results were in agreement with ours for a and k constants. However, we did not find a significant relationship between c and drying temperature. AKPINAR (2006) obtained regression equations for the four Midilli-Kucuk model constants and found a significant influence of both air temperature and air-flow rate. In our results, only the n constant depended significantly on temperature.

Figure 3 shows the variation with time of the experimental moisture ratio for 70 and 90°C, together with the prediction lines obtained using the logarithmic, two-term and Midilli-Kucuk models.

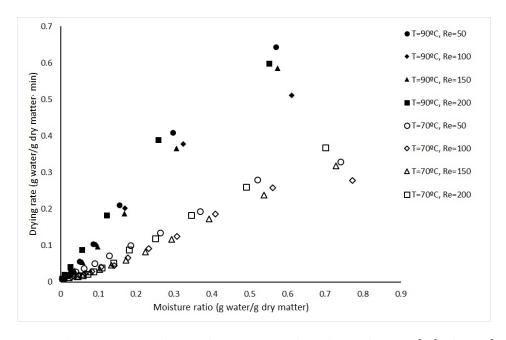


Figure 3: Experimental moisture ratio during drying time and prediction lines with the logarithmic model at 70°C (a) and 90°C (b).

It can be seen that the three model lines overlap almost completely; therefore, the three models describe the data equally well. Although a small influence of the variable flow rate on MR variation may be apparent from Fig. 3, the effect was not statistically significant. The drying time needed to arrive at MR values below 0.05 at 90°C was about one half the time needed at 70°C.

4. CONCLUSIONS

Drying rate curves for saffron floral bio-residues did not show a constant-rate drying period, and all the drying occurred during the falling-rate drying period. Moisture diffusivity obtained using the theoretical Fick's diffusion model was in the range of 0.78-1.86 x 10¹⁰ m² s¹ at 70-90 °C. The logarithmic, two-term and Midilli-Kucuk models were the best thin-layer model to fit the drying data, but other models, like the Verma model, gave also good agreement to the experimental data. The model constants were independent of air-flow rate. Regression equations were obtained to describe the influence of temperature on model constants. Increasing the temperature from 70 to 90 °C halved the drying time.

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