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Evaluation and Characterization of Vacuum Drying of Date Paste

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ABSTRACT

The drying behavior and characteristics of date paste were studied at temperatures of 60, 70, and 80ºC as thin layer with sample thicknesses of 1, 1.5, and 2 cm in a laboratory scale vacuum chamber. Modeling of drying kinetics of date paste was investigated based on the specific temperatures and sample thicknesses. The experimental moisture loss data were fitted to eight thin layer drying models available in the literature. The modified Henderson-Pabis, Verma, and Jena-Das models showed better fitness to the experimental drying data compared to the other models. The effective moisture diffusivity ranged between 6.0854×10^{-8} and 4.868×10^{-7} m^2 s^{-1}. Effective diffusivity increased with the increase in temperature and sample thickness. The temperature dependence of effective moisture diffusivity was expressed by an Arrhenius type equation.

Keywords: Date paste, Drying characteristics, Drying models, Effective moisture diffusivity, Vacuum drying.

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INTRODUCTION

Date fruit is an important commercial crop in the Middle East and Arab countries. Botanically, date fruit is a berry consisting of a single seed surrounded by a fibrous parchment like endocarp, fleshy mesocarp, and the fruit skin (pericarp). The production of date fruit in the world is estimated at 6.7 million tons. Dehydration of dates will ensure appropriate level of moisture in dates for better preservation and the product quality (Falade and Abbo, 2007). The objectives of drying process research in the food industry may be classified in three groups as follows: (a) economic considerations, (b) environmental concerns, and (c) product quality aspects. Though the primary objective of food drying is preservation, depending on the drying mechanisms, the raw material may end up a completely different material with significant variations in product quality (Gunhan *et al*., 2005). Dehydrated dates are used in food products like sweetmeats, snacks, confectionary, bakery products and health foods (Kulkarni *et al*., 2008).

In vacuum drying, since the removal of moisture takes place in the absence of oxygen, oxidative degradations, e.g. oxidation of fat and browning reactions, are minimized. In case of vacuum drying, drying temperature and sample thickness before drying influence the drying rate of samples and, hence, detailed investigations on the effect of these parameters need to be carried out (Jena and Das, 2007).

The drying kinetics are often used to describe the combined macroscopic and microscopic mechanisms of heat and mass transfer during drying and it is affected by drying conditions, type of dryer, and characteristics of dried materials (Giri and Prasad, 2007).

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One of the important aspects of drying technology is mathematical modeling of the drying processes and equipment. Its purpose is to allow design engineers to choose the most suitable operating conditions and size for the drying equipment and drying chambers accordingly to meet the desired operating conditions (Gunhan *et al*., 2005).

Thin-layer drying models are categorized as theoretical, semi-theoretical, and empirical. Theoretical models are developed from assumptions about the phenomena and are validated with experimental data. For drying process, these models are based on the mechanisms of heat and mass transfer. The most widely used theoretical models have been diffusion models derived from the Fick's second law. For theoretical thin layer models, internal resistance to moisture transfer is taken into account, while semi-theoretical and empirical models consider external resistance to moisture transfer between the product and the drying environment. Properties of the sample such as thickness, shape, and particle size, as well as drying air temperature and relative humidity are effective on moisture transfer phenomena (Akgun and Doymaz, 2005; Doymaz, 2005; Lertworasirikul, 2008).

In the present study, the focus was on the modeling of vacuum drying of date paste. Different semi-theoretical and empirical models were fitted to simulate the drying rate of date paste. Effect of sample thickness and drying temperature on the drying rate was investigated.

MATERIALS AND METHODS

Material

Dates of Kabkab cultivar were obtained from the Date Research Center (affiliated to the Ministry of Agriculture, Tehran, Iran). Fresh dates were packed in polyethylene bags and stored at –18°C. Before each experiment, dates were taken out from the freezer and, after stabilization at ambient temperature, they were pitted manually and ground with a meat grinder (Kenwood, Pro 1600, UK) to prepare date paste. The average moisture content of the date paste samples was 95.7%. i.e. 22.4 kg water kg^{-1} dry solid

Vacuum Chamber

A laboratory scale vacuum dryer (Vision, VS-1202V5, South Korea) was used for carrying out the drying experiments. The dryer consisted of a heating vacuum chamber with an electrical heater and a temperature controller, an oil vacuum pump (Motor Division, C55JXHRL-4205, USA), and a moisture trap. The heating plate temperature was set at 60, 70, and 80°C. An oil vacuum pump was used to create the necessary vacuum inside the drying chamber. The moisture trap was a Pyrex tube containing an absorbing material (P_2O_5) to absorb moisture of the exhausted air from the heating chamber and prevent the moisture from entering the pump.

Experimental Set up

The date paste was dried at temperatures of 60, 70, and 80ºC and sample thicknesses of 1, 1.5, and 2 cm. Drying process started when steady-state condition was achieved in the dryer. The date paste was placed uniformly in Pyrex plates (10 cm in diameter). The samples were spread on the plates by a ruler and accurately adjusted to the aforementioned thicknesses in all directions. Then, the plates were kept inside the drying chamber at an absolute pressure of 20 kPa.

During the drying process, the vacuum was broken and the samples were taken out and weighed at hourly intervals using a digital electronic balance (Sartorius, TE214S, Germany). The accuracy of the weighing system was 0.0001 g. Weight measurements took one minute and, after putting the sample in the drying chamber, the vacuum was restored. Moisture content of the samples was calculated as (kg water kg dry solid^{-1}). Drying was continued until three consecutive measurements showed constant weights. Three replicates for each of the experiments were done. At the end of each experiment, the moisture ratio at any time was calculated and the drying data was expressed as moisture ratio versus drying time and, also, drying rates at various drying times were calculated and drying rate curves were plotted.

Modeling of Vacuum Drying

Modeling of the drying behavior is of great importance for investigation of the drying characteristics of date paste. In this study, the experimental vacuum drying data of the date paste at different temperatures and thicknesses were fitted to some commonly used thin layer drying models (Table 1). In these models, MR represents the dimensionless moisture ratio:

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

$$\text{Drying rate} = \frac{M_t - M_{t+dt}}{dt} \quad (2)$$

Where, M and M_0 are the moisture content at any given time and the initial moisture content (kg water kg^{-1} dry solid), respectively, M_e is the equilibrium moisture content (kg water kg^{-1} dry solid), M_t and M_{t+dt} are moisture content at time t and moisture content at $t+dt$ (kg water kg^{-1} dry solid), and t is the drying time. In Table 1, k, n, a, b, c, g, and h are model constants.

Since the temperatures used in this study were higher than the saturation temperature of water, pure water will boil inside the dryer. It was, therefore, possible to completely dehydrate the material and the equilibrium moisture content of the dried material was considered to be zero. Thus the dimensionless moisture ratio, MR, can be simplified to $MR= M / M_0$ (Wang *et al.*, 2007; Jena and Das, 2007).

Sigmaplot 2000 version 6.0 software was used to calculate the constants and coefficients of the selected models. The goodness of fit of the tested mathematical models to the experimental data was evaluated using the coefficient of determination (R^2), the reduced chi-square (χ^2), and the root mean square error (RMSE). The higher the R^2 values and the lower the χ^2 and *RMSE* values, the better are the goodness of fit (Togrul and Pehlivan, 2002). These parameters can be calculated by the following equations:

$$\chi^2 = \frac{\sum_{i=1}^{N} (MR_{\exp,i} - MR_{pre,i})^2}{N - n} \quad (3)$$

$$\text{RMSE} = \sqrt{\frac{1}{N}\sum_{i=1}^{N} (MR_{pre,i} - MR_{\exp,i})^2} \quad (4)$$

Where $MR_{exp,i}$ and $MR_{pre,i}$ are the experimental and predicted moisture ratios, respectively, and N and n are the number of observations and constants, respectively (Menges and Ertkin, 2006).

Determination of Effective Moisture Diffusivity

The experimental drying data for the

Table 1. Thin layer drying mathematical models used for drying of date paste.

Model No	Model name	Model	References
1	Lewis	$MR= exp(-kt)$	Lewis, 1921
2	Page	$MR= exp(-kt^n)$	Page, 1949
3	Modified Henderson-Pabis	$MR= a\ exp(-kt)+b\ exp(-gt)+c\ exp(-ht)$	Karathanos, 1999
4	Wang and Sing	$MR= 1+at+bt^2$	Wang and Singh, 1978
5	Midilli	$MR= a\ exp(-kt^n)+bt$	Midilli *et al.*, 2002
6	Verma	$MR= a\ exp(-kt)+(1-a)\ exp(-gt)$	Verma *et al.* 1985
7	Two-Term exponential	$MR= a\ exp(-kt)+(1-a)\ exp(-kat)$	Glenn, 1978
8	Jena-Das proposed model	$MR= a\ exp(-kt+b\sqrt{t})+c$	Jena and Das, 2007

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determination of moisture diffusivity were interpreted by Fick's second law of diffusion. The solution to this law, developed by Crank (1975), can be used for various regularly shaped bodies such as slab, cylindrical and spherical products. An analytical solution to this law in the case of drying an infinite slab of thin layer, assuming one dimensional moisture movement without volume change, constant diffusivity, uniform initial moisture distribution, and negligible external resistance, can be developed in the form of the following equation (Jena and Das, 2007; Nguyen and Price, 2007):

$$MR=\frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\exp\left(-\frac{(2n+1)^2\pi^2 D_{eff}t}{4l^2}\right) \quad (5)$$

Where, MR is the moisture ratio, D_{eff} ($m^2 s^{-1}$) is the effective moisture diffusivity, l (m) is the sample thickness of slab, t (s) is the drying time, and n is a positive integer.

At sufficiently large times, only the first term in the series expansion needs to be taken into account (Heldman and Lund, 2007). Equation (6) is obtained by taking the natural logarithm of both sides of the Equation (5). This shows that the time to reach the given moisture content will be directly proportional to the square of the thickness of slab and inversely proportional to D_{eff}. Effective moisture diffusion coefficient (D_{eff}) is determined by plotting ln MR as a function of time from the integrated form of Fick's equation:

$$\ln MR = \ln\frac{8}{\pi^2}-\frac{\pi^2 D_{eff}t}{4l^2} \quad (6)$$

The diffusion coefficient is also typically calculated by using the slope of the Equation (6), when natural logarithm of MR versus time is plotted, and, thus, the plot gives a straight line with a slope of:

$$\text{Slope} = \frac{\pi^2 D_{eff}}{4l^2} \quad (7)$$

And D_{eff} for all conditions of the experiment is calculated. The temperature dependence of the moisture diffusivity is described with an Arrhenius type equation:

$$D_{eff} = D_0 \exp\left(\frac{-E_a}{RT}\right) \quad (8)$$

Where, D_0 is the pre-exponential factor of the Arrhenius equation ($m^2 s^{-1}$), E_a is the activation energy of the moisture diffusion ($kJ\ mol^{-1}$), T is the air absolute temperature (K), and R is the universal gas constant ($8.3143\ kJ\ kmol^{-1}\ K^{-1}$).

Taking natural logarithm, Equation (8) can be linearized as follows:

$$\ln(D_{eff}) = \ln(D_0) - \frac{E_a}{R}\frac{1}{T} \quad (9)$$

The activation energy is calculated from the slope of the straight line ($-E_a/R$).

RESULTS AND DISCUSSION

Drying Characteristics

The variation in the moisture ratio as a function of the drying time for all tests has been shown in Figure 1. It is observed from these plots that moisture content decreased continuously with drying time. The increase in the drying temperature and the decrease in sample thickness resulted in a decrease in drying time. The drying times required to obtain three consecutive constant weights for all the nine tests were about 23-58 hours. The decrease in drying time with an increase in the drying air temperature have been reported for many food stuffs such as apricot (Togrul and Pehlivan, 2003), eggplant (Ertekin and Yaldiz, 2004; Wu *et al*., 2007), olive cake (Akgun and Doymaz, 2005), apple pomace (Wang *et al.*, 2007), pumpkin slices (Doymaz, 2007), and onion slices (Jain and Pathare, 2004; Kumar *et al*., 2006; Sarsavadia *et al*., 1999; Sharma *et al*., 2005). As shown in Figure 1, at higher temperatures (80°C), the remaining moisture content in the date paste, especially in the minimum thickness (1 cm), is higher compared to the lower temperatures. The reason may be the fact that, when drying is performed at a high temperature, a surface hardening effect occurs faster for thin slabs rather than thicker ones. Surface hardening

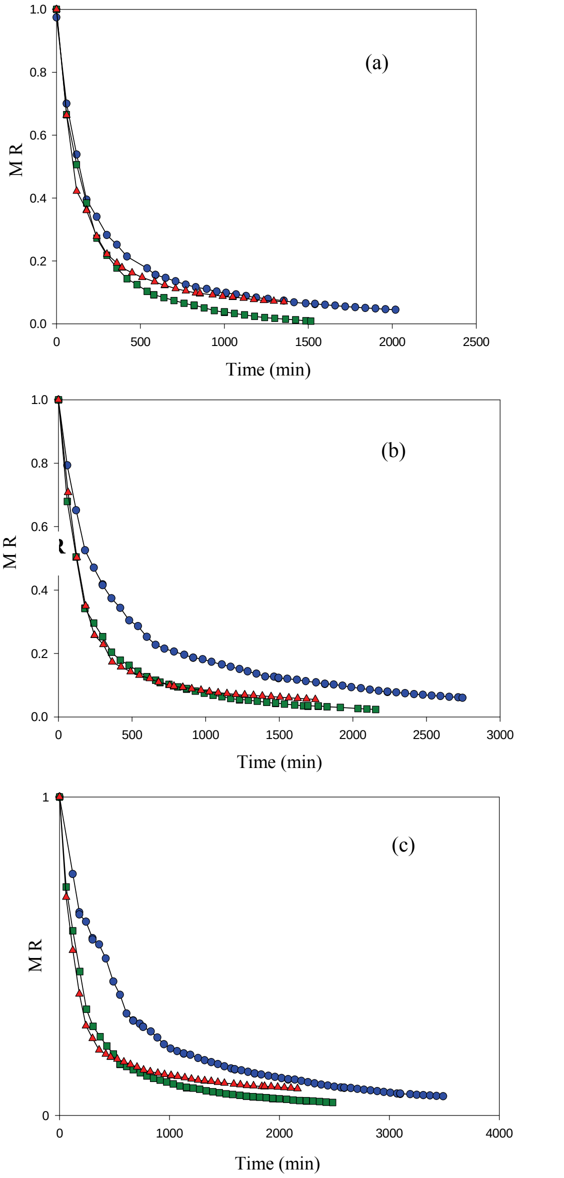

Figure 1. Vacuum drying curves of date paste at different vacuum chamber plate temperatures and constant sample thicknesses of (a) 1 cm, (b) 1.5 cm and (c) 2 cm: ● (T=60 ºC), ■ (T=70 ºC), ▲ (T= 80ºC).

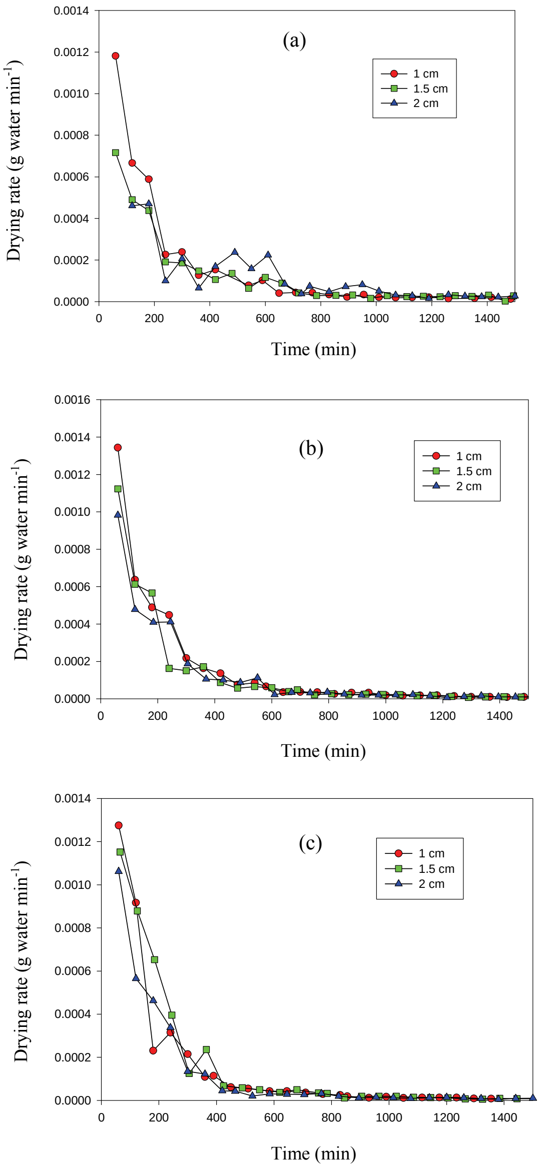

Figure 2. Drying rate of date paste undergoing vacuum drying for different sample thicknesses at 60ºC(a), 70 ºC(b) and 80 ºC(c).

at high temperatures may be due to high sugar content.

Drying Rate

Drying rates of date paste were calculated using Equation (2). The changes in the drying rate versus time for the various thicknesses and temperatures are shown in Figure 2.

It is apparent that the drying rate decreases continuously with drying time. It is also noted that the drying rate increased with decrease in sample thickness. Date paste did not exhibit a constant drying rate period and all the drying operations occurred in the falling rate period. This is due to the removal of moisture from the surface and shows that the diffusion is the dominant drying phenomena. The results obtained showed that the internal mass transfer resistance, due to the presence of a falling rate drying period, controls the drying time. At the beginning of the drying process, the drying rate was very high but decreased as the drying time increased. Similar results have been presented for aromatic plants (Belghit *et al*., 2000), single apricots (Togrul and Pehlivan, 2003), mushrooms (Giri and Prasad, 2007), red chillies (Kaleemullah and Kailappan, 2007), red peppers (Akpinar *et al.*, 2003), and onion slices (Pathare and Sharma, 2006; Sarsavadia *et al*., 1999). In drying rate curves of date paste, more than one break appeared in the falling rate period due to the inherent properties of hygroscopic materials.

Drying Models

The moisture ratio from all nine experiments were calculated and fitted to the selected thin layer drying models listed in Table 1. The statistical results of the different models, including the comparison criteria used to evaluate goodness of fit such as the values of coefficient of determination (R^2), reduced chi-square (χ^2), and root mean square error (RMSE), and also the values of model constants for various samples with thickness of 1 cm are presented in Table 2. Similar results were also observed for other sample thicknesses.

It can be deduced that Wang-Singh model has the least fitness as the coefficient of determination and reduced chi-square of this model ranged between 0-0.5994 and 0.0148-0.0311 for all the experiments, respectively. The Lewis, Page, Midilli, and Two-term exponential models predicted the drying data relatively well, specially at 70ºC. Modified Henderson-Pabis, Verma, and Jena-Das models seemed to have better fitness in all cases with the R^2 values more than 0.99. The values of R^2, χ^2 and *RMSE* for these three models ranged from 0.9912–0.9995, 2.4372×10^{-05}–0.00694, and 0.00464324–0.078883, respectively. In Figure 3, the prediction values of the best models are compared with our experimental data for 1.5-cm samples dried at 70 ºC. Similar results were obtained in other experiments.

As it can be seen, Jena-Das, Verma, and modified Henderson-Pabis models provide very good fitness between experimental and predicted moisture ratios. Figure 4 indicates the comparison of the predicted and the experimental moisture ratio values for 1-cm sample thickness at 70ºC.

It can be seen that there was very good agreement between the experimental and predicted MR values, which are closely banding around at a 45º straight line. Accordingly, this indicates the suitability of the Jena-Das, Verma, and modified Henderson-Pabis models in describing drying behavior of date paste.

Effective Moisture Diffusivity

Applying Equation (6) and by plotting the natural logarithm of moisture ratio (ln MR) versus drying time, effective diffusivity, D_{eff}, could be determined from the slope of the straight line. The values of effective diffusivity at different drying temperatures and thicknesses are presented in Table 3.

Effective moisture diffusivity values varied between 6.085×10^{-8} and 4.868×10^{-7} $m^2\ s^{-1}$ for the entire experimental range. These values are comparable to the diffusivity values of 7.026×10^{-10} - 3.326×10^{-9} $m^2\ s^{-1}$ for the vacuum drying of coconut presscake in the temperature range of 65-75°C (Jena and Das, 2007). It can be seen that the values of D_{eff} increased with the increase in the sample thickness and drying temperature. Even though, it has been assumed in the diffusion model that the thickness and moisture diffusivity remains constant; however, in reality, thickness changes due to shrinkage and moisture diffusivity reduces with the reduction in

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Table 2. Values of model constants and statistical results for sample thickness of 1 cm.

Model No.	Temperature (ºC)	R^2	χ^2	*RMSE*	Constants						
					k	n	a	b	c	g	h
1	60	0.9279	0.0041	0.0632	0.0037	_	_	_	_	_	_
	70	0.9783	0.0011	0.0322	0.0049	_	_	_	_	_	_
	80	0.8764	0.0051	0.0704	0.0048	_	_	_	_	_	_
2	60	0.9929	0.0004	0.0196	0.0419	0.5838	_	_	_	_	_
	70	0.9977	0.0001	0.0102	0.0196	0.7528	_	_	_	_	_
	80	0.9841	0.0006	0.0247	0.0560	0.5591	_	_	_	_	_
3	60	0.9995	2.970×10^{-05}	0.0050	0.0007	_	0.1811	0.3525	0.4536	0.0038	0.0101
	70	0.9993	3.327×10^{-05}	0.0052	0.0019	_	0.2416	0.0693	0.6891	0.7382	0.0069
	80	0.9978	9.124×10^{-05}	0.0085	0.0006	_	0.1511	0.4394	0.4116	0.0051	0.0138
4	60	0.5958	0.0230	0.1475	_	_	-0.0016	0.0000	_	_	_
	70	0.5994	0.0198	0.1360	_	_	-0.0021	0.0000	_	_	_
	80	0.4638	0.0222	0.1439	_	_	-0.0023	0.0000	_	_	_
5	60	0.9969	0.0002	0.0125	0.0255	0.6764	0.9925	0.0000	_	_	_
	70	0.9982	8.683×10^{-05}	0.0087	0.0172	0.7796	1.0034	0.0000	_	_	_
	80	0.9948	0.0002	0.0136	0.0282	0.6983	1.0069	0.0001	_	_	_
6	60	0.9989	6.161×10^{-05}	0.0074	0.0009	_	0.2656	_	_	0.0076	_
	70	0.9989	5.173×10^{-05}	0.0068	0.0022	_	0.3248	_	_	0.0085	_
	80	0.9974	0.0069	0.0789	0.0091	_	0.7696	_	_	0.0010	_
7	60	0.9573	0.0024	0.0479	0.0101	_	0.2712	_	_	_	_
	70	0.9932	0.0003	0.0177	0.0129	_	0.2900	_	_	_	_
	80	0.9225	0.0032	0.0547	0.0126	_	0.2800	_	_	_	_
8	60	0.9968	0.0002	0.0128	0.0022	_	0.9331	-0.0388	0.0587	_	_
	70	0.9979	0.0001	0.0094	0.0034	_	0.9823	-0.0295	0.0206	_	_
	80	0.9955	0.0002	0.0126	0.0039	_	0.9222	-0.0380	0.0824	_	_

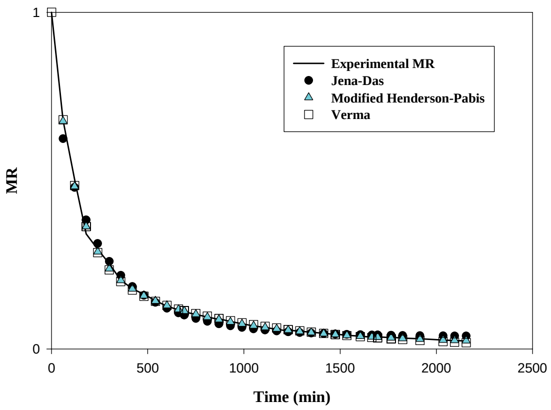

Figure 3. Comparison of drying curves of date paste undergoing vacuum drying for sample thickness of 1.5 cm at 70ºC for Jena-Das, Verma, and modified Henderson-Pabis models.

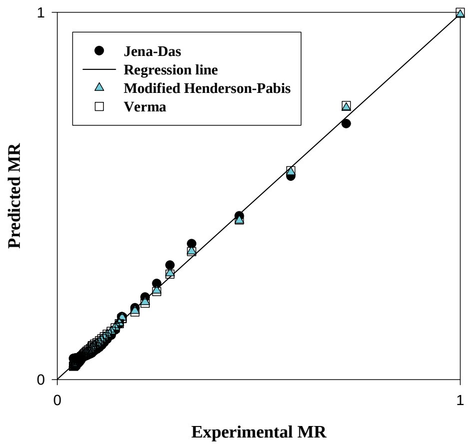

Figure 4. Comparison of experimental and predicted moisture ratio by the Jena-Das, Verma, and modified Henderson-Pabis models for 1 cm sample thickness at 70ºC.

Table 3. Values of effective moisture diffusivity of date paste during the vacuum drying.

Initial thickness of date paste (m)	Vacuum chamber temperature (°C)	Effective moisture diffusivity ($m^2 s^{-1}$)
0.010	60	6.085×10^{-8}
0.010	70	1.055×10^{-7}
0.010	80	1.339×10^{-7}
0.015	60	1.095×10^{-7}
0.015	70	1.826×10^{-7}
0.015	80	3.377×10^{-7}
0.020	60	2.434×10^{-7}
0.020	70	2.921×10^{-7}
0.020	80	4.868×10^{-7}

moisture content and temperature. When drying was performed at a high temperature, a surface hardening effect occurred in the thin slabs faster than in the thick slabs, due to quicker initial rate of moisture evaporation from the surface. This hardening effect slowed down the drying rate in the thin slabs. This, in turn, made the difference between the drying rate of 1-cm and 2-cm slabs decrease faster at the higher drying temperatures than at the lower ones. This effect also could be helpful to explain why the diffusion coefficients in 1-cm slabs were smaller than in 2-cm slabs. Edge effects might enhance the removal of moisture from thick slabs. Further, the hardening effect might hinder the transfer of moisture in thin slabs after drying for some hours. Both of these reasons would explain why the values of *D* for the thick slabs were higher than those of the thin slabs. These findings confirm the findings of Nguyen and Price (2007).

Activation Energy

The temperature dependence of D_{eff} can be described by Arrhenius type equation as given in Equation (8). The activation energy was calculated from the slope of the plot of ln (D_{eff}) versus $1/T$ for the slabs of 1, 1.5 and 2 cm (Figure 5).

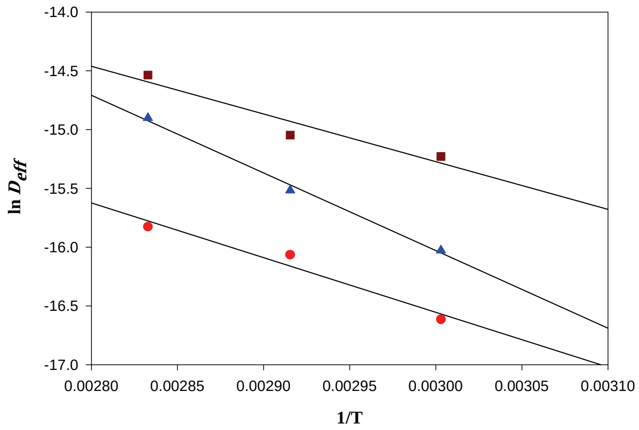

Figure 5. Arrhenius type relationship between the effective diffusivity (D_{eff}) and *1/T* for thicknesses of 1 cm (●), 1.5 cm (▲), and 2 cm (■).

The final values of E_a was found to be 38.67, 54.96, and 33.71 kJ mol^{-1}, for 1, 1.5 and 2 cm slab thicknesses, respectively. Equation (9) is calculated for the thicknesses of 1, 1.5, and 2 cm as Equations (10-12), respectively (Figure 5).

$$\ln(D_{eff}) = -2.6023 + 4650.6\frac{1}{T} \quad R^2 = 0.96 \qquad (10)$$

$$\ln(D_{eff}) = 3.8014 + 6610.2\frac{1}{T} \quad R^2 = 0.99 \qquad (11)$$

$$\ln(D_{eff}) = -3.1107 + 4054\frac{1}{T} \quad R^2 = 0.92 \qquad (12)$$

Equations (10-12) can be used for calculating D_{eff} based on temperature and thickness in designing vacuum dryers for date paste.

CONCLUSIONS

Drying kinetics of date paste was investigated in a vacuum dryer at various vacuum chamber temperatures and sample thicknesses. The increase in temperature and decrease in sample thickness significantly reduced the drying time. Drying curves of date paste did not show a constant drying rate period in this experiment and showed only the falling rate period. The moisture content and drying rate was influenced by the drying temperature and sample thickness. The modified Henderson-Pabis, Verma, and Jena-Das models provided the best representation of date paste drying kinetics and fitted better to the drying curves than the other models. Effective moisture diffusivity was calculated based on Fick's second law of diffusion, the values of which varied between 6.085×10^{-8} and 4.868×10^{-7} m^2 s^{-1}. Effective moisture diffusivity was found to increase with increase in both the sample thickness and drying temperature. The temperature dependence of the effective moisture diffusivity was described by an Arrhenius type equation. The activation energy was found to be 38.67, 54.96, and 33.71 kJ mol^{-1}, for 1, 1.5 and 2 cm slab thicknesses, respectively.

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بررسی و توصیف خشک کردن تحت خلا خمیر خرما

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چکیده

رفتار و توصیف خشک کردن خمیر خرما در دماهای ۶۰، ۷۰ و ۸۰°C بصورت نمونه های لایه نازک با ضخامت ۱، ۱/۵ و ۲ سانتی متری در محفظه تحت خلا آزمایشگاهی ارزیابی شد. مدلسازی سینتیک خشک کردن خمیر خرما در دماها و ضخامت های مختلف بررسی شد. تطابق داده های خشک کردن با هشت مدل بکار گرفته شده در مطالعات خشک کردن لایه های نازک انجام شد. مدلهای جنا-دس، هندرسون پابیس و ورما تطابق بهتری با داده های خشک کردن تجربی نسبت به سایر مدلها نشان می دهند. تغییرات نفوذ رطوبت موثر بین $۶/۴۴۵۸۰ \times ۱۰^{-۸}$ و $۴/۸۶۸۳۵ \times ۱۰^{-۷}$ است. نفوذ موثربا افزایش دما و ضخامت نمونه افزایش می یابد. وابستگی نفوذ رطوبت موثر به دما بوسطه رابطه آرنیوس بیان شد.

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