Comparative kinetic analysis of convective and vacuum dried *Opuntia ficus-indica* (L.) Mill. cladodes

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Abstract: *Opuntia ficus-indica* (Linnaeus) Miller more usually known as fodder palm or nopal belongs to family Cactaceae. In the present study, the drying behavior of the *O. ficus-indica* cladodes was observed. The study concentrates on comparatively studying two types of commercial drying methods viz., forced convective drying (tray drying) and vacuum drying to dry nopal cladodes at three different temperatures viz. 40, 50 and 60°C. The equilibrium moisture contents for forced convective drying was achieved at 540–720 min and for that of vacuum drying at 600–840 min. Three mathematical drying models for thin layer drying viz. Page, Lewis and Henderson-Pabis model were evaluated for both convective drying and vacuum drying. Statistical parameters such as the coefficient of determination (R^2), root mean square error and reduced χ^2 were used to fit the models. Page model was found to be satisfactory for both forced convective and vacuum drying of the nopal cladodes at 40 and 50°C respectively. Among these, two drying methods, forced convective drying method was found to be more suitable than the vacuum drying method for nopal cladodes on the basis of drying time and statistical parameters.

Keywords: nopal cladodes; forced convective drying; vacuum drying; mathematical models; Page model

Climate change is the most devastating reality, threatening the human kind in recent years. The most prominent effect of climate change and global warming in recent future will be on agriculture and food systems (Brown, Funk 2008). Increase in temperature and a decline in precipitation over semiarid regions will reduce the yield of various agronomic crops such as wheat, rice and corn, resulting in food insecurity. Owing to this fact the rescue could come with some crops we may think as unlikely solutions such as prickly cactus (*Opuntia ficus*-indica (Linnaeus) Miller).

O. ficus-indica more commonly called as fodder palm or nopal belongs to family Cactaceae.

Originating in Mexico, this plant has now made its home around the globe (VIANA et al. 2014). It has been consumed in the Mexican diet since prehistoric times (Contreras-Padilla et al. 2012). Nopal is a highly adaptable plant and grows in variable climatic conditions, including arid and semi-arid regions. Its fruits, cladodes and seeds are considered as a rich source of nutrients in such areas. It contains a good amount of nutrients such as fibers, proteins, phenolic compounds flavonoids and its derivatives (Kossori et al. 1998). The carotenoid levels of cactus are higher than that of most of the vegetables (Betancourt-Domínguez et al. 2006). Young cladodes are generally regard-

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ed as consumable as fiber content increases with maturity (Contreras-Padilla et al. 2012). In addition to nutrition, nopal are also now studied for their clinical and nutraceutical properties against numerous diseases and disorders such as diuretic, antiinflamatory, analgesic, antiulcerous, hyperglycemia, gastritis, arteriosclerosis, diabetes and prostatic hypertrophy (STINTZING et al. 2005; Ennouri et al. 2006; Feugang et al. 2006; Gala-TI et al. 2007). Food industries have now found good uses for this underutilized commodity, owing to numerous physical viz. rheological (CORNEJO-VILLEGAS et al. 2010), chemical (CONTRERAS-PADILLA et al. 2012) and nutritional (Kossori et al. 1998) significance it possesses. However, nopal cladodes have a high amount of water and are low in pH, thus are highly susceptible to microbial degradation (Contreras-Padilla et al. 2012). Thus, post-harvest processing of nopal cladodes is suggested to preserve it.

Drying is one such ageless low cost technology for the preservation of several agricultural commodities. It helps reduce the water content of the food to an extent where microbial spoilage becomes unlikely (MRAD et al. 2012). In addition, drying reduces the mass and volume of the product, facilitating ease of handling, transportation and storage. Different methods of drying consume a different amount of time and energy in the form of heat and have different effects on the commodity.

The present study thus concentrates on comparatively studying two types of commercial drying methods viz., forced convective drying (tray drying) and vacuum drying, to dry nopal cladodes. The drying kinetics of the nopal cladodes at variable temperature was studied to obtain the best suitable commercial drying conditions for this valuable commodity.

MATERIAL AND METHODS

The wild variety of the nopal (*O. ficus-indica*) cladodes was obtained from the Hoshiarpur region of Punjab, India. Nopal cladodes were cleaned using water to remove the dirt, extraneous matter and rubbed against the abrasive surface to remove the spines. The cladodes were cut into 2.5 cm length, 1 cm width and 0.2 cm thickness.

Drying equipment and procedure. The laboratory scale tray dryer (Labfit India Pvt. Ltd., India)

having temperature controller, fan and a five sets of trays was used for the experimentation. The trays were arranged vertically and placed horizontally in a self-contained chamber. A thin layer of 0.2 cm thickness of nopal cladodes was spread on each tray. Drying experiment was conducted at a temperature range of 40, 50 and 60°C. Samples of nopal cladodes were weighed using digital weighing balance at the interval of every 60 min. The initial moisture content of each sample was determined using the hot air oven with a fixed temperature of 105°C until the equilibrium moisture content was reached. Weight loss was calculated on the dry mass basis (GARBA et al. 2015), using Eq. 1:

$$w = \frac{m - m_{\rm d}}{m_{\rm d}} \tag{1}$$

where: w – moisture content (dry basis); m – total mass of the sample (g); $m_{\rm d}$ – mass of the dry matter (g) present in the sample

Mathematical modeling and fitting the drying models. The data obtained was interpreted using three different drying model equations (Eq. 2: Page model, Eq. 3: Lewis model, Eq. 4: Henderson-Pabis model):

$$MR = \frac{M_t - M_e}{M_o - M_e} = \exp(-kt^N)$$
 (2)

where: MR – moisture ratio; M_t – moisture content at any time t; $M_{\rm e}$ – equilibrium moisture content; $M_{\rm o}$ – initial moisture content; k – drying rate constant; N – drying constant

$$MR = \frac{M_t - M_e}{M_o - M_e} = \exp(-kt)$$
 (3)

$$MR = \frac{M_t - M_e}{M_o - M_e} = a \exp(-kt)$$
 (4)

where: a – drying constant

The data calculated using moisture ratio for thin layer drying of nopal cladodes from the three models were plotted against the drying time of different drying temperature used, and evaluated using coefficient of determination (R^2).

The best model for the thin layer drying of the nopal cladodes samples was determined based on the lowest value of the root mean square error (RMSE), χ^2 and residual sum of squares (RSS). The RMSE, χ^2 and RSS were calculated using Eqs. 5–7 (McMinn 2006; Garba et al. 2015) as given below:

RMSE =
$$\sqrt{\frac{1}{N} \sum_{i=1}^{N} (MR_{\exp(i)} - MR_{\text{pre}(i)})^2}$$
 (5)

where: N – number of observations; i – ith observation of N samples; $MR_{\rm exp}$ – experimental moisture ratio; $MR_{\rm pre}$ – predicted moisture ratio

$$\chi^2 = \frac{1}{N - n} \sum_{i=1}^{N} (MR_{\exp(i)} - MR_{\operatorname{pre}(i)})^2$$
 (6)

where: $n - n^{th}$ number of observation in the experiment

$$RSS = \sum_{i=1}^{N} (MR_{\exp(i)} - MR_{pre(i)})^{2}$$
 (7

The prediction data are closer to the experimental when RMSE and χ^2 is reduced to zero. RMSE and χ^2 represents the difference in between the predicted moisture ratio and experimental moisture ratio, while minimum RSS depicted is important in the non-linear regression process (Panchariya et al. 2002).

Effective moisture diffusivity. The data for effective moisture diffusivity of nopal cladodes was derived using Fick's second law of diffusion of different shaped products such as spherical, rectangular and cylindrical products (Tulek 2011) as shown in Eq. 8:

$$\frac{\delta M}{\delta t} = \nabla \left[D_{\text{eff}} \left(\nabla M \right) \right] \tag{8}$$

where: M – moisture content; $D_{\rm eff}$ – effective moisture diffusivity (m $^2 \cdot s^{-1}$)

For the slab geometry equation is used by assuming uniform moisture content during initiation of drying, shrinkage effect, and external resistance for moisture transfer as negligible (Khawas et al. 2014), as Eq. 9:

$$MR_{i} = \frac{3}{\pi^{2}} \sum_{n=0}^{\infty} \frac{1}{(2b+1)^{2}} exp \left(-\frac{(2n+1)^{2} \pi^{2} D_{eff} t}{4L_{o}^{2}} \right)$$
(9)

where: n – number of observations; b – positive integer; L_0 – thr half value thickness of slab sample (m)

Above mentioned Eq. 9 can be simplified during the longer period of drying (Kumar et al. 2010), as Eq. 10:

$$\ln MR = \ln \frac{8}{\pi^2} - \frac{\pi^2 D_{\text{eff}} t}{4L_0^2}$$
 (10)

Where $D_{\rm eff}$ is calculated using slope obtained from the graph in between ln MR versus time. The calculation of the slope (Eq. 11) and $D_{\rm eff}$ (Eq. 12) is as follows:

$$Slope = \frac{\pi^2 D_{\text{eff}}}{4L_o^2} \tag{11}$$

$$D_{\text{eff}} = \frac{4L_0^2 \times \text{slope}}{\left(\pi^2\right)}$$
 (12)

where: Slope = $(\ln MR_2 - \ln MR_1)/(t_2 - t_1)$; $MR_2 - \text{moisture ratio at time } t_1$

Activation energy. The moisture diffusivity describing the temperature dependence can be described using Arrhenius equation (AKPINAR et al. 2003) as given in Eq. 13:

$$D_{\text{eff}} = D_0 \exp\left[-\frac{E_a}{R(T + 273.15)}\right]$$
 (13)

where: D_0 – Arrhenius factor (m²·s⁻¹); E_a – activation energy (kJ·mol⁻¹); R – universal gas constant (8.314 J·mol⁻¹·K⁻¹); T – absolute temperature (K)

The slope obtained from the graph plotted in between $\ln D_{\rm eff}$ versus 1/T was used to calculate the activation energy (Eq. 14):

$$Slope = \frac{E_a}{R}$$
 (14)

RESULTS AND DISCUSSION

Drying behavior of nopal cladodes

The nopal cladodes had initial moisture content of 1,228.96% on dry weight basis (db). The final moisture content of nopal cladodes reached the level of 5% db when drying at 40–60°C by forced convective drying, whereas, this moisture content reached a level of 50% db when drying at 40–60°C by vacuum drying. The equilibrium moisture contents for forced convective drying reached at 540 to 720 min and for that of vacuum drying reached sat 600–840 min.

Drying time and drying rate

The drying time reduced with the increase in temperature at the rate of 10°C (Kumar et al. 2010). The drying mainly occurred in falling rate intervals (Aghbashlo et al. 2011). With the increase in the drying time a steady decrease in the percent moisture content (db) was observed. This decrease is expected as the sample is kept for a longer drying time, which leads to a greater loss of moisture from the cladodes. The drying rate increased continuous-

Table 1. Empirical	l constants of Page,	Lewis and Hende	erson-Pabis model

M-41 1	Temperature	Page model			Lewis model		Henderson-Pabis model		
Method	(°C)	$k (h^{-1})$	N	R^2	k (h ⁻¹)	R^2	k (h ⁻¹)	а	R^2
Forced convective drying	40	0.3922	0.7003	0.9892	0.8450	0.9552	0.8450	2.8213	0.9552
	50	0.3853	0.3064	0.9722	0.5990	0.9868	0.6667	1.9425	0.9527
	60	0.2370	1.6033	0.7866	0.9142	0.8661	1.2072	6.3426	0.8184
Vacuum drying	40	4.5649	0.56	0.9589	0.4844	0.8838	0.6490	5.2751	0.7471
	50	0.0248	1.6856	0.9777	0.2441	0.9136	0.2320	2.3520	0.9374
	60	0.0936	1.7963	0.9379	0.5199	0.8763	0.5199	2.0287	0.8763

k – drying rate constant; N, a – drying constant; R^2 – regression coefficient

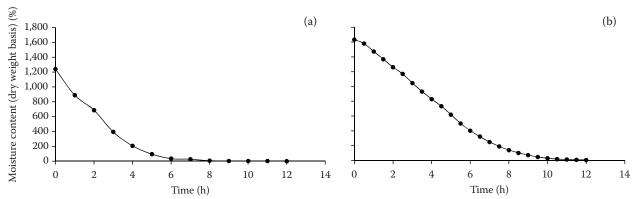


Fig. 1. Drying curve for Page model at 40°C by forced convective drying method (a), at 50°C by the vacuum drying method (b)

ly with an increase in the initial moisture content. This increase is expected as higher the moisture content present in food more rapidly the moisture will decrease and higher will be the drying rate. Higher values of rate constant were observed when the sample was dried at 40°C obtained for both forced convective and vacuum drying (Table 1). Figs 1a, b show the drying curve for Page model at 40°C by forced convective drying method and at 50°C for vacuum drying method, respectively.

observed by many researchers (AGHBASHLO et al. 2011; Revaskar et al. 2014; Garba et al. 2015). It can be observed from Table 2 that the RMSE, reduced χ^2 , PE values were minimum in case of Page model when compared to the Lewis and Henderson-Pabis model for both vacuum and forced convective dried samples. Page model at 40°C for forced convective drying method was found to be best fitted (Fig. 2a). In case of vacuum drying method, Page model was best suitable at 50°C (Fig. 2b).

Evaluation of the models

The experimental moisture content obtained from the drying experiment was first converted into moisture ratio (MR) and then incorporated to three different drying models viz. Page, Henderson-Pabis and Lewis model. The statistical tools such as RMSE, χ^2 and percentage error (PE) were employed to test the different models. Based on this statistical analysis the Page model was found to be the most suitable model for both forced convective drying method (at 40°C) and vacuum drying method (at 50°C) as evident from Table 2. Similar results are

Effective moisture diffusivity and activation energy

Fick's diffusions and Arrhenius model was employed to measure the moisture diffusivity and activation energy, respectively. The effective diffusivity ranged between 3.93×10^{-7} to 5.35×10^{-7} m²·s $^{-1}$ for forced convective drying and 2.96×10^{-7} to 3.73×10^{-7} m²·s $^{-1}$ for vacuum drying (Table 3). Similar results were reported by Doymaz and İsmail (2012) and Garba et al. (2015), where it was observed that with the increase in drying air temperature, the values of effective diffusivity also increased.

Table 2. Statistical results obtained from different thin-layer model

Method	Temperature	Page model			Henderson-Pabis model			Lewis model		
	(°C)	RMSE	$\chi^2 \times 10^{-2}$	PE	RMSE	$\chi^2 \times 10^{-2}$	PE	RMSE	$\chi^2 \times 10^{-2}$	PE
Forced	40	0.0366	0.1942	1.0827	0.1416	2.8985	0.5347	0.1515	2.9845	0.3365
convective	50	0.0389	0.2274	0.9900	0.0845	1.0710	0.4180	0.0974	1.2654	0.2848
drying	60	0.0475	0.3769	0.9888	0.3818	24.300	0.0797	0.1805	4.6559	0.4161
Vacuum	40	0.0673	0.6191	0.5994	0.5169	36.4385	0.8403	0.2016	5.0852	0.4264
Vacuum drying	50	0.0565	0.3778	1.2541	0.2317	6.3465	0.4500	0.2299	5.9774	0.4375
	60	0.0540	0.4587	0.2997	0.1283	2.5895	0.5249	0.2089	6.0037	0.4589

RMSE - root mean square error; PE - percentage error

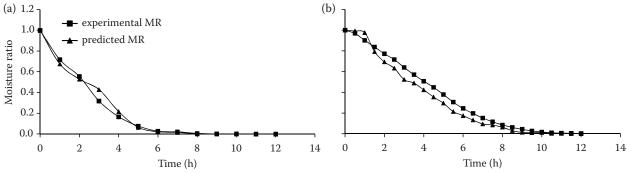


Fig. 2. Experimental and predicted moisture ratio (MR) vs. time for Page model at 40°C by forced convective drying method (a), at 50°C by vacuum drying method (b)

Table 3. Effective diffusivity of nopal cladodes

Method	Temperature (°C)	Drying time (min)	$D_{eff} (m^2 \cdot s^{-1}) \times 10^{-7}$	$E_{\rm a}$ (kJ·mol ⁻¹ ·K ⁻¹)
	40	720	3.99	
Forced convective drying	50	660	3.93	39.712
	60	540	5.35	
	40	840	2.96	
Vacuum drying	50	720	2.58	41.692
	60	600	3.73	

 $D_{
m eff}$ – effective moisture diffusivity; $E_{
m a}$ – activation energy

The value of the activation energy of forced convective dried samples was $39.712~\text{kJ·mol}^{-1}\cdot\text{K}^{-1}$ and that for the vacuum dried samples was recorded to be $41.692~\text{kJ·mol}^{-1}\cdot\text{K}^{-1}$, respectively. According to Zogzas et al. (1996), the values of the activation energy for nopal cladodes lay in the range of 12.7 to $110~\text{kJ·mol}^{-1}\cdot\text{K}^{-1}$ for food materials. The obtained values are well in the acceptable range.

CONCLUSION

The time consumed for drying the nopal cladodes is less by the convective drying method as compared to vacuum drying method at the temperature of 40,

50 and 60°C. The drying rate constant was found to be more in case of convective dried samples compared to the vacuum dried samples. Similarly, effective diffusivity was found to be higher in the convective dried samples than the vacuum dried samples. However, activation energy was high for the vacuum dried samples (41.692 kJ·mol⁻¹·K⁻¹) than the convective dried samples (39.712 kJ·mol⁻¹·K⁻¹). On the basis of statistical parameters, the Page model was considered as the best fitted model for both the forced convective dried cactus cladode samples at 40°C and the vacuum dried cactus cladode samples at 50°C. The convective drying process is widely used for dried food production or foods with reduced water content as the dried food products exhibit extended shelf life along with good quality. In addition, post-

harvest losses can also be reduced. Thus, by using the mathematical models used to outline the experimental data in drying process, several laboratory tests can be reduced. Moreover, important parameters can also be obtained such as drying rate, drying time and mass diffusion coefficient that are important in designing the processes and dryers.

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