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RESEARCH ARTICLE

Drying of horse chestnut (*Aesculus indica*) starch: drying kinetics and morphology

Syed Insha Rafiq[1], Syed Mansha Rafiq[2], Khalid Muzaffar[1], D.C. Saxena[1]

[1] Department of Food Engineering and Technology, SLIET, Longowal, Punjab India
[2] Dairy Technology Division, ICAR-National Dairy Research Institute, Karnal Haryana, India

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ABSTRACT

An investigation was carried out to study the drying kinetics of Horse Chestnut starch dried in hot air oven and the impact of temperature on morphology of starch granules. Starch extracted from Horse Chestnut flour was dried at different temperatures of 30, 40 and 50°C. The experimental drying data obtained was then fitted to six different theoretical and/or empirical drying models. Nonlinear regression analysis was performed to find the best fit model. Among all the models, the Diffusion model was found to best fit the experimental data. The activation energy (Ea) for drying process was 13.66kJmol-1. Morphological analysis of the starch revealed that starch dried at 40°C showed better quality with smooth surface of starch granules and thus may be considered as a limiting temperature for drying of Horse Chestnut starch.

Keywords: Horse chestnut starch, drying kinetics, regression analysis, activation energy, morphology

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INTRODUCTION

Indian Horse Chestnut (*Aesculus indica* Colebr) or Himalayan chestnut locally recognized as *Bankhor* in Himachal Pradesh and *handun* in Kashmir. The seeds of Indian Horse Chestnut contain moisture (50.5%), starch (38.3%), sugars (5.85%), ash (1.93%) fiber (1.73%), fat (1.3%) and proteins (0.39%), respectively (Singh et al., 2003). The seeds contain high amount of starch that can be utilized for various industrial purposes.

Drying, a simultaneous heat and mass transfer process under controlled conditions wherein the water content is transferred via diffusion to the interface and from the interface by convection to the air stream (Radhika et al., 2011). It intends to enhance the shelf life of the products and also minimizes the packaging, storage and transportation costs by reducing the weight and volume of the products (Srinivasakannan and Balasubramaniam, 2006). The drying of foods is carried out either by traditional sun drying or mechanical methods like convective drying in hot air dryers (Akpinar and Bicer, 2008). The most important factor affecting the drying process is the temperature of the air (Mercer, 2012). The drying kinetics study is necessary to reduce the energy consumption by determining the optimum drying conditions (Babalis and Belessiotis, 2004). The drying kinetics are predicted accurately by numerous drying models taking into consideration the temperature, moisture ratio, drying rate, diffusivity and drying

* *For correspondence*: S. I. Rafiq (Email: syedinsha12@gmail.com)
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time. The drying characteristics and simulation models of food products being dried are essential for the design, construction and operation of drying systems. The drying time of numerous products can be estimated from the drying equations as well as to generalize drying curves (Krokida et al., 2003). The aim of the present study was to evaluate the effect of drying temperature on drying characteristics of the Horse Chestnut starch and the morphology of starch granules.

MATERIALS AND METHODS

Procurement of raw material

Horse Chestnut seeds were harvested from trees of the area Anantnag, Jammu & Kashmir, India and then washed with water to remove impurities.

Starch Isolation

Starch was isolated as per the modified method of Rafiq, et al. (2016). Horse Chestnut flour was dipped in sodium hydroxide solution (0.25%, w/v) with ratio of 1:3 (w/v) and kept at 4°C. The slurry was agitated and kept for settlement after filtration through 300 mesh sieve and 2–3 washings with distilled water followed by centrifugation at 3000 rpm for 15 min. After centrifugation the aqueous phase was discarded and the upper brown layer was scraped off while inner white starch layer was re-suspended and centrifuged again for 2–3 times. The starch obtained was then collected and dried.

Drying of Starch

For the study drying kinetics, isolated Horse Chestnut starch was dried on trays in an oven (M/s. Linco, Ambala, India), equipped with a fan, electric heater and temperature indicator. Around 10 g of starch sample was spread uniformly on trays. Sample was removed periodically after 15 min and weighed. The starch was dried till equilibrium moisture content was reached. The experiments were done at drying temperatures of 30, 40 and 50 °C.

Determination of drying characteristics of Horse Chestnut Starch

Average moisture content

The average moisture content (*MC*) of starch sample was evaluated by the hot air oven method and was calculated from equation (1):

$$MC = \frac{W_o - W_f}{W_o} \qquad (1)$$

Where, W_o = initial sample weight (g); W_f = final sample weight (g)

The moisture content of Horse Chestnut starch at time "t" can be transformed to moisture ratio (MR) using the Page's equation (Goyal et al., 2007):

$$MR = \frac{M - M_e}{M_i - M_e} \qquad (2)$$

Where, MR= moisture ratio (dimensionless); M= average moisture content at time t (kg water/ kg dry matter); M_i = initial moisture content (kg water per kg dry matter); M_e= equilibrium moisture content (kg water per kg dry matter).

Drying rate

Drying rate of starch for each temperature was calculated based on amount of moisture removed per unit time per kilogram of dry matter (Agarry et al., 2005).

Analysis of drying data

The drying process during the falling rate period can be described by Fick's second law of diffusion (Tulek, 2011). Based on Fick's second law of diffusion the moisture ratio versus drying time curves for different drying air temperatures in thin layer drying can be calculated. The data obtained from drying process was fitted to six drying models (**Table 1**) using nonlinear least square regression analysis. One of the main criteria to select the best model that describes the drying curves of dehydrated samples is the determination of correlation coefficient (R^2). In addition to R^2, reduced chi-square (χ^2) and Root Mean Square Error (RMSE) were used to determine the quality of the fit as calculated below:

$$RMSE = \sum_{i=1}^{N} \sqrt{\frac{(MR_{Experimental\ Value} - MR_{Predicted\ Value})^2}{N}} \qquad (3)$$

$$\chi^2 = \sum_{i=1}^{N} \frac{(MR_{Experimental\ Value} - MR_{Predicted\ Value})^2}{(N-n)} \qquad (4)$$

Where, *N* is number of experimental data points, and *n* is number of model constants.

Effective moisture diffusion and activation energy

The dependence of effective diffusivity on temperature was evaluated using an Arrhenius-type equation from which the activation energy (E_a) was calculated (Xiao *et al.,* 2010):

$$\mathrm{k} = \mathrm{k_o}\, \mathrm{e}^{-\mathrm{E_a}/\mathrm{RT}} \qquad (5)$$

Where, "E_a" is activation energy of moisture diffusion (kJ/mol); "k_o" is diffusivity value for an infinite moisture content (m^2/s); "T" is the drying air temperature (ºK) and "R" is the universal gas constant (kJ/mol K).

Morphology of dried starch

Scanning electron micrographs of starches dried at 30, 40 and 50°C were observed by SEM (scanning electron microscope, JSM-6100, JEOL Ltd. Japan). The starch sample was mounted on SEM stubs coated with platinum by means of double-sided adhesive tape and micrographs were taken at 10 kV acceleration potential with 500 X magnification.

RESULTS AND DISCUSSION

The initial moisture content of Horse Chestnut starch isolated under optimum conditions was found to be 30.5%. The drying experiments were carried out at different temperatures of 30, 40 and 50°C till equilibrium moisture content reached.

Moisture ratio

The moisture content values of Horse Chestnut starch at particular time for each temperature were converted to moisture ratio and plotted against time as shown in Figure 1. The moisture ratio of the Horse Chestnut starch decreased continuously with time at all drying temperatures. The constant decrease in moisture ratio indicated a diffusion controlled internal mass transfer (Lewicki, 2006). At first, the moisture ratio decreased rapidly and then gradually with increase in the drying time. At any instant of time, moisture ratio was lower at higher drying temperature than at lower drying temperature (Akpinar and Bicer, 2008). It was apparent that the time for equilibrium moisture content shared an inverse relationship with drying air temperature. The effect of temperature, time, initial and final moisture content on drying constants have been studied extensively (Midilli and Kucuk, 2003).

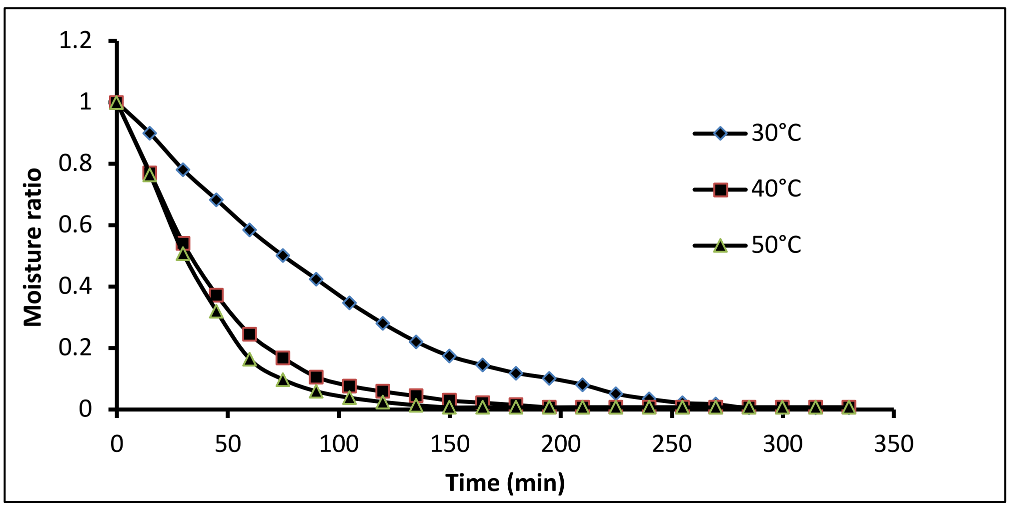

Fig. 1. Variation in moisture ratio as a function of temperature during drying of Horse Chestnut seed starch

Drying rate

The drying rate curves for Horse Chestnut starch at 30, 40, and 50°C temperatures are presented in Figure 2. It was observed that the drying rate decreased constantly throughout the drying period. The entire drying process of starch took place in falling rate period that shows diffusion was the key physical mechanism for moisture movement in Horse Chestnut starch (El-Sebaii et al., 2002). The drying rate for same moisture content was initially observed to be highest at 50°C as compared to other temperatures as higher drying air temperature removed moisture at a faster rate. The rate of moisture loss was observed to be more at higher temperatures and total drying time was reduced significantly with increase in drying temperature. The required drying time for Horse Chestnut starch was highest at 30°C and lowest at 50°C.

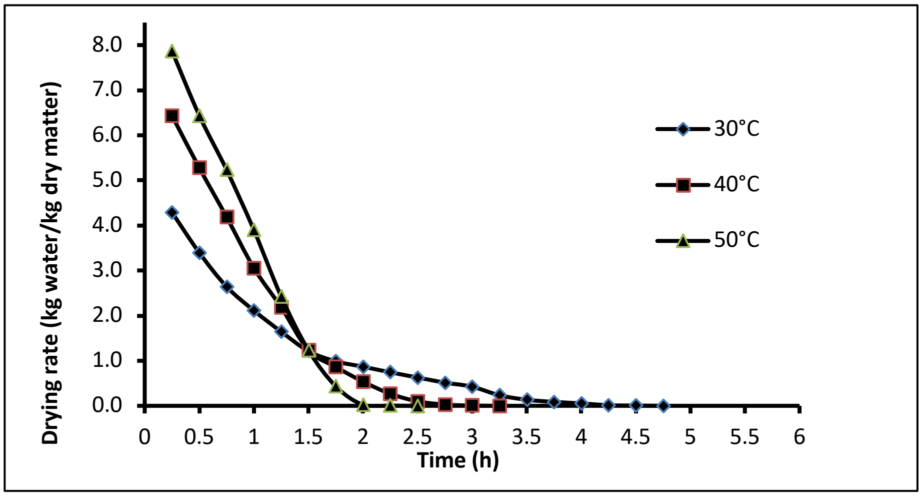

Fig. 2. Drying rate of Horse Chestnut seed starch as a function of time at different dry temperatures

Drying data analysis and model fitting

For each drying air temperature, results of moisture ratio at corresponding drying time of Horse Chestnut starch were fitted to six different drying kinetic models as listed in Table 1. The drying models were used to depict the drying characteristics of Horse Chestnut starch in thin layer drying oven (Krokida et al., 2003). The models were fitted to the experimental moisture ratio data by means of nonlinear regression in MS Excel. The results of regression analysis obtained after fitting the experimental data to six different drying models along with Correlation coefficient (R^2), root mean square error (RMSE) and chi-square (χ^2) are presented in Table 2. Based on the minimum chi-square and RMSE values as well as maximum R^2 value for the best fit, the diffusion model (Eq. 6) was found to be the best fit model to experimental drying data.

Table 1: Mathematical models for drying kinetics of Horse Chestnut seed starch

S. No.	Model Name	Model Equation	References
1	Newton	MR = exp(-kt)	Demir et al. (2004)
2	Modified Page	MR = exp $(-kt)^2$	Demir et al. (2007)
3	Wang and Singh	MR = $at^2 + bt + 1$	Mohapatra and Rao (2005)
4	Two-term	MR = a $\exp^{(-kt)}$ + b $\exp^{(-nt)}$	Ertekin and Yaldiz (2004)
5	Two-term exponential	MR = a $\exp^{(-kt)}$ + (1-a) $\exp^{(-kat)}$	Akpinar (2006)
6	Diffusion	MR = a $\exp^{(-kt)}$ + (1-a) $\exp^{(-kbt)}$	Cihan et al. (2007)

a, b, k, n are empirical constants in drying models; (t) is the drying time (min); (MR) is the moisture ratio

Table 2: Model parameters and statistics used to evaluate the suitability of the models for drying of Horse Chestnut seed starch

Model	Temp. (°C)	R^2	χ^2	RMSE	k	a	b	n
Newton	30	0.98491	0.00148	0.03767	0.010627	-	-	
	40	0.99619	0.00028	0.01628	0.022412	-	-	
	50	0.98889	0.00079	0.02757	0.026115	-	-	
Modified Page	30	0.98491	0.00148	0.03767	0.005314	-	-	
	40	0.99619	0.00028	0.01628	0.011206	-	-	
	50	0.98889	0.00079	0.02757	0.013057	-	-	
Wang and Singh	30	0.99524	0.00049	0.02116	-	-0.007443	0.000014	
	40	0.79004	0.01597	0.12077	-	-0.010300	0.000024	
	50	0.70380	0.02219	0.14235	-	-0.010732	0.000025	
Two term exponential	30	0.98491	0.00155	0.03767	0.010600	-18.7105	-	
	40	0.99619	0.00029	0.01628	0.022412	0.709227	-	
	50	0.98889	0.00083	0.02757	0.026115	0.689270	-	
Two term	30	0.98939	0.00115	0.03159	0.01128	6.81830	-5.75138	0.017885
	40	0.99704	0.00024	0.01433	0.02307	-3.13940	4.171040	0.022412
	50	0.99083	0.00072	0.02505	0.02720	-10.4951	11.54360	0.040617
Diffusion	30	0.99886	0.00012	0.01035	0.01944	-8.40365	0.923800	
	40	0.99962	0.00003	0.00511	0.06339	-0.177985	0.212522	
	50	0.99931	0.00005	0.00687	0.121502	-1.42187	0.640740	

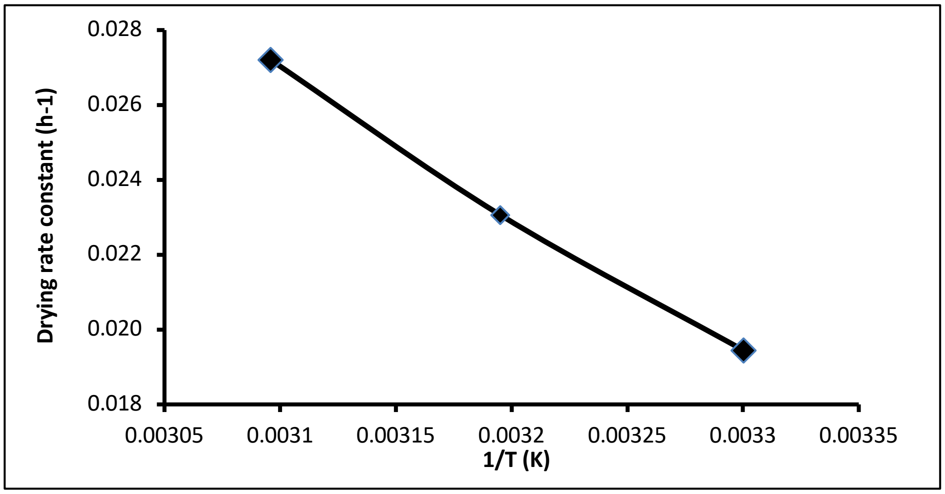

Fig. 3. Temperature dependence of drying rate constant for Horse Chestnut starch

Rate constant and activation energy

The rate constant (k) which is a measure of the drying rate, calculated from most suitable drying model (diffusion model) increased (0.01944–0.121502 h^{-1}) with increase in drying air temperature (30 to 50ºC). The activation energy (E_a) was determined by plotting the natural logarithm of rate constant 'k' (calculated from most suitable drying model, diffusion model) against the reciprocal of drying air temperature (1/T) according to the Eq. 5. Plot of drying rate constant against drying time showed a linear relationship (Fig. 3) and the computed value of activation energy for drying of Horse Chestnut starch was 13.663 $kJmol^{-1}$. The activation energy is the relative ease of moisture movement within the food product and a lesser value indicates high moisture diffusivity (Sharma and Prasad, 2004).

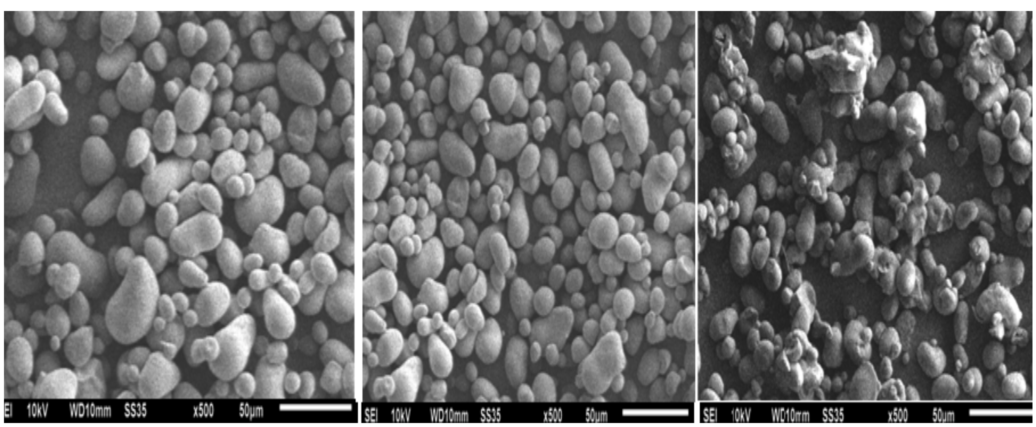

Fig. 4. Micrographs of Horse Chestnut seed starch dried at 30, 40 and 50°C

Effect of drying air temperature on starch morphology

The scanning electron micrographs of the starch dried at 30, 40 and 50°C are presented in Fig. 4. The HCN starch granules dried at 30 and 40°C appeared to be round or oval with smooth surface when viewed under Scanning Electron Microscope. Surface granular disruption was observed in starch dried at 50°C as the drying temperature produced changes in the physical structure of starch granules. The surface degradation of starch granules may be due to the high temperature during drying process that resulted in the partial gelatinization of moist granular surface. The best quality granules were observed in starches dried at 30 and 40°C but time of drying was observed more at 30°C and thus drying air temperature of 40°C was considered as an optimum temperature for starch drying in order to retain the quality of the starch as well to minimize the drying time. Attanasio et al. (2004) have shown the morphological characteristics of fresh chestnut starch granules to be round and oval with smooth surface but after drying the surfaces were quite rough and roughness increased at higher temperature (60ºC).

CONCLUSIONS

Drying kinetics of Horse Chestnut starch was studied at three different temperatures of 30, 40 and 50°C. Drying of Horse Chestnut starch occurred only in the falling rate period. Both the moisture ratio and drying rate decreased with time. The experimental data was compared with the predicted values of the six drying models. Although, all the models were considered fit with the experimental data, but the Diffusion model showed the highest R^2 (coefficient of determination) and least RMSE (root mean square error) and χ^2 (Chi-square) values. Higher drying temperature resulted in decreased drying time but the increase of temperature causes damage to the starch as revealed from the micrographs and the results suggest that the effective air temperature should not exceed 40 °C to maintain a good quality of Horse Chestnut starch.

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