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Osmotic Dehydration of Litchi Using Sucrose Solution: Effect of Mass Transfer

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Abstract

Minimal processing techniques like osmodehydration have been finding a significant place in post-harvest practices of fruits and vegetables. Osmodehydration is adopted for extending shelf life of certain fruits. The overall effectiveness of the process is determined by process parameters affecting the mass transfer phenomena. In the present study this technique has been extensively applied on Litchi (*Litchi chinensisSonn*), a sub-tropical fruit. The process has been effectively modeled and the findings of the result suggest that sugar concentration and temperature significantly influence the process. Developed model could adequately predict the equilibrium point. The effective diffusion coefficients for water loss and solid gain obtained for the process ranged from 0.23 to 0.348×10^{-10} m^2s^{-1} for water loss and from 0.0428 to 0.0721×10^{-10} m^2s^{-1} respectively.

Keywords: Osmo-dehydration; Mass transfer; Drying rate

Introduction

Central preoccupation of the Food Processing Industry is, ensuring the supply of safe product, for consumption. Presently, the industry has a renewed interest in minimal processing techniques for preservation of food quality especially the fruits and vegetables. Litchi (*Litchi chinensisSonn)*, is an important sub tropical evergreen fruit crop belonging to family Sapindaceae. Being exacting in climatic requirement it is confined to few areas of India, China etc. Annual production of litchi in India is about 50,000 tons. It is a means of livelihood of small and marginal farmers native to the locality. The food value of litchi mainly lies in its sugar content and presence of vitamins like: vitamin B, Riboflavin, Vitamin C etc. It makes an excellent raw material for canned fruit, squash, pickles, preserved wine, dried litchi etc. Litchi possesses poor shelf life (2-3 days under ambient conditions) and therefore needs specific treatment before packaging and being transported to long distances. With proper post harvest treatment (precooling, sulfuric acidification, storage at low temperature) it can be preserved for only 2-3 weeks. Also, the existing chemical preservation methods have been restricted; hence, there is an immediate need for alternative methods.

Contextually osmotic dehydration is one of the varied and most adopted techniques in vogue, for extension of the shelf life of food products. It is the phenomenon of removal of water from lower concentration of solute to higher concentration levels, across a semi permeable membrane, finally resulting in equilibrium state on both sides of membrane [1]. The process is executed by immersing the sample in a hypertonic solution [2,3]. In the industry it is adopted as a pretreatment process for freezing, freeze drying, vacuum drying etc. The process finds increased application in preservation since, it lowers water activity without altering the nutritional, flavor and textural integral characteristics of the initial product [4]. Further, the low operational temperatures also prevent the enzymatic and oxidative browning thereby enhancing the storage life of the product [5,6].

Osmotic dehydration process is a multicomponent diffusion process that involves three types of counter mass transfer phenomena. These include, water outflow from the food tissue to the osmotic solution, solute transfer from the osmotic solution to the food tissue and leaching out of the food tissue's own solutes (sugars, organic acids, minerals, vitamins) into the osmotic solution [7,8]. The latter transfer is quantitatively negligible compared with the other two types. Thus, the driving force in this process is the difference in the osmotic pressure of solutions on both sides of the semi-permeable membrane.

The diffusion of water and low-molecular weight substances from the tissue structure during the osmotic dehydration is accompanied by the counter-current diffusion of osmoactive substances [9]. All these mass exchanges between the osmotic solution and foodstuff have an effect on the overall quality of the dehydrated product i.e. nutritional value, texture, color and taste. Hence, diffusion, osmotic processes, flux interactions, and tissue shrinkage should all be taken into account for accurate description of the mass transfer phenomena during osmotic dehydration. Water diffusivity rate from a sample and uptake of solids is dependent on several factors such as types of osmotic agent, concentrations of osmotic agent, processing temperatures, agitation or stirring process, pretreatment methods and presence of coating if any [10-13]. Efforts have been made by researchers to increase the rate of osmotic mass transfer for reducing the processing time [14-17] and minimizing the uptake of osmotic solids, as it can severely alter the organoleptic and nutritional profile of the product [18-20]. Thus an understanding of the various underlying factors affecting the process and their modeling is essential for process optimization. The mass transfer process has been modeled based on the theories of Fickian diffusion, irreversible thermodynamics, multicomponent diffusion, and hydrodynamic flow. Evaluation of the long-term equilibrium and the distribution of the phases in the tissue provide a better understanding of the phenomena that control the mass transfer processes in osmotic dehydration.

Hence the present work has been undertaken to study mass transfer parameters during osmotic dehydration of litchi. It also includes examination of the predictive capacity of modified Fick's model of diffusion presented in earlier reports [21,22].

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Mathematical Basis of the Osmotic Dehydration Modeling Studies

In the studies reported, mass transfer in osmotic dehydration of fruits at atmospheric pressure has been modeled either by a phenomenological approach using Crank's model (which is a Fick's law solution) or empirically, using models developed from polynomial equations, mass balances, or relations between process variables (i.e., Magee's, Azuara's, and Page's models). Most of the models for mass transfer evaluation during the osmotic process are based on the mathematical solutions of Fick's Law of Diffusion. Crank has reported that it could be used for the determination of the water and sugar diffusion coefficients [22]. Similar applications have been made for varied range of foods [23-28]. Several reports on process kinetics of the dehydration process showing a good fit to the experimental data for various fruits and vegetables are available [29,30]. For all of these models, experimental data are required to determine the values of their adjustable parameters for specific processing conditions. Comparative evaluation of the various models have demonstrated that Azuara's and Page's models yield better correlations (with mean absolute errors less than 1.26% for *ML* and 0.46% for *SG*) than Magee's and Crank's models (with mean absolute errors of up to 2.98 and 1.68% for *ML* and *SG*, respectively). A two-parameter equation, developed by Azuara et al. from mass balance considerations, has been used in these latter models to predict the kinetics of osmotic dehydration and the final equilibrium point [31]. The model was tested using kinetic data from different experiments. Model was able to predict water loss and solids gained over long periods of osmotic dehydration. The final equilibrium point could also be estimated using data obtained during a relatively short period of time.

Simple equations were obtained when the model was related to Fick's second law for unsteady one-dimensional diffusion through a thin slab, and diffusion coefficients were readily estimated from these equations.

The mass balance of water inside the food sample is given by equation (1):

$$WL = WL_{\infty} - WS \tag{1}$$

where, WL is the fraction of water lost by the sample at any time, WL_{∞} is the fraction of water lost by the sample at equilibrium and WS is the fraction of water that can diffuse out, but remains inside the sample at time t.

In equation (1) WL_{∞} has a fixed value for the established conditions of temperature and concentration. On the other hand, WL and WS are functions of the rate of water loss and time. A relationship between WL, WS, and a constant (S_1) related to the water loss is given by equation (2):

$$WL = \frac{S_1 t (WL_{\infty})}{1 + S_1 t} \tag{2}$$

in which S_1 is a constant related to the water loss and t is time.

When $t \to \infty$ (at equilibrium), WL tends to the asymptotic value WL_{∞}. The values of S_1 and WL_{∞} can be calculated by a non-linear estimation program or by linear regression, using experimental data obtained during a short time and the linear form of equation (2).

Similar equations can be written for the gain of soluble solids by the product:

$$SG = \frac{S_2 t (SG_{\infty})}{1 + S_2 t} \tag{3}$$

in which SG is the fraction of soluble solids gained by the food at time t; SG_{∞} is the fraction of soluble solids gained by the food at equilibrium, and S_2 is a constant related to the rate of soluble solid incoming to the food sample.

Based on Fick's second law, Crank proposed an equation for one dimension diffusion in a flat sheet in contact with an infinite amount of solution. Its simplified form when "t" is small is given by equation (4):

$$\frac{WL_t}{WL_{\infty}} = 2\left(\frac{Dt}{\pi l^2}\right)^{\frac{1}{2}} \tag{4}$$

in which WL_t is the amount of water leaving (WL) or solute entering (SG) the food sample at time t; WL_{∞} is the amount of water leaving or solute solids entering the sample at infinite time (WL_{∞} or SG_{∞}); D is the effective diffusion coefficient; and L is the half thickness of the slab.

From equation (4), equation (5) is obtained, which is a simple expression from which D can be easily calculated at different times.

$$D_t = \frac{\pi t}{4}\left[\left(\frac{S_i l}{1 + S_i t}\right)\left(\frac{WL_{\infty}^{\mathrm{mod}}}{WL_{\infty}^{\exp}}\right)\right]^2 \tag{5}$$

in which $S_i = S_1$ or S_2; WL_{∞}^{model} is the equilibrium value obtained from Equations (2) or (3); WL_{∞}^{Exp} is the equilibrium value experimentally obtained; D_t is the effective diffusion coefficient at time t, which can be readily obtained.

The theoretical equilibrium value (X_{∞} model), and the constant, S, are estimated using the experimental data (X_t) and linear regression:

$$\frac{t}{X_t} = \frac{1}{SX_{\infty,\mathrm{mod}}} + \frac{t}{X_{\infty,\mathrm{mod}}} \tag{6}$$

The expression presented by Azuara et al. was tested on data pertaining to the osmotic treatment of apple, pineapple and beef and fishes [31]. Rate constants and diffusion coefficients for the initial stages of the process are determined from plots of $\frac{X_t}{X_{\infty}}$ vs.t ^{½}

$$\frac{X_t}{X_{\infty}} = Kt^{1/2} \tag{7}$$

Where, $K = 2\left(\frac{D}{\Pi L^2}\right)^{1/2}$, X_t is the amount of solute entering/water leaving the sample at time t; X_{∞}, amount of solute entering/water leaving the sample at equilibrium; D, diffusion coefficient for solute / water flow; L, half-thickness of slab; t, time.

Based on this model Crank and Azuara et al. presented an expression from which the diffusion coefficient can be calculated at different times during the osmotic process, and not just only for the initial stages of dehydration.

Materials and Methods

Sample preparation

Litchi was procured from local markets of Kolkata. The fruit samples were purchased in batches to avoid variation in samples. Samples were washed thoroughly, manually peeled, de-seeded and cut into slices 0.5mm thick. Samples were gently blotted with tissue paper to remove adhering surface water and weighed.

Aqueous sugar solutions of required ^{0}Brix (30-50^0 Brix) were used as osmotic dehydration media. Sugar solution was prepared by dissolving appropriate amount (30, 40 and 50 g) of sugar per 100 g of solution.

Osmotic dehydration

Dehydration i.e. the osmotic process was carried out at different sucrose concentrations (30-50^{0}Brix). Trials were conducted by keeping the solution temperatures (30-50°C) with agitation of 90 rpm. The slices were placed in 250 ml beakers containing the osmotic solution and maintained inside a temperature-controlled bath. In order to avoid significant dilution, subsequent decrease of the driving force during the process, the mass ratio of osmotic medium to fruit sample was kept at 20:1. Samples were removed from the solution at 15, 30, 45, 60, 75, 90, 120, 150, 180 and 240 min after immersion, drained and the excess of solution on the surface was removed by absorbent tissue paper.

Average moisture and dry matter content of the samples were determined by AOAC, method by drying the dehydrated samples at 70°C for 24 h in an oven [32]. All the experiments were done in triplicate and the average value taken for calculations.

Water loss (WL) has been expressed as the net water loss from the freshly peeled and sliced litchi samples after osmotic dehydration based on initial sample mass. Solid gain (SG) has been defined as the net solid uptake by the litchi sample, based on initial sample weight [33]:

$$\%WL = [\frac{(M_i - M_o)}{W_i}]\times 100 \quad (8)$$

$$\%SG = [\frac{(S_0 - S_i)}{W_i}]\times 100 \quad (9)$$

in which M_i is the initial moisture content (g); M_o is the moisture content of osmotically treated sample (g); S_i is the solids content of osmotically treated sample (g); S_o is the initial solids content (g); W_i is the initial total sample mass (g).

Using the experimental measurements the moisture loss and solid gain were calculated and also estimated as functions of the solution concentration and temperature. Curves of water loss and solid gain as a function of time were constructed using experimental data. Values for the parameters like drying rate, sugar gain rate were estimated. The resulting experimental data has been evaluated for fitting a representative Azuara model equation. With the model equation, the diffusion coefficient was calculated at different times during the osmotic process including the initial stages of dehydration. Equilibrium values of moisture and solid content and effective diffusivities were determined from the developed model equation. Likewise the time dependent diffusivities were also evaluated.

Results and Discussion

Percentage Water loss and Solid gain

For the experimental samples the % water loss and % solid gain were calculated using equations (8) and (9) respectively. The values were estimated for samples treated at different ^{0}Brix, varied temperature and kept at distinct duration of time. Graphical representations of the observations have been represented in Figures 1-3.

Significant WL was observed during first 150 minutes which gradually decreased with time till equilibrium point. As may be observed the same pattern was obtained for samples at varied ^{0}Brix solution. This can be attributed to the large osmotic driving force between the fruit and the surrounding hypertonic medium. This result corroborates with those obtained by several research groups studying osmotic dehydration of cantaloupe, mango slices, apricot and guava cubes [34-37].

It was observed that the solid content of litchi slabs was also significantly affected by sugar concentration of immersion solution, temperature and time as depicted in the Figures 4-6. From Figures, it may be observed that there was a rapid increase in solid gain during the first 150 minutes and then the decrease was gradual with time. The increase in solid gain with increasing sucrose concentration is comparatively lower than the corresponding water loss under the same conditions. For 3 hour osmosis period the highest water loss (0.53 g water/gm solid) and solid gain (0.92 g solid/gm solid) were observed with 50^{0}Brix sucrose solution, while the lowest water loss (0.35 g water/solid.) and solid gain (0.048 g solid/g solid.) was with 30^{0}Brix sucrose solution.

These results indicate that some benefits in terms of faster WR and WL could be achieved by choosing a higher concentration of medium. However, a much greater SG is also observed and reported elsewhere. This finding also confirmed that highly concentrated sucrose solution (50^{0}Brix) is a mass transfer rate limiting parameter during osmotic dehydration process. Lazarides et al. noted that at increased temperatures, high rates of WL during the osmotic dehydration of apples seem to prevent the development of proportionally high rates of counter current sucrose diffusion [38]. They added that whenever it is desirable to achieve higher water removal and lower solids gain, a higher process temperature (within allow-able limit) should be used. This may

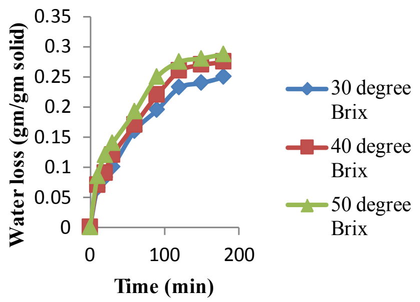

Figure 1: Water loss in different Brix solutions at 30°C.

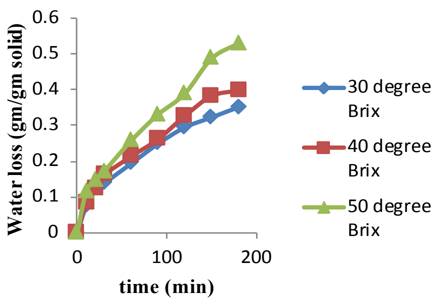

Figure 2: Water loss in different Brix solution at 40°C.

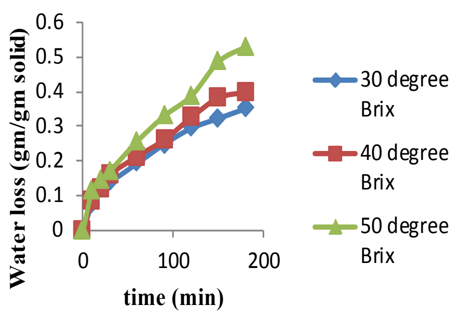

Figure 3: Water loss in different Brix solution at 50°C.

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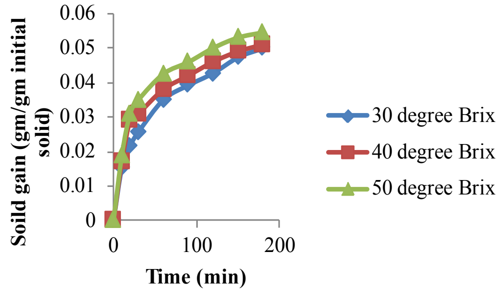

Figure 4: Solid gain in different Brix solution at 30°C.

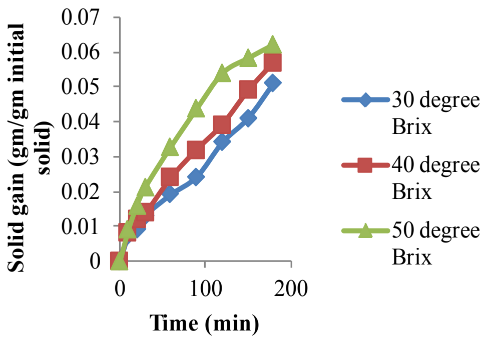

Figure 5: Solid gain in different Brix solution at 40°C.

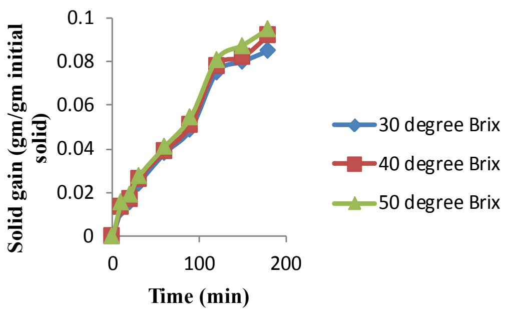

Figure 6: Solid gain in different Brix solution at 50°C.

be explained since osmotic dehydration is a two way diffusion process which is strongly dependent on temperature. Similar observations on the influence of temperature on osmotic dehydration rate were made and reported [39,40]. While temperature can be advantageously used to complete osmotic dehydration rapidly, it should be noted that higher temperature may adversely affect colour and flavor. Consideration of these and other factors such as tissue integrity lead Pointing et al. to suggest a maximum temperature limit of 49°C for osmotic dehydration [41].

Rate of change in moisture content (MC) and content (SC)

Rate of removal of moisture is a characteristic of prime importance to every dehydration process as it is indicative of process effectiveness. It is also suggestive of the productive duration of the process. MC data for litchi slabs were used to calculate the rate of change in moisture (Rate MC). This was done by calculating the difference in MC (g H_2OgDM^{-1}) between consecutive sampling times (t, t+1), and dividing this value by the time interval (min). A plot of the rate of change in moisture (dM/dt) in litchi slabs vs. immersion time has been shown in Figures 7-9.

Generally, during the first hour, the higher the solution temperature, the higher the drying rate. It may further be observed that rate is highest at the beginning and declines rapidly within the first hour of dehydration.

Drying rates were highest for slabs at 50°C, and lowest for slabs at 30°C. Initial rates of dehydration averaged 0.0086, 0.0088 and 0.0119 g H_2Og DM^{-1} min^{-1}) for dehydration at 30, 40 and 50°C respectively by 50° Brix. Beyond 2h of dehydration, rate changes were negligible regardless of sugar solution temperature. Also In the plots given it may be observed that there are no periods of constant water removal and therefore no constant rate period. Rates declined with declining MC. This means that the drying rate of slabs was dependent on the moisture concentration inside the lichhi.

It is generally accepted that mass transfer during osmotic dehydration of fruits is governed by internal diffusion, that is, movement under the influence of a concentration gradient. Where a constant rate of drying does occur, the period is brief and does not exceed tens of seconds [42]. As also shown by Lenart and Lewicki for the osmotic dehydration of fruit, the relationship between rate and MC

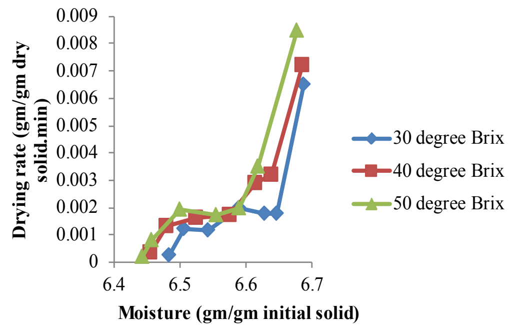

Figure 7: Drying rate in Brix at 30°C.

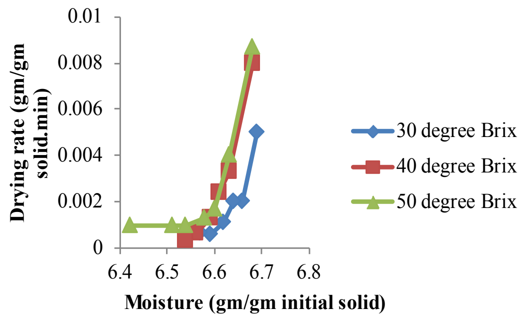

Figure 8: Drying rate in Brix at 40°C.

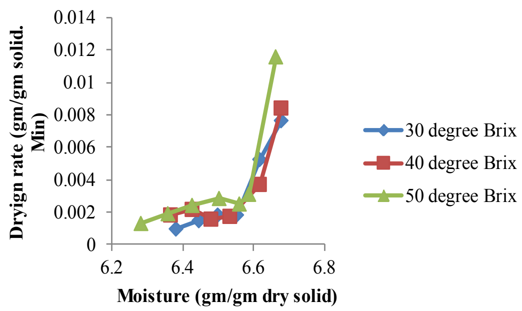

Figure 9: Drying rate in Brix at 50°C.

of litchhi during the falling rate period is firstly exponential in character [43]. However, when the MC falls below a certain critical value, in this case approximately 6.6 g H_2Og DM^{-1}, the plot was linear.

High rates of WL and solids gain during the initial stages of dehydration followed by drastically lower rates have been attributed to the large initial osmotic driving force between the sample and the surrounding hypertonic solution, structural changes such as shrinkage leading to the compaction of the surface layers of the tissue and the decreasing availability of free or loosely bound water leading to the progressively slower moisture removal as the process goes on [43,44].

Diffusion coefficient vs time at different temperatures

The diffusion coefficients for WL in litchi slabs were calculated at different times during the osmotic process using eqn 2.The observations made have been represented in Figures 10-14. Diffusivities were significantly affected by immersion time and immersion solution temperature. Generally values were highest approximately after 1 h of dehydration. Thereafter there is initially a rapid decline followed by a gradual decline. The diffusion coefficients for WL increased as sucrose temperature increased. As shown in figures water diffusivity values for the first hour of dehydration averaged 6.7, 7.5 and 7.9×10^{-10} m^2s^{-1} for dehydration by 30, 40 and 50^0 Brix, respectively at 30^0C. The results were found to be comparable with the results of model no. 2, which is applicable only to the initial stages of dehydration. The advantage of using this approach is therefore the ability to calculate diffusivities for the entire duration of the osmotic treatment and not just the initial stages. As shown in figures, there is a similar tendency at 40 and 50˚C.

Similar observations have been for the first hour of dehydration averaged 8.5, 8.6 & 11.5×10^{-10} $m^2s^{-1}$for dehydration by 30, 40 and 50^0 Brixas reported by Azuara et al. where data of Favetto et al. [21,24] for the salting of beef was modeled and the diffusion coefficient estimated .It was found that it was not constant for the duration of the diffusion process. At 85°C, D-values decreased from an initial value of $4.0\text{-}1.5\times10^{-5}$ cm^2s^{-1} in 3 h. At 30°C, D-values increased from an initial value of $0.5\text{-}1.0\times10^{-5}$ cm^2s^{-1} in 3 h.

Rate constants and diffusion coefficients

The experimental data obtained at varied conditions was tested with Model no: 1and model no:2 to estimate the rate constants and diffusion coefficients When the solution given by Crank for a well-stirred solution was applied to the WL and SG by litchi slabs, the $t^{1/2}$ law given in eqn 1 could be applied satisfactorily to the linear section of the data which corresponds to the first 2 h of dehydration. The slopes of these plots have been given in Table 1.

At higher the temperature of the osmotic treatment, slope of the graph was steeper and the value of constant K was also higher. Increasing the sucrose solution temperature from 30 to 50˚C resulted in an increase in the constant for moisture diffusion from 0.0632-0.084 $min^{0.5}$ at 30^0Brix, which was almost one and half fold increase. While the constant for water diffusion was 0.074-0.086 $min^{0.5}$ when sucrose solution was of Brix 40^0 it varied from 0.082-0.088 $min^{0.5}$ at 50^0 Brix sucrose solution.

Likewise based on the approach of using Crank's solution for a well-stirred solution, presented by Azuara et al. the plot of t/WL vs. t based on the straight line equation (eqn 3) was used to generate S-values (intercept) and equilibrium values (slope). The obtained values have been given in Table 2.

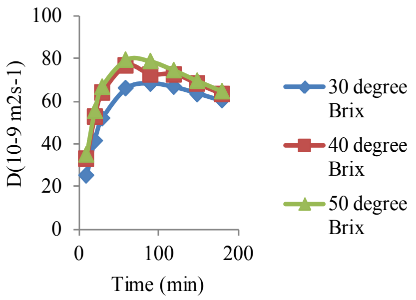

Figure 10: Diffusion coefficients (D) for water loss in litchi slabs at 30°C.

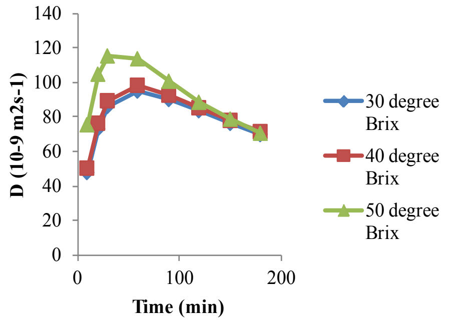

Figure 11: Diffusion coefficients (D) for water loss in litchi slabs at 50°C.

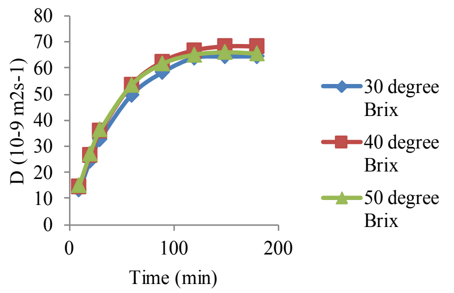

Figure 12: Diffusion coefficients (D) for sugar gain in litchi slabs at 30°C.

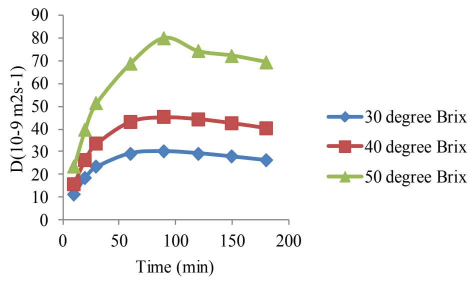

Figure 13: Diffusion coefficients (D) for sugar gain in litchi slabs at 40°C.

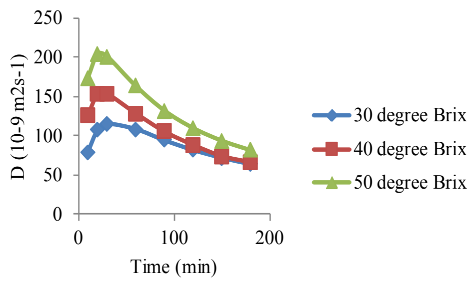

Figure 14: Diffusion coefficients (D) for sugar gain in litchi slabs at 50°C.

Temp (^{0}C)	^{0}Brix	Water loss			Sugar gain		
		K	R^2	D	K	R^2	D
30	30	0.063	0.941	0.797	0.041	0.99	0.338
	40	0.075	0.992	1.13	0.042	0.99	0.354
	50	0.084	0.977	1.42	0.046	0.99	0.425
50	30	0.08	0.99	1.28	0.093	0.99	1.73
	40	0.083	0.99	1.48	0.115	0.98	2.66
	50	0.088	0.99	1.67	0.119	0.99	2.85
40	30	0.074	0.99	1.31	0.043	0.99	1.83
	40	0.078	0.99	1.39	0.045	0.99	2.74
	50	0.086	0.99	1.79	0.058	0.97	2.96

Table 1: Constants (K) and diffusion coefficients (D) obtained using water loss (WL) and sugar gain (SG) data and model no. 1 (Crank, 1975) for litchi slabs at different temperatures.

Temp (^{0}C)	^{0}Brix	Water loss		Sugar gain	
		S-value (min^{-1})	$WL_{\alpha model}$ (g H_2OgDM^{-1})	S-value (min^{-1})	$WL_{\alpha model}$ (g sugarDM^{-1})
30	30	0.0194	0.23	0.0128	0.0428
	40	0.0257	0.2453	0.0143	0.0477
	50	0.0295	0.2646	0.0158	0.051
40	30	0.0268	0.294	0.0281	0.0578
	40	0.0275	0.325	0.0398	0.0567
	50	0.031	0.326	0.0439	0.0605
50	30	0.0281	0.289	0.0297	0.0621
	40	0.0287	0.294	0.0312	0.0682
	50	0.033	0.348	0.0348	0.0721

Table 2: Water loss (WL) from model and S value for litchi slabs at different Sugar solution.

All r^2-values were > 0.99. S-values, which are related to the rate of WL, increased from 0.0194 to 0.033 min^{-1} as sugar solution (from 30 to 50^0 Brix) temperature was increased from 30 to 50°C. The S-value is a measure of the rate of the diffusion process, with 1/S being the time taken for half the diffusible material to diffuse in or out. For example, for WL at 30°C, this corresponds to 70 min, while for WL at 50°C, this corresponds to 35 min.

Conclusion

The rate of water loss and sugar gain during osmotic dehydration of litchi was directly related to the sugar concentration and temperature. Since the experimental equilibrium conditions were not completely reached in short processing times, the Azuara et al. model was used to predict the equilibrium point. When the predicted values were compared to the experimental ones it was found that the model adequately describes the experimental values, especially for water loss. The osmotic dehydration or the process time to attain a specific water loss can be predicted using the proposed model when the model parameters are known.

The Azuara equation well described the experimental data and also well predicted water loss and solid gain. The effective diffusion coefficients obtained from the Azuara equation ranged from 0.23 to 0.348×10^{-10} m^{2}s^{-1} for water loss and from 0.0428 to 0.0721×10^{-10} m^2/s for solids gain.

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