Mass Transfer Modelling During Osmotic Dehydration of Aonla (*Emblica Officinalis* Gaertn.) Slices Cv. Chakaiya Prior to Air Drying

M.S. Jadhav¹, H.G. More², C.A. Nimbalkar³

¹ Associate Professor, Processing and Food Engineering, Dr. Anasaheb Shinde College of Agricultural Engineering, Mahatma Phule Krishi Vidyapeeth Rahuri 413722 (India)
²Ex. Director of Extension Education, Mahatma Phule Krishi Vidyapeeth Rahuri 413722 (India),
³ Associate Professor, Department of Agricultural Statistics, Post Graduate Institute, Mahatma Phule Krishi Vidyapeeth, Rahuri, India-413722

Abstract

Investigation on osmotic dehydration of aonla slices of Chakaiya variety was carried out to remove partial moisture prior to convective drying. Three sugar concentration levels (50, 60 and 70 °Brix), three temperatures levels of osmotic solution (40, 50 and 60 °C) and constant solution to fruit ratio of 6:1 (v/w) were employed and observations on water loss and solute gain were recorded at an interval of 15, 30, 60, 90, 120, 180 and 240 min. A two parameter mathematical model developed by Azura et al. was used for describing the mass transfer kinetics during osmotic dehydration of aonla slices. Effects of immersion time on mass transfer was studied and constants of two parameter model and final equilibrium points for water loss and solute gain were determined. Water loss from and solute gain by the aonla slices increased non-linearly with the duration of osmosis at all sugar concentrations and both were higher in the initial period of osmosis than the later period. Further, both the water loss and solute gain were increased with increasing sugar concentration. Model was able to predict mass transfer data of osmotic dehydration up to the equilibrium point using data of relatively short duration of osmosis.

Keywords- Aonla, Modeling, Water loss, Solute gain, Equilibrium water loss, Equilibrium solute gain

I. INTRODUCTION

Osmotic dehydration (OD) is a process for the partial removal of water from plant tissues such as fruits and vegetables by immersion in an aqueous concentrated solution of soluble salts. A driving force for the diffusion of water from the tissue into the solution is provided by the difference in osmotic pressure or concentration gradient between the food and surrounding osmotic solution. A diffusion of water is accompanied by the simultaneous counter diffusion of solute from the osmotic solution into the tissue. Since the membrane responsible for the osmotic transport is not perfectly selective, other solutes such as sugar, organic acids, minerals, salts and vitamins present in the cells can also be leached into the osmotic solution (Giangiacomo et al. [8] and Tortoe et al. [19]). But this flow can be quantitatively neglected.

The rate of mass transfer (water loss and solute gain) was found to be a function of many variables such as solution temperature, solution concentration, composition of osmotic solution, immersion time, nature of food and its geometry, solution to fruit ratio. A large number of studies are available in literature regarding the influence of different osmotic parameters on the rate and amount of mass transfer based on specific food geometry for fruits and vegetables such as banana (Pokharkar and Prasad [14]), radish (Herman-Lara et al. [9]), apple, banana and potato (Tortoe et al. [19]), papaya (Jain et al. [10]). They have developed and reported several equations based on Fick’s second law of diffusion. Further the equations developed for osmotic dehydration (Beristain et al. [3]; Ertekin and Cakaloz [6] and Chenlo et al. [4]) were usually for specific processing conditions and...
geometric considerations and can’t predict the end equilibrium point of the process. Several proposed equations based on Fick’s second law are not useful practically due to the fact that some of the assumptions are unrealistic and also because of complexity of equations. Mathematical modelling of mass fluxes during osmotic dehydration process gives invaluable information to have clearer understanding of dehydrated material composition and operational design. Azura et al. [2] recommended simpler empirical equation including parameters with physical meaning. This empirical equation has been used to model the rate of dehydration of different plant-based materials (Schmit et al. [6] and Mercali et al. [13]). However, in literature, the suitability of this equation to model the dehydration rate of aonla is scarce. Aonla is known for exceptionally high amount of ascorbic acid and is important fruit also highly valued among Indian medicines. Therefore, the present study was undertaken to investigate the effect of osmotic process parameters on mass transfer kinetics of aonla slices and to develop the mathematical model for water loss and solute gain in the form of Azura et al. [2].

II. MATERIALS AND METHODS

Fresh aonla fruits of variety Chakaiya (Neelam) were procured from an orchard of Department of Horticulture, Mahatma Phule Krishi Vidyapeeth, Rahuri (India). The aonla fruits were sorted for uniform size (32.5±1 mm), colour, maturity and physical damage; washed with potable water and then wiped with a muslin cloth to remove surface moisture. Fruits were blanched in boiling water for 5 min and cut to 5 mm thick uniform slices with specially designed radial type aonla cutter. Cut slices were separated with a sharp stainless steel knife and held in water until the entire batch was prepared to prevent enzymatic browning. Slices were then removed from the water and gently blotted with tissue paper prior to osmotic drying and determination of moisture content. Moisture content of the fresh as well as osmotically dehydrated aonla slices were determined by vacuum oven method. A pre weighed 3-5 g sample of aonla slices was kept in pre-dried and weighed petri-dishes. The petri-dishes with samples were placed in vacuum oven at 70°C maintaining vacuum between 85 to 100 mm of Hg till it attained constant weight. Petri-dishes were then cooled in desiccators for one hour and weighed. Average moisture content of three replicated samples was recorded (Ranganna [15]).

2.1 Preparation of sugar syrup as osmotic agent

Sugar syrups of three concentrations (50, 60 and 70°B) were prepared by dissolving known quantity of sugar in distilled water using glass rod as stirrer. Concentration of sugar syrup was checked by using hand refractometer (Erma, Japan make) of appropriate range (0-32, 28-62 and 58-92°B). Sugar was procured from local market and used as osmotic agent as it prevents food discolouration to a large extent and also imparts good taste to the final product.

2.2 Experimental procedure

In osmotic dehydration, a sample of aonla slices of 5 mm thickness each weighing 75 g were prepared. Constant syrup to fruit ratio (STFR) of 6:1 (v/w) was used. The 500 mL capacity glass beakers containing sugar syrup (50, 60 and 70°B) were placed inside the constant temperature circulatory water bath (Make: Classic Scientific India, Thane) at (40, 50 and 60°C) and slices were put into the syrup after attainment of desired temperature. Sodium metabisulphite (0.1%) was added to each beaker containing the syrup. For every 15, 30, 60, 90, 120,180 and 240 min interval one glass beaker was removed from the water bath and the aonla slices were immediately rinsed with distilled water to remove the solute adhered to fruit surface. Then, slices were spread on the tissue paper for 5 min to remove the surface moisture. The weight of osmotically dehydrated aonla slices was recorded. Slices were then put in pre-weighed petri-dish for moisture determination by vacuum oven method. Each treatment replicated thrice and average moisture content was recorded.
2.3 Osmotic dehydration parameters

Water loss and solute gain were calculated using the equations given by Pokharkar and Prasad [14]:

\[
WL = \frac{W_iX_i - W_0X_0}{W_i} \times 100 \quad \ldots (1)
\]

\[
SG = \frac{W_0(1 - X_0) - W_i(1 - X_i)}{W_i} \times 100 \quad \ldots (2)
\]

where,

- \(WL\) = Water loss (% or g per 100 g mass of sample)
- \(SG\) = Solute gain (% or g per 100 g mass of sample).
- \(W_0\) = Mass of slices after time \(\theta\), g
- \(W_i\) = Initial mass of slices, g
- \(X_0\) = Water content as a fraction of mass of slices at time \(\theta\).
- \(X_i\) = Water content as a fraction of initial mass of slices, fraction.

2.4 Modelling of mass transfer kinetics

Azuara et al. [2] developed a two-parameter model for water loss and solute gain, which was able to estimate the equilibrium point using data, obtained during a relatively short period of osmosis and was successfully used to predict the entire osmotic dehydration process up to the equilibrium point. Further, the model could characterize osmotic dehydration of different types of foodstuffs without restrictions of geometric considerations. Therefore, it was decided to develop the mathematical model for osmotic dehydration of aonla slices in the form of equation proposed by Azuara et al. [2] based on experimental mean data of three replications for water loss and solute gain.

2.4.1 Water Loss

A two-parameter equation developed by Azuara et al. [2] for water loss could be written as,

\[
WL_t = \frac{kt(WL_\infty)}{1 + kt} = \frac{t(WL_\infty)}{\frac{1}{k} + t} \quad \ldots (3)
\]

where,

- \(WL_t\) = The fraction of water lost by sample at any time \(t\) (%)
- \(k\) = The constant related to the rate of water diffusion out from the product
- \(t\) = Time (min)
- \(WL_\infty\) = The fraction of water lost by sample at equilibrium

During the osmotic dehydration of aonla slices, the constants \(k\) and water loss at equilibrium \((WL_\infty)\) could be determined by linear regression, using experimental data. The linear form is

\[
\frac{t}{WL_t} = \frac{1}{kWL_\infty} + \frac{t}{WL_\infty} \quad \ldots (4)
\]

2.4.2 Sugar gain

A two-parameter equation developed by Azuara et al. [2] for solute gain can be written as,

\[
SG_t = \frac{kt(SG_\infty)}{1 + kt} = \frac{t(SG_\infty)}{\frac{1}{k} + t} \quad \ldots (5)
\]

During the osmotic dehydration of aonla slices, the constants \(k\) and solute gain at equilibrium could be determined by linear regression, using experimental data obtained. The linear form is,
\[
\frac{t}{SG_t} = \frac{1}{k} \frac{t}{SG_\infty} + \frac{t}{SG_\infty} \quad \text{(linear form)} \quad \ldots \quad (6)
\]

where,

\(SG_t\) = Solute gain fraction at any time (\%)

\(k\) = The constant related to the rate of solute diffusion in the product

\(t\) = Time (min)

\(SG_\infty\) = Fraction of solute gain by the sample at equilibrium

Furthermore, as the time (\(t\)) becomes much greater than the values of \(1/k\) (i.e. \(t = \infty\)), the water loss or the solute gain (\(WL_t\) or \(SG_t\)) approaches equilibrium value \(WL_\infty\) or \(SG_\infty\) asymptotically. Thus, if the experimental plot of \(t/WL_t\) and \(t/SG_t\) respectively against \(t\) shows linearity, then the parameter values could be determined from the intercept and the slope. The model (Eq. 4 and 6) were used to predict the mass transfer kinetics.

1.5 Verification of model for osmotic mass transfer kinetics

Model was validated by fitting the experimental water loss ratio \((WL_t/WL_\infty)_{\text{expt.}}\) at different immersion times to the water loss ratio \((WL_t/WL_\infty)_{\text{pred.}}\) predicted by the proposed model. Model was assessed for goodness of fit on the basis of highest coefficient of determination (\(R^2\)), highest adjusted coefficient of determination (Adj. \(R^2\)), lowest chi-square statistic (\(\chi^2\)), root mean square error (RMSE), mean bias error (MBE) and per cent error modulus. According to Deng and Zhao [5] a model with per cent error modulus less than 10 % is considered acceptable. Similar procedure was adopted in validating the solute gain ratio.

III. RESULTS AND DISCUSSION

3.1 Effect of osmotic dehydration process parameters on water loss

Average initial moisture content of fresh aonla fruits of variety Chakaiya was found 86.587 (\% w. b.). Water loss and solute gain both were found in the range of 15.65 to 62.28 and 3.69 to 18.14 \%, respectively during 4 h osmosis.

Data of water loss and solute gain as influenced by various sugar syrup concentrations, sugar syrup temperatures (40, 50 and 60\(^\circ\)C) and immersion time (15, 30, 60, 90, 120, 180, and 240 min) during osmotic dehydration of aonla slices is plotted in Fig. 1 and 2. Water loss and solute gain increased non-linearly with immersion time at all concentrations (50, 60 and 70\(^\circ\)B). Similar trends were also observed for other temperatures (40, 50 and 60\(^\circ\)C). Water loss and solute gain were very fast at the beginning of the process and rate was gradually decreased with the increase of immersion time for all the treatment combinations. It can be further seen that as the immersion time increased, the water loss was increased; however, the equilibrium point could not be reached after short duration (4 h) of the osmotic dehydration process. This result is in confirmation with Lenart and Flink [12]; Jain et al. [10] and Alam and Singh [1].

This may be due to rapid water loss or solute uptake uptake near the surface in the beginning might have resulted in structural changes leading to compaction of these surface layers and increased mass transfer resistance for sugar uptake (Lenart and Flink [12]). Similar trends have been reported for other fruits and vegetables during osmosis by Sutar and Gupta [17] and Ertekin and Cakaloz [6].

3.2 Modelling of mass transfer on the basis of time

Inadequate control of product characteristics results in major difficulties regarding process modelling. Raw material variability is probably the most crucial source of deviations. Variety, maturity level, even cultivation procedures drastically affect the composition and barrier properties of a specific fruit or vegetable. The complex non-homogeneous structure of natural tissues seriously
complicates any effort to study and understand the mass transport mechanisms of several interacting counter flows (water, osmotic solutes, soluble product solids). Further several factors like solution concentration, temperature, immersion time, sample size and shape, solution to sample ratio and applied pre-treatments affect the kinetics of mass transport.

Figure 1 Effect of immersion time and sugar concentration on water loss (%) at 6:1 STFR during osmotic dehydration of aonla slices at a) 40; b) 50 and c) 60°C

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A two parameter equation based on mass balance developed by Azura et al. [2] was used to predict the dehydration kinetics during osmosis process and to determine final equilibrium point. This empirical equation have been used to model the water loss and solute gain obtained experimentally at temperatures (40, 50 and 60°C), concentrations (50, 60 and 70°B) and immersion time (15, 30, 60, 90, 120, 180 and 240 min) at STFR 6:1 (v/w) during osmotic dehydration. Mean data of the three replications was fitted by regression analysis to this equation using statistical package SAS 9.3. The representative experimental graphs of t/WLₜ and t/SGₜ as calculated from the water loss and solute gain for different sugar syrup concentrations at different temperatures were
plotted against immersion time as shown in Fig. 3 and 4 respectively. A linear trend was observed in all the cases. Therefore, linear regression fitted the straight line, the parameter values were determined from the intercept and the slope. Parameter values of $k$ and WL$_\infty$ for water loss kinetics and $k$ and $\Delta$ for solute gain kinetics were estimated from intercept and slope of the respective plots and presented in Table 1 and 2 including regression diagnostic criteria.

**Figure 3** Linear plots of Azura et al. model for determination of WL$_\infty$ and $k$ at different sugar syrup concentrations at 6:1 STFR during osmotic dehydration of aonla slices at a) 40; b) 50 and c) 60°C.
3.3 Predictions by proposed model

The proposed model was able to predict mass transfer kinetics up to the equilibrium point using data for relatively short period of osmosis. Model was validated by fitting the experimental water loss ratio (WL\text{exp}/WL_{\infty}) to different immersion time to the water loss ratio(WL\text{exp}/WL_{\infty})pred by the proposed model and goodness of fit was checked using regression diagnostic criteria. Similar procedure was adopted in validating the solute gain ratio.

Figure 4 Linear plots of Azura et al. model for determination of SG\text{\infty} and k at different sugar syrup concentrations at 6:1 STFR during osmotic dehydration of aonla slices at a) 40; b) 50 and c) 60°C
3.4 Evaluation and validation of model for water loss

Model parameters k and WL∞ and criteria for goodness of fit are presented in Table 1. The value of constant k is function of immersion time and rate of water loss. Value of k was increased with increase in concentration at constant temperature except for few treatments hence did not show any specific trend w.r.t. temperature and concentration. Equilibrium water loss (WL∞) was increased with increase in temperature at constant concentration and also with increase in concentration at constant temperature. Estimated equilibrium water loss varied from 47.70 to 66.91% for different temperature-concentration combinations. Further, high R² (0.993 to 0.998), high adjusted R² (0.991 to 0.998), lowest probability (4x10⁻⁸ to 1x10⁻⁶), lowest χ² (0.013 to 0.046), lowest RMSE (0.0566 to 0.0), MBE (3.2 x10⁻¹⁷ to 2.2 x10⁻¹⁶ ) and per cent error modulus (1.0243 to 2.2486) between experimental and predicted values were found suggesting Azura et al. [2] model fulfilled regression diagnostics criteria for all the treatment combinations. Per cent error modulus between experimental and predicted value is less than 10 per cent also revealed the model could be used successfully to predict the entire osmotic dehydration process up to the equilibrium point for water loss.

Table 1 Azura et al. model parameters and goodness of fit for water loss

<table>
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<tr>
<th>Temp (°C)</th>
<th>Conc. (°B)</th>
<th>k</th>
<th>WL∞ (%)</th>
<th>R²</th>
<th>Adj R²</th>
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3.5 Evaluation and validation of model for solute gain

Equation parameters k and and regression diagnostic criteria for goodness of fit are presented in Table 2. The value of constant k is function of immersion time and rate of solid gain. Value of k was increased with increase in concentration at constant temperature for all the treatments except for treatment (50°C -70°B). Equilibrium solid gain (SG∞) was increased with increase in concentration at constant temperature. Predicted equilibrium solid gain varied from 14.57 to 18.75% for different temperature-concentration combinations. Further, high R² (0.981 to 0.997), high adjusted R² (0.977 to 0.997) , lowest probability (1x10⁻⁸ to 2x10⁻³), lowest χ² (0.088 to 0.604), lowest RMSE (0.2806 to 0.8067), MBE (8.9 x10⁻¹⁶ to 1.1 x10⁻¹⁵) and per cent error modulus (0.0071 to 7.1914) between experimental and predicted values were found suggesting Azura et al. [2] model fulfilled regression diagnostics criteria for all the treatment combinations (Table 2). Per cent error modulus between experimental and predicted values is less than 10 per cent also revealed that the model could be used successfully to predict the entire osmotic dehydration process up to the equilibrium point for solute gain. Similar findings for modelling were reported by Sutar and Gupta [17] for onion slices; Ganjloo et al. [7] for seedless guava cubes and Kaur and Singh [11] for beetroot slices.
Table 2 Azura et al. model parameters and goodness of fit for solute gain

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IV. CONCLUSION

Water loss from and solute gain by aonla slices increased non-linearly with duration of osmosis at all concentrations and both increased at faster rate in the initial period of osmosis than later stage. Water loss and solute gain both increased with increasing sugar concentration and temperature. They can lose 15.65 to 62.28 % water and gain 3.69 to 18.14 % sugar depending upon the sugar syrup concentration (50-70°Brix) and temperature (40-60°C) in 4 h duration of osmosis.

The two parameter model developed by Azura et al. can describe the mass transfer kinetics in the osmotic dehydration process of aonla slices at any time satisfactorily when other conditions of osmosis are kept constant. Model can predict the equilibrium points on the basis of short duration of osmosis without conduction of experiments for several hours.

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