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Fractional Calculus Method Applied to the Candying of Apple Rings in an Osmotic Drying Process

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ABSTRACT

The effective diffusivities and shrinkage of water and solid were correlated for finite hollow cylinder-shaped apple samples during the candying operation in the osmotic solution. Experiments were conducted in the sucrose solution as an osmotic agent at different temperatures (i.e., 40, 50, and 60 °C) and at a constant concentration of 55 °Brix. The effective diffusivities of water and solid were calculated by fitting the water loss and solid uptake experimental data to Fick's second law and the fractional calculus method, considering the shrinkage of the samples during the candying process. The obtained results exhibited that the volume of the apples reduced linearly by increasing the water loss. For above conditions of the candying process, the effective diffusivities of water with Fick second law were determined in the range of 3.7×10^{-10} m^2/s–8.73×10^{-10} m^2/s*, and those with fractional calculus method were in the range of* 2.75×10^{-10} m^2/s–6.98×10^{-10} m^2/s*. The results indicated that the coefficient of determination for the fractional calculus method was more than the coefficient of determination for the Fick model. The value of the empirical parameter* α *for the Non-Fickian diffusion model was always higher than unity, meaning that the dehydration process had a super-diffusive mechanism.*

1. Introduction

The high water activity of the agricultural products, especially fruits, is the primary reason for their losses [1, 2]. Applying the candying process on fruits yields a significant decrease in water activity by osmotic dehydration [3, 4]. It is an intermediate drying procedure to achieve a partial dehydration level. In this process, food materials are submerged in a hypertonic solution for a determined period of time. One of the characteristics of hypertonic solutions is having high osmotic pressure and a water activity lower than that of fresh fruits and vegetables. The candying has numerous benefits in comparison to other drying methods including the elimination of unbounded water through semipermeable food structure without phase change with the lowest rate of energy [5, 6], as well as

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improving the organoleptic characteristics of the product [7, 8]. Sucrose and glucose are the most common osmotic agents in the osmotic dehydration of fruits [3]. Candied fruits can be added to food, e.g., bakery, deserts, and confectionery products [9].

A mathematical modeling is widely utilized in the food industry to simulate the mass and heat transfer during the food drying [2]. In the osmotic dehydration process, the mass transfer phenomena is an inherently slow operation, and the heat transfer is much faster than the mass transfer. Therefore, the coupled heat and mass transfer modelings are not necessary for the mathematical modeling of the osmotic dehydration [10]. During the osmotic dehydration in the solids, the mass transfer process is modeled using the Fickian diffusion equation [11]. The most important parameter of this model to be determined is the effective diffusion coefficient [12, 13]. Most of the studies on the mathematical modeling of the osmotic dehydration procedure are based on comparing the ability of different experiential models with investigational data through a non-linear regression analysis [14, 15].

In some cases, the Fick's second law could not well represent the osmotic dehydration process since there were numerous phenomena that were not adapted to models based on Fick's second law. Both shrinkage and volume changes may happen due to the moisture transportation much slower or faster than that predicted by Fick's second law, known as the non-Fickian or anomalous diffusion. The reduction of the external volume is a common physical change during the drying of food materials. Inside the cellular structure of the food materials, both water loss and heating rate cause changes in the primary shape of the solids, which reduce the dimension of samples [16]. Vegetables and fruits contain high moisture contents and are prone to alteration of the original shape because of the considerable shrinkage during the candying operation [17].

A useful mathematical approach based on fractional calculus was proposed by Simpson et al. and could be used to demonstrate a phenomenological representation of the dehydration processes based on anomalous diffusion [18]. The fractional calculus model which can be applied to the diffusional process included a key factor called "fractional order" (α) defining that the drying process has a sub-diffusive ($0<\alpha<1$) or super-diffusive mechanism ($\alpha>1$) as compared with Fick's second law ($\alpha = 1$). Simpson and co-workers and Ramírez and co-workers showed the value of α was related to the microstructural characteristics of the solid materials [16, 19]. Nevertheless, the models proved that using the Fick's equation had their limitation in food materials because several assumptions of Fick's equation would not fit the heterogeneous tissues [20]. These limitations are induced by the volume changes, cell structure, and shrinkage of plant tissues. In this respect, it is recognized that fruits like apple have considerable structural alterations over the osmotic dehydration. Non-Fickian or anomalous diffusion has been demonstrated as a beneficial instrument for quantitatively explaining diffusion over the osmotic dehydration of foods since it takes into account numerous variations in the food over the procedure, such as porosity and shrinkage.

The present research has aimed at proposing and evaluating the determination of the diffusion mechanisms in the apple rings exposed to the blanching pre-treatment by applying the fractional diffusional formulation via the fractional calculus instrument. Indeed, the main goal is to predict the changes of moisture, the solid gain, and the diffusion coefficient inside a two-dimensional hollow cylinder apple with a finite radius and length as well as evaluating the experimental data based on Fick's second law and FC. Finally, it is tried to measure the effective diffusion coefficient values based on the experimental results and show the error of the models.

2. Theory

2.1. Fickian's diffusion model

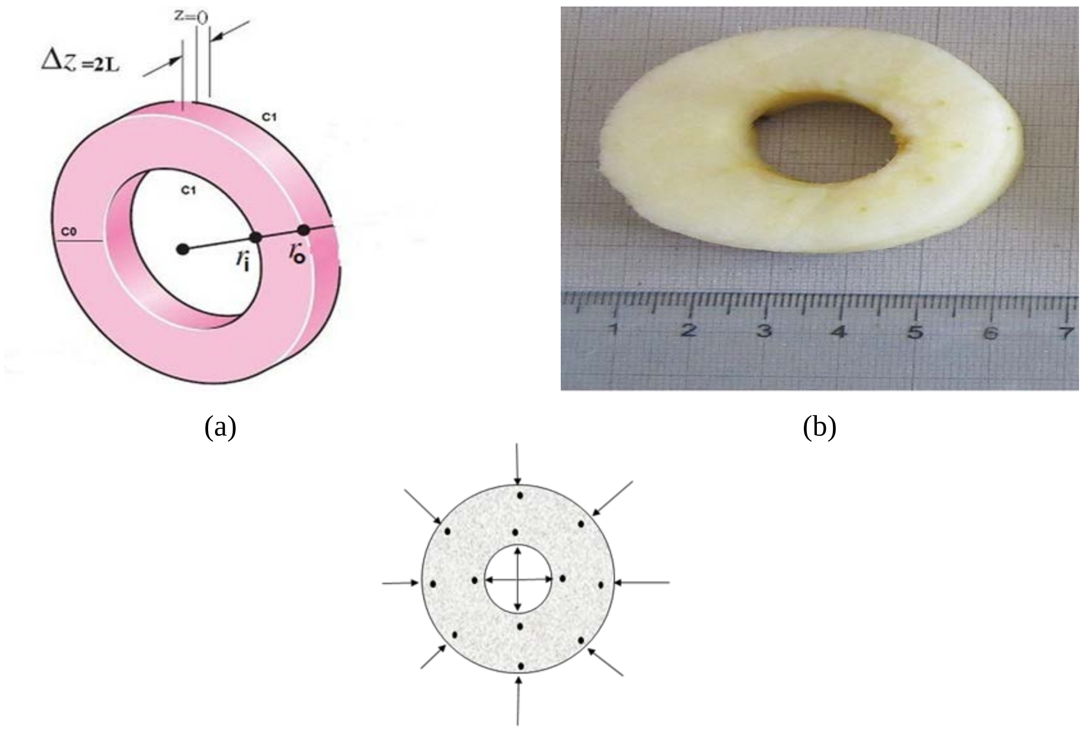
Fig. 1(a) schematically demonstrates a two-dimensional view of a typical ring-shaped apple with the inner radius of r_i, outer radius of r_o, and thickness of 2L.

Several apple rings were immersed in an infinite volume of an osmotic solution, and the solution was stirred continuously as well. Due to the well mixing of the osmotic solution, the variable time would be the only factor affecting the concentration of the solute in the solution.

The two-dimensional mass transfer at an unsteady state for a cylinder with the finite radius of R and the length of 2L, and immersed in an osmotic solution with a specified concentration, can be explained as [21]:

$$\frac{\partial C}{\partial t} = D\left(\frac{\partial^2 C}{\partial r^2}+\frac{1}{r}\frac{\partial C}{\partial r}+\frac{\partial^2 C}{\partial z^2}\right) \quad (1)$$

The shape of the samples was the hollow cylinder, as shown in Fig. 1(b).



(a) (b)

(c)

Figure 1. a) Schematic of apple ring in an osmotic solution, b) The shape of the apple hollow cylinder in an osmotic solution, c) Directions and locations for the measurement of thickness, outer and inner diameters.

The following assumptions were taken for solving the problem:

- The initial water and solid concentration are evenly dispersed.
- The procedure is isothermal.
- The diffusion coefficient is supposed to

be constant.

- The concentration of the osmotic solution is considered constant within the procedure because the apple-to-solution mass ratio is less than 1:20 (w/w).
- The diffusion of sugar to the fruit and the diffusion of water from the fruit are the only applied assumptions.
- Shrinkage and other transfer mechanisms of the specimen are ignored.

The primary and boundary conditions include:

$$C(r,z,0)=C_0 \ , \ \begin{cases} r_i \le r \le r_o \\ -L \le z \le L \end{cases} \quad (2)$$

$$\begin{aligned} C(r,-L,t) &= C_1 \\ C(r,L,t) &= C_1 \end{aligned} \quad (3)$$

$$\begin{aligned} C(r_i,z,t) &= C_1 \\ C(r_o,z,t) &= C_1 \end{aligned} \quad (4)$$

where C_1 (g/cm^3) is the concentration in the apple surface in equilibrium with the osmotic solution, C_0 (g/cm^3) is the concentration at the initial moment, C (g/cm^3) is the concentration in time t, D (m^2/s) is the effective diffusion coefficient in the apple texture, L is half of the sample's thickness, r_i and r_o are inner and outer diameters of the cylinder in meters respectively. Analytically solving the limited hollow cylinder equation by the separation of variables method leads to the formation of two equations for the infinite plane and infinite-length cylinder. The final solution is obtained by multiplying the solutions of the infinite plane by the infinite hollow cylinder equations [21-23].

Hence, the analytical solution to Fick's equation for water or solid diffusion in two dimensions is obtained by Eq. (5).

$$MR(t) \text{ or } SR(t) = \frac{32}{\pi^2\left(r_0^2-r_i^2\right)} \sum_{n=1}^{\infty} \frac{J_0(r_i\alpha_n)-J_0(r_0\alpha_n)}{\alpha_n^2[J_0(r_i\alpha_n)+J_0(r_0\alpha_n)]} \times \sum_{m=1}^{\infty} \frac{1}{(2m+1)^2} \exp\left(-D_W(\alpha_n^2+(2m+1)^2\frac{\pi^2}{4L^2}t\right) \quad (5)$$

where $J_n(0)$ and $Y_n(0)$ represent Bessel functions of the first and second types with the index is n, and k_i is the positive roots of the transcendental equation:

$$J_0(a\alpha_n)Y_0(b\alpha_n)-J_0(b\alpha_n)Y_0(a\alpha_n)=0 \quad (6)$$

2.2. Anomalous diffusion model

As described before, first, we take into account the time-fractional radial diffusion in a finite hollow cylinder of inner and outer radii, i.e. r_i and r_o respectively (where $r_o \gg r_i$) and the thickness of L. The primary distribution of concentration is C_0. Also, we consider that the boundary condition at the outer and inner surface is kept at the constant concentration of C_1.

$$\frac{\partial^\alpha C}{\partial t^\alpha} = D\left(\frac{\partial^2 C}{\partial r^2}+\frac{1}{r}\frac{\partial C}{\partial r}+\frac{\partial^2 C}{\partial z^2}\right) \quad (7)$$

The primary and boundary conditions are expressed by Eq.(2) -(4). So, the diffusion equation in the finite cylinder geometry is given by:

$$MR(t) \text{ or } SR(t) = \frac{32}{\pi^2\left(r_0^2-r_i^2\right)} \sum_{n=1}^{\infty} \frac{J_0(r_ik_n)-J_0(r_0k_n)}{k_n^2[J_0(r_ik_n)+J_0(r_0k_n)]} \times \sum_{m=1}^{\infty} \frac{1}{(2m+1)^2} E_\alpha\left(-D(k_n^2+(2m+1)^2\frac{\pi^2}{4L^2}t^\alpha\right) \quad (8)$$

The solution of this equation is given in [24], where E_α is given by Eq.(9).

$$E_\alpha(x) = \sum_{n=0}^{\infty} \frac{x^k}{\Gamma(1+\alpha \times n)} \quad (9)$$

where E_α stands for the Mittag-Leffler function [25].

If $\alpha = 1$, then the fractional equation model changes to a Fickian model according to Crank, indicating that the central anomalous diffusion equation is the central Fick's diffusion equation [18].

2.3. Evaluation of shrinkage

The comparative evaluations of the thickness (L), outer diameter (d_o) and inner diameter (d_i) of apple rings during the osmotic dehydration was performed by the Vernier caliper (least count of 0.05 mm). The measurement locations on the apple ring were as follows: twelve-located thicknesses, two-located inner diameters, and four-located outer diameters of an apple ring, as shown in Fig. 1(c) and were computed on the mean. The sample volume was calculated at each time using Eq. (10).

$$V = \frac{\pi}{4}(d_0{}^2 - d_i{}^2)L \quad (10)$$

where V and L are the volume and thickness of the sample respectively. r_i and r_o are the inner and outer diameters of the cylinder in meters.

3. Materials and methods

3.1. Materials

Raw materials include sucrose bought from a local market, sodium metabisulfite bought from Pars Sulfite Chemical Inc., and the yellow apple of Urmia bought from a local market in Urmia.

3.2. Preparation of the solutions and samples

This study has been conducted on fresh golden apples (cv. Golden Delicious) purchased from a local grocery store in Urmia, Iran, and kept under refrigeration at 5 °C before the experimentation. The initial humidity of apples, defined by drying the specimens in the oven, was about 85 %. The size and color (yellow) of the apples were the same. The apples were peeled and cut into hollow cylinders by slicer and a knife (the outer diameter: 5 cm; the inner diameter: 2 cm; and the thickness: 2 cm). Before starting the osmotic dehydration process, several pre-treatments with specific benefits to the osmotic dehydration were performed. Apples were subsequently blanched in a citric acid solution and then immersed in a sodium metabisulfite solution to avoid fruits' enzymatic browning. The results of a study showed that blanching before drying the vegetables and fruits would affect the inhibition of discoloration [26]. Then, the sugar solution was prepared by diluting.

3.3. Experimental procedure

The candying process was performed on apple hollow cylinders at the concentration of 55 °Brix and osmotic solution temperatures of 40, 50, and 60 °C. After blanching the apples, the specimens were eliminated from the solution, dried with an absorbent paper, and weighed. The containers were placed on a stirrer to maintain its temperature. The magnet stirrers were used to homogenize the solution. Afterward, the samples were removed from solutions in the predetermined times (within the first 6 hours every 30 min, and then every 8 h until the end) and then was rinsed, weighed, and dried. Then, the apple cylinder samples were put in the oven with a constant temperature at 100 °C for determining their solid material. The osmotic dehydration tests were performed for over 3 days at various temperatures in stirred solutions. The solution-to- apple mass ratio was more than 20:1 (w/w). The mass transfer was performed to determine the solids gain (SG) and water loss (WL). The water loss and solid gain based on the initial fruit weight during the osmotic dehydration was evaluated according to the following equations:

$$WL_t = \frac{M_0 - S_0 + S_t - M_t}{M_0} \quad (11)$$

$$SG_t = \frac{S_t - S_0}{M_0} \quad (12)$$

where WL_t, SG_t, M_t and S_t are respectively the water loss, solid gain, fruit weight, and the weight of solids in the fruit at any time. M_0(g) and S_0(g) are the initial corresponding quantities of fruit weight and solid weight in the fruit. The equilibrium values of solid gain and water loss were determined by Azuara equations [27].

3.4. Analytical determinations

Analytical determinations of moisture and soluble solids were defined by drying the specimens for 24 h at 100 °C in an oven to a fixed weight based on the technique that was explained by the Association of Official Analytical Chemists [28] for fruits like apple. The water activity (a_w) of the apple samples was measured with a psycrometer (Novasina Lab Swift-aw, Lachen, Switzerland) at room temperature, by placing approximately 5 g of candied apple in a measuring container. The measurements were carried out in triplicates, and the average values were reported.

4. Results and discussion

4.1. Performance of osmotic drying process

Fig. 2 shows the water activity of samples at a constant osmotic solution of 55 °Brix and different dehydration temperatures, which was measured at the end of the dehydration process. As shown, the temperature of the osmotic solution during the dehydration process significantly influenced the moisture content of apples. The aw of the initial apple rings was 0.99, while the aw of the candied apple rings were 0.87, 0.84, and 0.78 for dehydration temperatures of 40, 50 and 60 °C respectively. Therefore, the aw of the candied samples was reduces by decreasing their final moisture content. As reported by Nieto and co-workers [29], a small reduction of aw creates a remarkable reduction in the microbial growth rate as well as the relative rate of deteriorative reactions. The obtained results of the current study are consistent with Assis et al.[30] research, who reported that the value of aw of the apple samples was reduced after the dehydration process. As a result, the temperature of the dehydration process also affected the rate of moisture removal and, the rate of aw reduction promoted by increasing the temperature [30].

In this study, the changes in the inner and outer diameter, thickness, and volume in different WL have been investigated. Shrinkage is one of the ordinary physical phenomena in the candying process. Therefore, in the mathematical modeling of the dehydration of the fruit, shrinkage should be considered for the calculation of the diffusion coefficient. Fig. 3 shows the experimental data and an empirical model presented for predicting the fruit shrinkage behavior in the candying process. As it can be seen, there is a linear relationship between shrinkage dimensions and the water loss, which consists of the findings by Moreira et al. and Souraki et al. [10, 31].

The variations of the sugar uptake and water immigration in apple samples are provided in

Fig. 4. As it can be seen, the solid uptake and water immigration increase nonlinearly with time for all the specimens which may be related to the increment of the diffusion coefficient with increasing the temperature. The rates of the water immigration and solid uptake (i.e., slopes of water loss and solid gain curves versus time) are relatively high in the first 120 min of the candying process and then decline. The maximum water loss was obtained at the higher dehydration temperature. Also, the enhancement of the water loss leads to an increase in the osmotic agent concentration inside the samples and, the solid gain was increased as the osmotic solution temperature increased, too. Souraki et al. [10] reported a similar result that the water loss and solid gain increased by increasing the solution temperature because of increasing the effective diffusion of the osmotic agent in the apple texture. As reported by Cichowska et al., the increase in temperature was followed by a decrease in solution viscosity, swelling, and damage to the cellular membrane structure of the fruit [32].

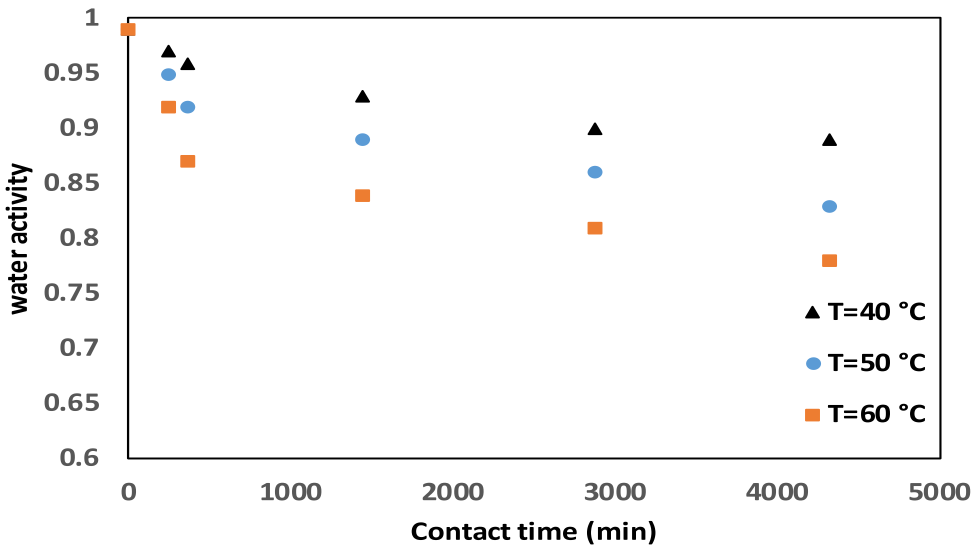
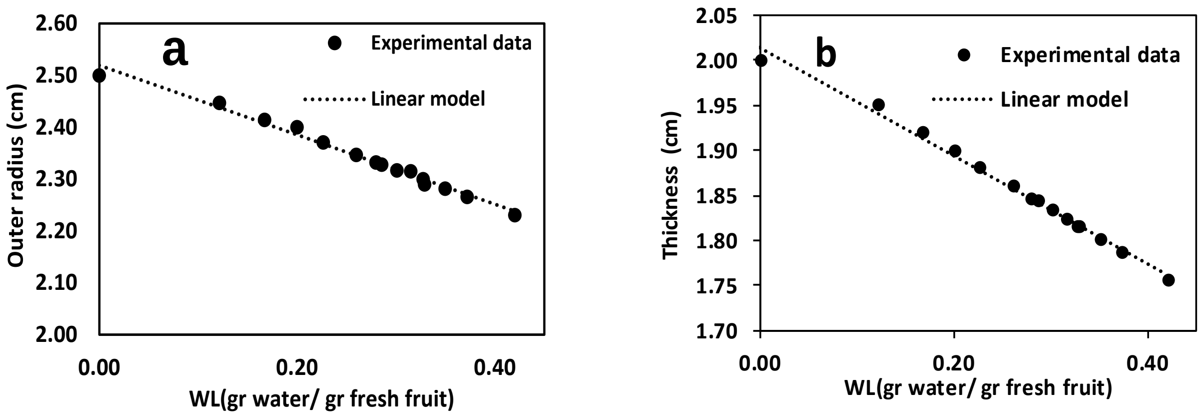


Figure 2. Relationship between the apple rings water activity and the contact time at various temperatures.



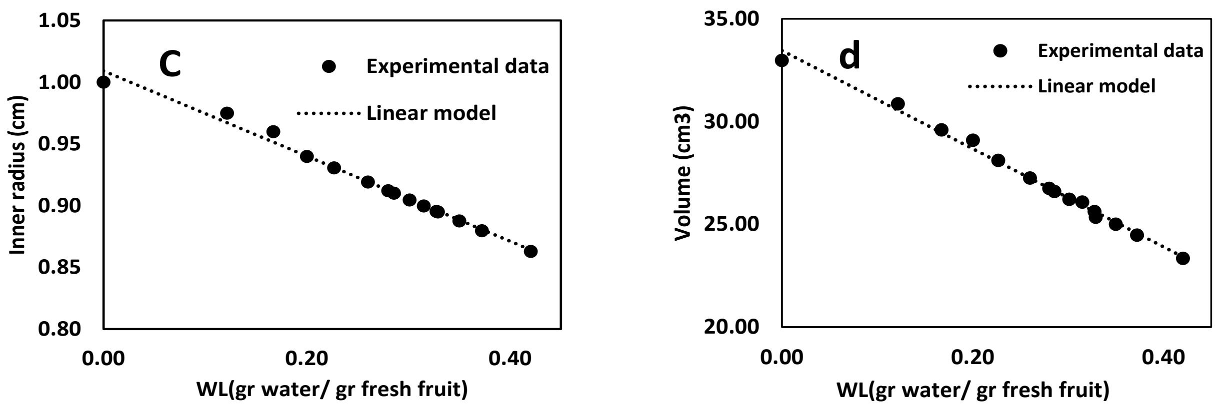


Figure 3. Experimental and linear model predicted values of shrinkage for the candying of apple rings at a concentration of 55 °Brix and 60 °C, a) outer radius, b) thickness, c) inner radius, d) volume (d).

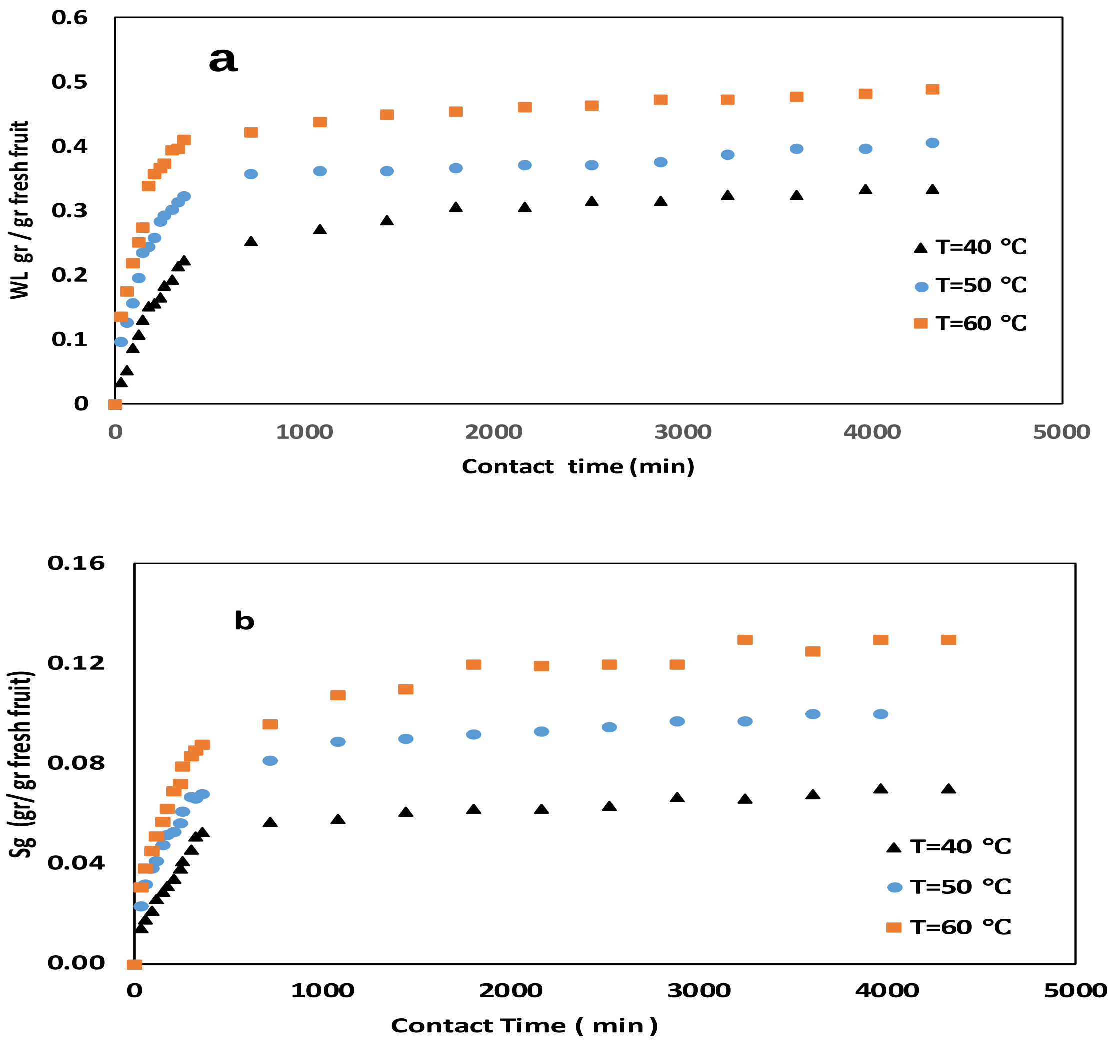


Figure 2. The influence of the solution temperature in the constant sucrose concentration of 55 °Brix. a) water loss, b) solid uptake.

4.2. Mathematical modeling

The Microsoft Excel Solver tool 2013 was utilized to fit the Fick's equation and the fractional calculus approach to the experimental data. The effective diffusion coefficient was estimated for various temperatures. Considering Eq. 8, the left side of this equation is known (experimental values at any time); however, the right side is unknown because the values of α and D are undefined. Therefore, initial values for α and D are estimated, and the difference of right and left side values is considered as an error. Using the Solver option of Microsoft Excel, the values of α and D are continuously changed until the sum of squares of errors is minimized. Finally, the values of α and D in the last step are reported.

Table 1 demonstrates the obtained values of the diffusion coefficients. Any increase in the temperature caused a corresponding increase in the effective diffusion coefficients of water and solid. According to Table 1, the lowest and highest amounts of the solid diffusivity were observed in samples submerged in the osmotic solution with temperatures of 40 and 60 °C respectively. Increasing the temperature from 40 to 60 °C, resulted in increasing the diffusion coefficient, probably because of the shrinkage phenomena that would alter the apple structure. Fig. 5 depicts the fitting of Fick's law and the fractional calculus approach for each group of experimental data. The experiments were carried out at an osmotic concentration of 55 °Brix and various temperatures. According to Fig. 5, the experimental data are not fitted with the diffusion model from Fick's equation as well. These changes can be described and justified by the damage and shrinkage of the cellular membrane structure of the fruit. Since Fick's theory is not satisfactory for treatments under a long time, it was attempted to provide an anomalous diffusion model. Fig. 5 represents a fitted- fractional model for each group of experimental data utilizing an osmotic solution of 55 °Brix. Moreover, it was found that the time-fractional order represents the super-diffusive existence of the anomalous diffusion procedure, with fractional orders larger than one for all actions. This fact shows that the effective diffusion coefficient is underestimated by Fick's second law, and the process can take less time than the time predicted by Fick's theory (Fig. 5). For instance, in Fig. 5b, in order to attain MR = 0.20, 2880 min of the process time was predicted using the anomalous calculus diffusion model, while with the Fick model, it would not reach the desired value. Table 1 represents the coefficient of determination (R^2) for both models. As it can be noticed, these values are more significant for the calculus-fractional model in all treatments, indicating the non-Fickian diffusion model explains the water diffusion process in apple rings as well.

Table 1

Effective water and solid diffusivities in apple rings.

	Fickian model				Fractional model					
T (°C)	**D_w×10^{10} (m^2/s)**	**R^2**	**D_s×10^{10} (m^2/s)**	**R^2**	**D_w×10^{10} (m^2/s)**	**α**	**R^2**	**D_s×10^{10} (m^2/s)**	**α**	**R^2**
40	3.71	0.89	2.47	0.92	2.75	1.28	0.96	1.94	1.25	0.95
50	5.53	0.84	3.04	0.88	4.65	1.36	0.94	2.63	1.36	0.92
60	8.73	0.78	4.14	0.80	6.98	1.51	0.91	5.61	1.45	0.89

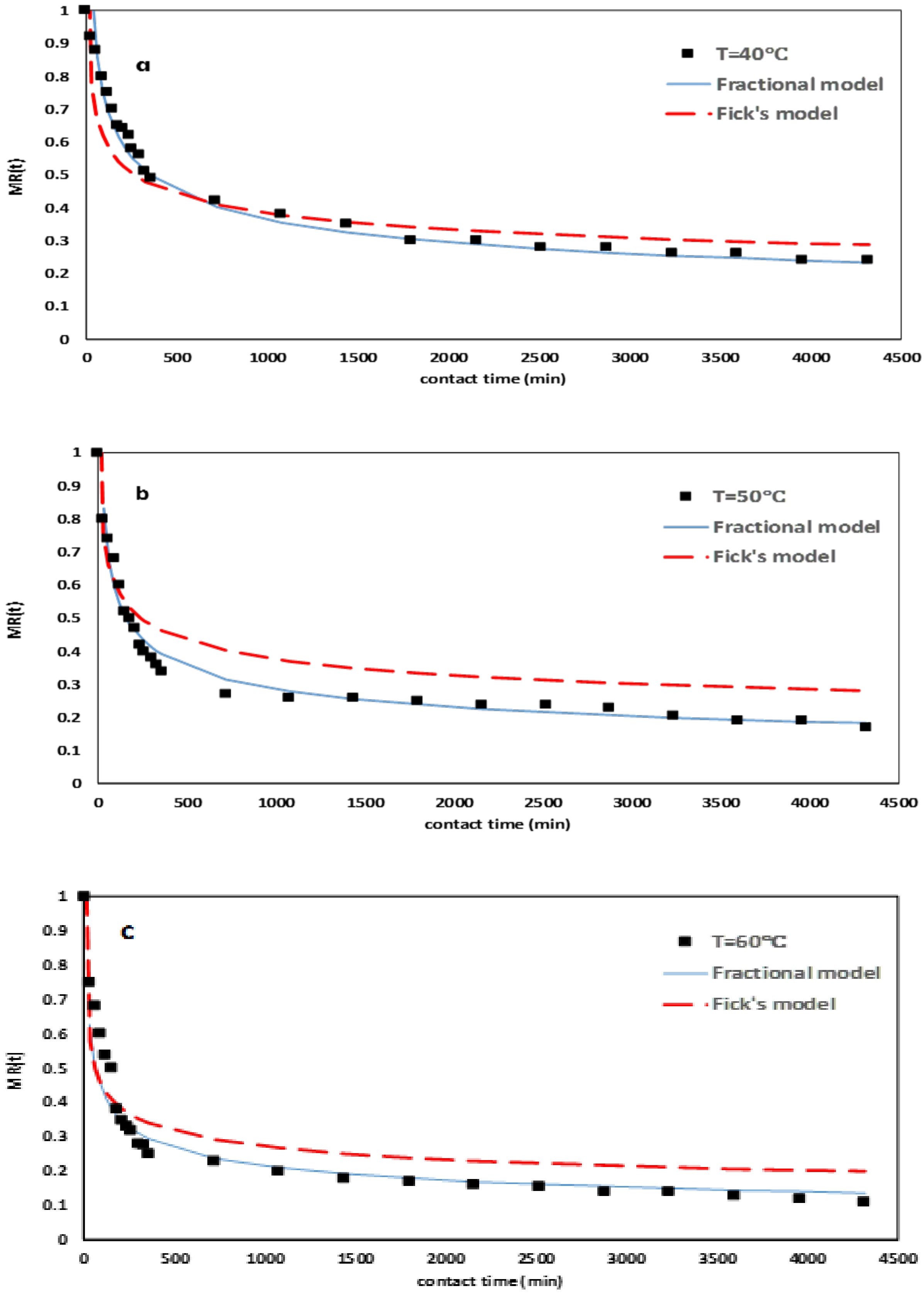


Figure 3. The anomalous diffusion and Fick's models for an osmotic solution of 55 °Brix and various temperatures.

5. Conclusions

In this study, the diffusion mechanism in the apple rings exposed to blanching pre-treatment has been determined by applying the fractional diffusion equation. The obtained mathematical model was subsequently confirmed by the experimental data. Analyzing the experimental results showed that the volumetric shrinkage of apple could be presented as a linear function of water loss. The water activity of samples was reduced as the dehydration temperature was increased. Also, the experimental and predicted diffusion coefficients were obtained in the order of 10^{-10}. The Non-Fickian dehydration process follows the super-diffusive mechanism based on the α value, with the exception of α=1 case. The results of the modeling with the Fractional calculus method and Fick's law were compared with the experimental results and, the results showed that the fractional calculus model (R^2>0.90) fitted better than Fick's law (R^2<0.90). In general, it has been concluded that the fractional calculus method is a good way to predict the candying process of fruit in the food industry.

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Nomenclature

WL	dimensionless water loss [gr water/gr fresh fruit].
SG	dimensionless solid gain [gr solid/gr fresh fruit].
S	mass of sucrose [g].
M	mass of fruit [g].
D	effective diffusivity [m^2/s].
C	concentration (g/cm^3 of fruit].
t	contact time [s].
L	half of the thickness in the z-direction [m].
r_i	inner diameters [m].
r_o	outer diameters [m].
r	the radius of the cylinder [m].
E_a	Mittag-Leffler function.
J_0	Bessel function of the first kind order 0.
Y_0	Bessel function of the second kind order 0.
t	at time t.
s	solid.
w	water.
0	initial value.
∞	equilibrium condition.
α_n	the nth positive roots of the Eq. (6).

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