

Research Article

Mass Transfer during Osmotic Dehydration of Tunisian Pomegranate Seeds and Effect of Blanching Pretreatment

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In this work, the osmotic dehydration (OD) of Tunisian pomegranate seeds of “El Gabsi” variety was investigated. To optimize the process operating conditions, the effect of temperature, hypertonic solution solid content, and stirring speed was studied. The best conditions resulting in the higher water loss and the minimum of fruit damages found are 40°C, 50°Bx, and 440 rpm. In these conditions, the effect of blanching pretreatments on the solute and water transfer kinetics during the OD was investigated. The blanching pretreatments were carried out using two methods: blanching in a boiling water bath and in a microwave oven. The mass diffusion kinetic depends on time, temperature, hypertonic solution solid content, stirring speeds, and pretreatment process. Peleg’s model showed a good fit to the experimental data. By applying blanching pretreatments, the water and solute effective diffusivities passed from the order of 10^{-9} to the order of 10^{-8} , and the OD equilibrium time was significantly reduced.

1. Introduction

Pomegranate (*Punica granatum* L.) is one of the oldest known edible fruits belonging to the family Punicaceae [1]. This fruit is frequently grown in arid and semiarid areas, especially in parts of Asia, North Africa, around the Mediterranean, and in the Middle East [2, 3]. Tunisia is one of the main producers in the Mediterranean region, and its production is mainly located in Gabes in southeastern Tunisia. The El-Gabsi variety represents about 35% of the annual Tunisian pomegranate production [4].

The pomegranate fruit is an important source of beneficial bioactive and nutritive compounds for human beings. It is rich in organic acids, minerals (such as potassium), vitamins (C, A, and K) [2, 5], and phenolic compounds such as hydrolysable tannins, condensed tannins, and phenolic acids [1,6–9]. Particularly, the edible pomegranate part contains sugars, organic acids, anthocyanins, minerals, proteins, unsaturated fatty acids, polysaccharides, and vitamins [10, 11].

The rich composition of this fruit gives it hypolipidemic, antioxidant, antiviral, antineoplastic, anticancer, antibacterial, antidiabetic, antidiarrheal, helminthic, vascular, and digestive

protection properties. Due to its immense potential for health benefits, pomegranate has achieved the title of “superfood” [12], and it has been used in traditional medicine over centuries [6].

Thus, the conservation of the edible part of pomegranate is of great interest. The traditional conservation methods affect seriously the fruit’s quality. OD reduces the product water activity by maintaining its sensory and nutritional characteristics. Water activity reduction slows down deteriorative reactions and increases microbial stability, thus prolonging the fruit shelf life [13]. OD allows partial water removal from cellular tissue by immersion in a concentrated aqueous solution [14]. The driving force for water removal is the difference in osmotic pressure between the fruit and the hypertonic solution [15]. The water removal from the product is always accompanied by a simultaneous counter diffusion of solutes from the osmotic solution into the tissue [16]. The cell membrane, the exchange surface between the osmotic solution and the product, exerts high resistances to mass transfer and slows the OD rate [17]. For this reason, the mass transfer can be supported by combining the DO with pulsed vacuum [18, 19], pulsed electric field [20–22],

ultrasound [14, 23], freezing [24, 25], centrifugation [20, 26], and blanching [13, 17].

The benefits of such pretreatment processes have been widely reported in the literature. Corrêa et al. [27] affirmed that the application of a vacuum pulse on tomato slices is strongly recommended for reducing NaCl incorporation in osmotic processes with ternary solutions. Also, the centrifugal force combined with the OD of carambola slices gives higher water loss and less solid gain [28]. However, pretreating kiwi slices with ultrasound more than 10 min causes the formation of microchannels through the membranes which improve the mass exchange by OD [14]. Similarly, freezing pretreatment was used with several fruits to improve the OD kinetics. It causes the formation of cracks on the cell membrane which facilitate both solute and water transfer. Some of frozen pretreated products are as follows: pomegranate seeds [25], pumpkin [17], tomatoes [24], and mango [29]. The pulsed electric field was also used as a pretreatment or simultaneously with OD to accelerate the mass transfer. Using this pretreatment, the cell membrane seems not to be affected. It was applied on apple slices [30], on carrot tissue [20], and on bell peppers [22].

Blanching is an alternative pretreatment for OD. It is a heat treatment process that can be carried out either by the immersion of the samples in a hot solution bath [17, 27], by a hot steam exposure, or by ohmic heating in a microwave [13] for a short time (a few minutes) in a temperature range of 85°C to 100°C [4]. This pretreatment increases the cell membrane permeability and removes the gas occluded in plant tissues [28]. Thereafter, the mass transfer becomes faster during the further processing [13]. Thus, the main objective of the present study is to accelerate the mass transfer during the OD of “El Gabsi” pomegranate seeds in a sucrose solution. So, blanching was used as a pretreatment, and its effects on the solute and water transfer were investigated.

2. Materials and Methods

2.1. Sample and Solution Preparation. Fresh pomegranate fruits (*Punica granatum* L.) at full ripeness, from the same region with homogeneous size were bought at a local market in Gabes, south of Tunisia. The fruits were cleaned with wet paper, wiped very well with blotting paper, and then stored at 5°C until use.

Sucrose solutions of 30°Bx, 40°Bx, 50°Bx, and 60°Bx, were used in the OD. It was prepared by dissolving analytical grade D(+)-sucrose crystals supplied by Carlo Erba Reagents Laboratory, France, in distilled water with a solute/water ratio of 1/2. The solid content of the solution was verified before using.

2.2. Blanching Pretreatment. The pomegranate fruit was hand peeled, and the blanching pretreatments prior to OD were performed in two different ways:

- (i) Microwave blanching: the pomegranate seeds were put in a sucrose solution (50°Bx) with a sample/solution ratio of 1/4. The mixture was put in a microwave at 600 W for one minute.

- (ii) Boiling water bath blanching: the pomegranate seeds were immersed in a water bath at 92°C during one minute and were wiped very well with blotting paper.

2.3. Osmotic Dehydration. Blanched and unblanched pomegranate seed samples of “El Gabsi” variety were submitted to OD. OD was carried out in batch mode in a sucrose solution with a magnetic stirring of 440 rpm. The sample to solution ratio was 1/4. During OD, samples were withdrawn at regular intervals (30 min), quickly rinsed with distilled water to remove osmotic solution from the surface, drained over by absorbent paper to eliminate excess of water, weighed, and oven-dried as described in Section 2.4 to determine their dry matter. The OD kinetics was monitored for seven hours.

2.4. Parameter Measurement. The dry matter of samples was determined by stove-drying at 105°C until reaching a stable weight.

The solid content was directly measured using a refractometer model Sopenem 3127. The zero of the refractometer was adjusted using distilled water.

2.5. Theoretical Consideration. Equations (1)–(4) were used to calculate the weight reduction, the solid gain, the water loss, and the dehydration rate, respectively:

$$WR = \frac{(w_0 - w_t)}{w_0} * 100, \quad (1)$$

$$WL = WR + SG, \quad (2)$$

$$SG = \frac{(s_t - s_0)}{w_0} * 100, \quad (3)$$

$$DR = \frac{WL}{t}, \quad (4)$$

where w is the pomegranate seeds' weight; s is the pomegranate seeds' dry matter; and t is the OD duration (min). The indexes 0 and t refer to initial or after an osmotic treatment for a period of time t , respectively.

Peleg's model was used to describe the mass transfer kinetics. It is presented by the following equation:

$$Y_t = Y_0 \pm \frac{t}{k_1 + k_2 t}, \quad (5)$$

where Y is the water loss or the solid gain; k_1 and k_2 are the Peleg parameters; and t is the OD duration. The indexes 0 and t refer to initial or after an osmotic treatment for a period of time t , respectively. The “±” symbol is a minus for the water loss and a plus sign for the solid gain.

For a long processing time, Equation (5) gives the Equation (6) leading to the equilibrium values of water losses and solid gains. These values will be used to determine the effective diffusivities using Fick's second law.

$$Y_{eq} = Y_0 \pm \frac{1}{k_2}, \quad (6)$$

where Y_{eq} is the water loss or the solid gain at equilibrium (g/g of dry matter).

The differential form of Fick's law is given by the following equation:

$$\varphi_i = -D_{eff,i} S \frac{\partial C_i}{\partial x}, \quad (7)$$

where φ_i is the material flow through the surface S during the unit of time; S is the outer surface of a seed; C is the solute concentration; $D_{eff,i}$ is the effective diffusivities; and x is the distance in a direction normal to the seeds' surface. The index i is relative to the diffusing species, $i = w$ for water and $i = s$ for solute.

By considering that seeds are homogeneous spheres, the initial solid and water contents are uniform throughout the volume of the material, the equilibrium solid and the water contents are uniform throughout the surface, the diffusion coefficient is constant, and the resistance to the external mass transfer is negligible compared to the internal resistance, the solution of Fick's equation is given by

$$W_i = \frac{Y - Y_{eq}}{Y_0 - Y_{eq}} = \sum_1^n \frac{6}{(n\pi)^2} \exp\left(- (n\pi)^2 D_{eff,i} \frac{t}{R^2}\right), \quad (8)$$

where $D_{eff,i}$ is the effective diffusivity of water or solute; n is the number of series terms; R is the equivalent radius of sphere; t is the time; W_i is the dimensionless amount of water losses or solid gains, respectively; and i is an index indicating water ($i = w$) or solute ($i = s$).

3. Results and Discussion

3.1. Effect of Processing Time on the Mass Transfer during OD. OD incorporates a double product transformation in its drying process: there is water removal as well as the solute incorporation, resulting in the overall product weight reduction. Figure 1 shows the evolution of the water loss and solid gain during the OD of pomegranate seeds at $T = 40^\circ\text{C}$, $C = 50^\circ\text{Bx}$, $N = 440$ rpm, and sample/solution weight ratio equal to 1:4. Both dehydration and sugar impregnation depend on processing time. Indeed, kinetics is faster at the beginning where the potential between the osmotic solution and the pomegranate seed separated by the cell membrane is greater (phase 1), and then it slows down gradually (phase 2) until achieving a nearly constant value, beyond 300 minutes (phase 3: the near equilibrium) [24, 31, 32]. The quasi-equilibrium is caused by the progressive decrease in the driving force and the formation of a solute layer on the cell membrane surface which can prevent the passage of molecules of water and solute [24, 33]. On the other hand, there is a quantitative difference between the water loss and the solid gain occurring during OD reflecting the cell membrane selectivity. Indeed, given its small size, the passage of the water molecule across the cell membrane is favored over that of sucrose which in most will be trapped in the pores.

3.2. Effect of Hypertonic Solution Solid Content on the Mass Transfer during OD. Figure 2 gives the water loss and solid

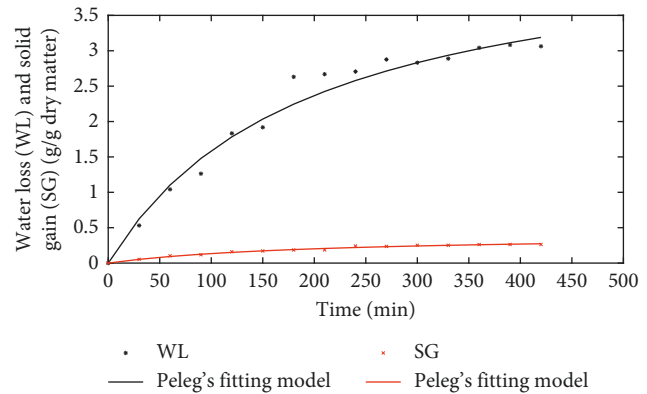


FIGURE 1: Effect of processing time and membrane selectivity on the mass transfer during OD of pomegranate seeds: ($T = 40^\circ\text{C}$, $C = 50^\circ\text{Bx}$, $N = 440$ rpm, and sample/solution weight ratio = 1:4).

gain evolution during the pomegranate seeds' OD as a function of osmotic solution's soluble solid content (Brix). Experiments were carried out at 20°C and 330 rpm. The equilibrium was not reached for all the concentrations given the low temperature and stirring speed. This does not preclude the clarity of the effect of the osmotic solution's initial soluble solid content on water loss and solid gain.

On the first hand, an increase in the osmotic solution concentration results in an increase in water loss and solid gain. On the other hand, the slopes of mass transfer curves as a function of time increase with concentration, which reflects the improvement in transfer kinetics. Indeed, by increasing the concentration, the difference in the water load between the osmotic solution and the product increases, and subsequently the transfer becomes faster and more important. When moving from 30°Bx to 40°Bx and to 50°Bx , a remarkable increase in water loss and solid gain was noted. For the 50°Bx and 60°Bx , the water loss curves are almost confounded while the solid gain continues to increase. This can be explained by the formation of a layer of sugar on the surface of the pomegranate seed, which forms a barrier to water transfer and leaves the sugar exposed to the pores. Finally, we can conclude that the concentration has a significant effect on mass transfer during OD of pomegranate seeds, but very high concentrations are not helpful to not prevent the water transfer while favoring that of sugar. 50°Bx was chosen as the optimal concentration for the OD of pomegranate seeds.

3.3. Effect of Temperature on the Mass Transfer during OD. OD is achievable even at low temperatures, which avoids the adverse effects of heat on the food. After seven hours of OD of pomegranate seed at room temperature (20°C), the water loss was about 41% accompanied with 3% of solid gain. A reasonable increase of temperature leads to an increase in water loss and solid gain as well as an acceleration of mass transfer kinetics, as shown in Figure 3. Indeed, the increase of temperature causes the cell membrane softening and the opening of its pores which allows a more intense and faster mass transfer. By increasing the temperature from 20°C to 30°C , the water loss was almost not affected, but the solid

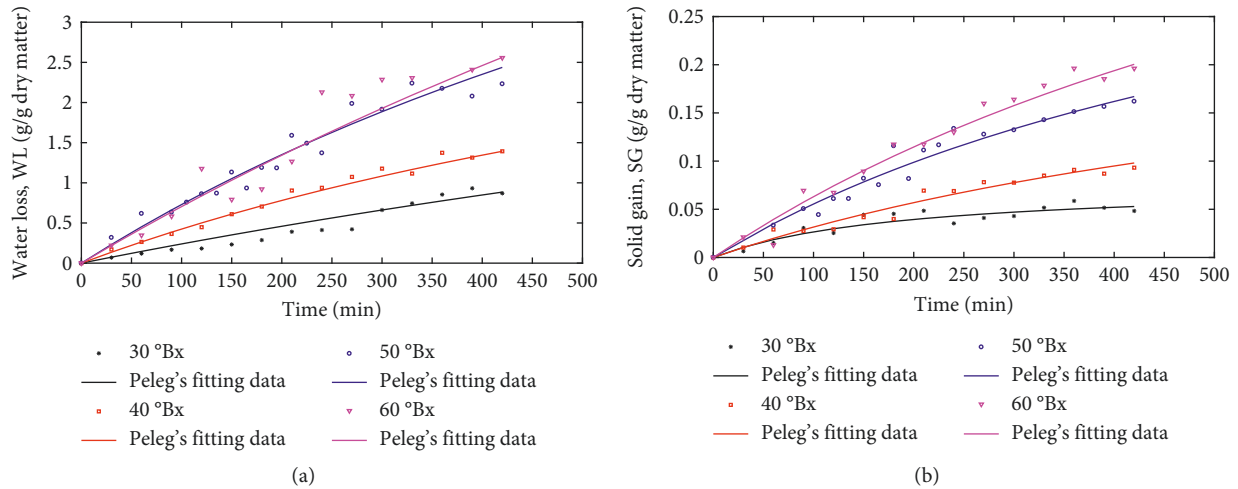


FIGURE 2: Effect of osmotic solution solid content on the mass transfer during OD of pomegranate seeds: (a) water loss and (b) solid gain ($T = 30^{\circ}\text{C}$, $N = 330$ rpm, and sample/solution weight ratio = 1 : 4).

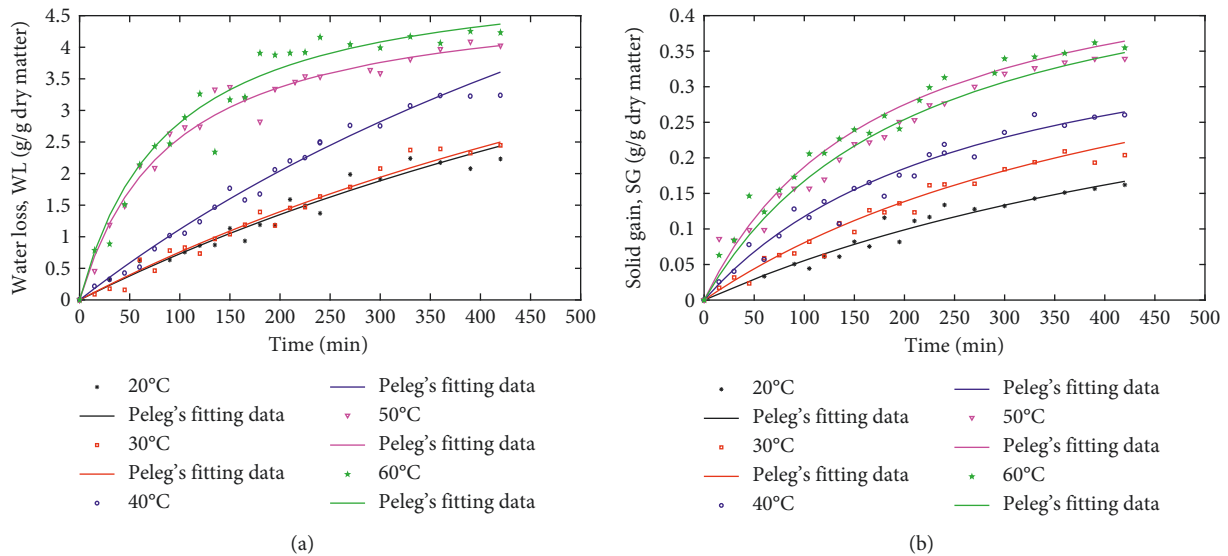


FIGURE 3: Effect of temperature on mass transfer during the OD of pomegranate seeds: (a) water loss and (b) solid gain ($C = 50^{\circ}\text{Bx}$, $N = 330$ rpm, and sample/solution weight ratio = 1 : 4).

gain increased. So, the cellular structure was affected, but the sugar impregnation took precedence over dehydration. Moving from 30°C to 40°C , both water loss and solid gain increased. By going to higher temperatures, there is a very significant acceleration of mass transfer kinetics with close to perfect pseudo first-order curves. From the point of view of mass transfer, fast kinetics and clear equilibrium bearing accompanied with a significant water removal are desirable. But, at these two temperatures, the osmotic solution becomes pink indicating the hard affection of product texture and the loss of cell membrane semipermeability. The appearance of this color reflects the transfer of pomegranate seeds' own solutes, in particular the anthocyanin which is responsible for its pink coloration, to the osmotic solution and the assignment of the product quality. It has been previously reported that such high temperatures caused

irreversible damage and a selectivity loss of cell membrane [34, 35]. Similarly, Khan defined 50°C as a reasonable limit temperature for vegetables and fruit to the deterioration of flavor, texture, and heat-sensitive compounds of product [36]. Indeed, enzymatic browning and deterioration of fruit flavor begins at a temperature of 49°C . In recap, a moderate temperature increases to improve the water and solute transfer during OD. But, relatively high temperatures cause undesirable irreversible effects on aliment texture. So, in the case of pomegranate seeds, 40°C was chosen to reach the maximum water removal without damaging the cell membrane and losing the fruit quality.

3.4. Effect of Stirring Speed on OD Kinetics. Magnetic or mechanical agitation is very useful in similar cases. It makes

it possible to ensure the mixture homogeneity and the temperature uniformity, and it reduces the mass transfer resistance of the cell membrane [37]. Figure 4 shows the effect of stirring speed on water loss and solid gain during pomegranate OD at 50°Bx, 40°C, and a product/solution ratio of ¼. At 330 rpm, the water loss and solid gain evolution are almost linear: this low stirring speed did not sufficiently reduce the cell membrane resistance to mass transfer. By going to 440 rpm, it is noted that the effect of the stirring speed is located mainly at the beginning of OD process without actually affecting the final transferred solute and water. The resistance to mass transfer was reduced, and the water loss and solid gain curves are getting closer to pseudo first-order shape. Similar results were found by [36]. By using a stirring speed of 550 rpm, the water loss and solid gain increased significantly, and the osmotic solution changed the color to pink. Indeed, the high stirring speed causes the damage of seeds and produces some cracks on its surface which explain the osmotic solution's new color caused by the transfer of some own solutes' pomegranate seeds and the quantitatively larger transfer of water and sugar through the damaged cell membrane.

Finally, 50°Bx, 40°C, and 440 rpm were chosen as the optimal OD operating conditions allowing to higher water loss and less product damage. The optimal conditions were used to unfold the effect of blanching on the pomegranate seeds' OD kinetics.

3.5. Effect of Blanching Pretreatment on OD Kinetics. The difference of the OD from one cellular tissue to another depends on the cellular tissue characteristics, the solute nature, the operating conditions, and the pretreatment. These conditions can accelerate or slow down the mass transfer. In this work, the blanching pretreatment affects the pomegranate seeds' texture and of course the mass transfer during the following treatment. Figure 5 shows the water loss and solid gain evolution during the OD of pomegranate seeds in sucrose solution for blanched and raw samples and the corresponding kinetic Peleg's model fitting. During the first and the second phase of OD, remarkable differences in both water loss and solid gain were noted between the untreated sample and the blanched samples. But, at the end of process, at the near-equilibrium, water loss and solid gain in the case of blanched samples tend to reach almost the same amounts such as the control sample. This behavior can probably be explained by the reversibility of the cell membrane permeabilization [38, 39]: The blanching pretreatment opens the cell membrane pores but does not destroy the cellular texture. The cellular tissue returns to its initial state after a period. Therefore, the OD near-equilibrium for the pretreated samples has the same level as the control sample.

Peleg's parameters (k_1 and k_2) were determined for water loss and solid gain using equation (5), as shown in Table 1. All the correlation coefficient values demonstrate the goodness of fit.

The Tunisian pomegranate seeds have already a high sugar content. Wherefore, we need to eliminate water with a

minimum of sucrose impregnation. Therefore, the final water loss to solid gain ratio values were calculated for raw and blanched seeds and reported in Table 1. They were 8.729, 11.432, and 12.764 for control sample, microwave-blanched seeds, and boiling-water-blanched seeds, respectively. The pomegranate seeds blanched in a boiling water bath gave the higher WL/SG. It corresponds to the best treatment.

3.6. Effect of Blanching Pretreatment on the Dehydration Rate of Pomegranate Seeds. Figure 6 reports the dehydration rate as a function of time for both blanched and raw samples. For economic reasons, the OD must be stopped when reaching the half of the maximum of the dehydration rate [35]. The maximum dehydration rates of pomegranate seeds during the OD were in the order of 0.0176 g/min, 0.054 g/min, and 0.056 g/min for unblanched samples, microwave-blanched samples, and boiling-water-blanched samples, respectively. The maximum dehydration rates for the two blanched samples were slightly different. In particular, the time necessary to osmodehydrate unblanched pomegranate seeds was 300 min. Nevertheless, it was about 85 min for the two blanched samples. In fact, a processing time reduction of 72% was achieved by applying blanching as a pretreatment of the OD.

The reduction of OD's processing time was mostly due to cellular membrane softening caused by the blanching treatment, which facilitated the water and solute transfer as previously described in Section 3.5.

3.7. Solute and Water Effective Diffusivities Prediction. The solution of Fick's diffusion law, given by equation (8) was used to evaluate effective diffusion coefficients. For long periods of dehydration, the sum in equation (8) can be limited to the first term of the series [40].

Table 2 shows the effective diffusivities values for water and solute calculated using Fick's model, which also presented a good fit to experimental data, showing an average correlation coefficients (R^2) close to unity. The water and solute effective diffusivities of the control sample were the lowest one. They were, respectively, 8.9×10^{-9} and 5.2×10^{-9} . Indeed, the cell membrane was intact and had the highest firmness. Therefore, its permeability for solute and water was lower. These values are comparable to those found by Herman-Lara et al. [41] working on the OD of radish in NaCl solutions. Bchir et al. [42] working on the OD of pomegranate seeds in sucrose solution found lower values of effective diffusivities, in the order of 10^{-12} . This comparison should however take into account the experimental conditions, the different estimation methods employed, and the varieties of pomegranate fruit studied.

The diffusion coefficients increased by applying the blanching pretreatment using the two methods. The moisture diffusivities increased from 8.9×10^{-9} to 2.8×10^{-8} for the microwave blanching and to 1.8×10^{-8} for the boiling water bath blanching. Similarly, solute diffusivities increased from 5.2×10^{-9} to 1.8×10^{-8} for the microwave blanching and to 4.1×10^{-8} for the boiling water bath blanching. This behavior was probably due to cellular tissue

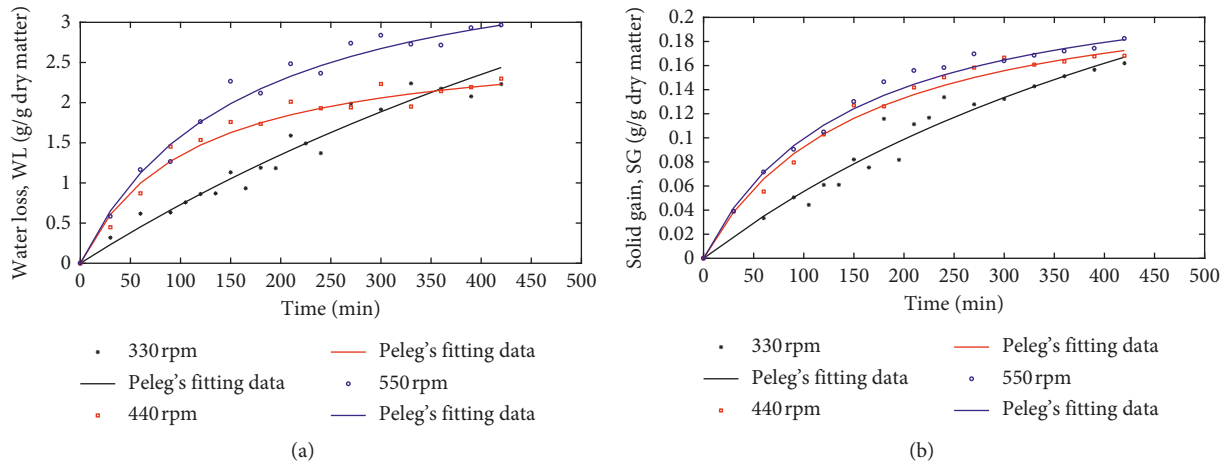


FIGURE 4: Effect of stirring speed on mass transfer during the OD of pomegranate seeds: (a) water loss and (b) solid gain ($C = 50^\circ\text{Bx}$, $T = 40^\circ\text{C}$, and sample/solution weight ratio = 1 : 4).

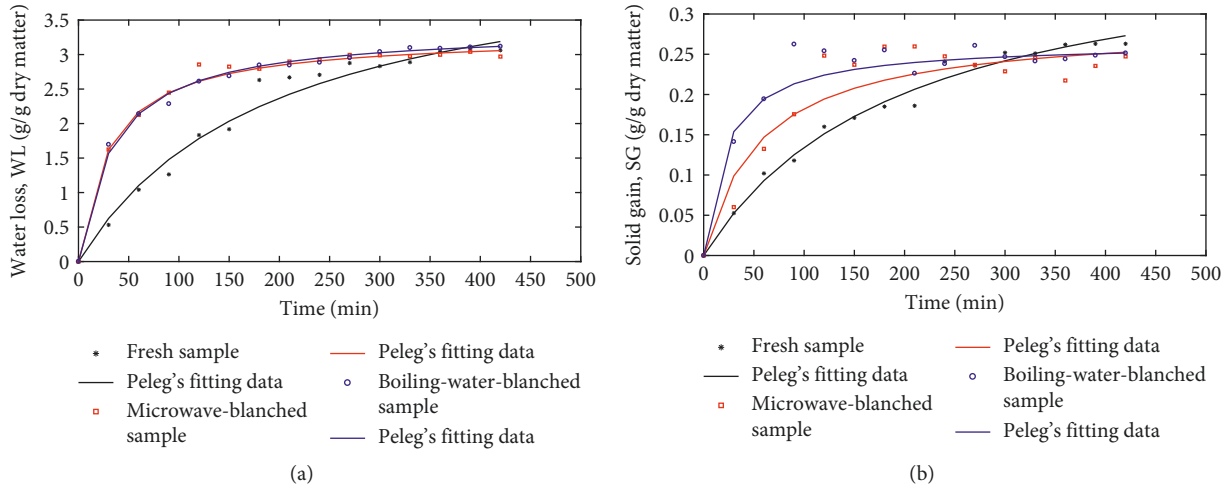


FIGURE 5: Effect of blanching pretreatment on the (a) water loss and (b) solid gain during OD of pomegranate seeds ($C = 50^\circ\text{Bx}$, $T = 40^\circ\text{C}$, $N = 440$ rpm, and sample/solution weight ratio = 1 : 4).

TABLE 1: Peleg's parameters.

Sample	Water loss (WL)			Solid gain (SG)			WL_{eq}/SG_{eq}	
	k_1	k_2 (min)	R^2	k_1	k_2 (min)	R^2		
Temperature	20°C	126.006	0.110	0.977	1580.3	2.2	0.976	—
	30°C	120.700	0.112	0.989	1033.6	2.1	0.985	—
	40°C	81.044	0.084	0.992	620.892	2.299	0.983	—
	50°C	18.560	0.204	0.988	406.691	1.904	0.989	—
	60°C	16.693	0.189	0.978	339.998	1.936	0.991	—
Concentration	30°Bx	400.000	0.1781	0.969	2458.9	13.1	0.941	—
	40°Bx	214.811	0.2065	0.994	2815.2	3.5	0.980	—
	50°Bx	126.006	0.1102	0.978	1580.3	2.2	0.976	—
	60°Bx	135.010	0.0688	0.965	1423.9	1.6	0.988	—
Stirring speed	330 rpm	126.006	0.110	0.978	1580.1	2.2	0.976	—
	440 rpm	38.699	0.357	0.987	654.785	4.238	0.994	—
	550 rpm	38.706	0.245	0.990	593.496	4.094	0.995	—
Fresh seeds	19	0.284	0.96	496	2.48	0.99	8.729	
Microwave-blached seeds	9	0.305	0.99	99	3.47	0.94	11.432	
Boiling-water-blached seeds	10	0.296	0.99	82	3.78	0.97	12.764	

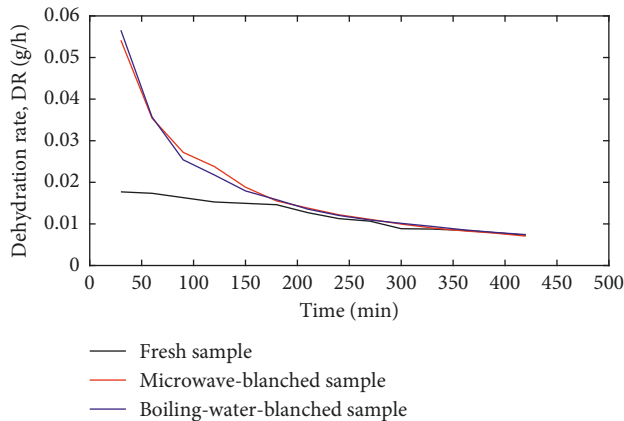


FIGURE 6: Effect of blanching pretreatment on the dehydration rate of pomegranate seeds ($C = 50^{\circ}\text{Bx}$, $T = 40^{\circ}\text{C}$, $N = 440$ rpm, and sample/solution weight ratio = 1 : 4).

TABLE 2: Solute and water effective diffusivities during OD.

Sample	Water loss (WL)		Solid gain (SG)	
	$D_{\text{effw}}^{\text{eff}}$ $\text{m}^2 \cdot \text{s}^{-1}$	R^2	$D_{\text{effs}}^{\text{eff}}$ $\text{m}^2 \cdot \text{s}^{-1}$	R^2
Fresh seeds	8.885×10^{-9}	0.988	5.157×10^{-9}	0.981
Microwave-blanching seeds	2.846×10^{-8}	0.987	1.764×10^{-8}	0.939
Boiling-water-blanching seeds	1.797×10^{-8}	0.961	4.072×10^{-8}	0.975

softening caused by the blanching treatment, which facilitated the water and solute transfer [39]. Different authors reported similar results for various vegetables and fruits: for pumpkin by Kowalska et al. [17], for leek slices by Doymaz [43], and so on.

For samples blanched in the sucrose solution in the microwave, the solute effective diffusivity is lower than those for the sample blanched in boiling water. Indeed, the blanching in the hypertonic solution causes a solute impregnation inside the sample during the pretreatment stage. Therefore, when going to the dehydration stage, pomegranate seeds were already enriched in sucrose and their pores were partially occupied by solute, which makes the sugar diffusion during the OD more difficult [39].

4. Conclusions

In this study, the optimal operating conditions for the OD of Tunisian pomegranate seeds were assessed. The best conditions giving the higher water losses and the best fruit quality were found to be 50°Bx , 40°C , and 440 rpm. At these optimum values of temperature, hypertonic solution solid content, and stirring speeds, the effect of blanching on water and solute transfer during the OD was investigated. Blanching has been applied in two techniques: microwave blanching and blanching in a boiling water bath. Pretreatment such as blanching improves the OD kinetics and reduces the dehydration processing time. The effective moisture and solute diffusivities are in the order of 10^{-9} for

the raw samples and in the order of 10^{-8} for the blanched samples. In terms of processing duration and WL/SG ratio, the best combination is by coupling the OD to the blanching in boiling water bath.

Data Availability

The experimental data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

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