Characteristics of Apple Juice and Sugar Infused Fresh and Frozen Blueberries

Felix Akharume¹, Kaushlendra Singh¹, and Litha Sivanandan²

¹School Natural Resources, West Virginia University, Morgantown, USA

²Extension Service, West Virginia University, Morgantown, USA

(Corresponding Author: Kaushlendra Singh, 322 Percival Hall, PO: 6125, Morgantown,

WV 26505-6125, Email: Kaushlendra.Singh@mail.wvu.edu)

Abstract

Use of apple juice concentrate and standard sugar-based osmotic solution were compared during osmotic dehydration (OD) of fresh and frozen blueberries for solid gain and water loss. Additionally, fresh and frozen blueberries were characterized for moisture desorption using a thermo-gravimetric analyzer (TGA) at an isothermal temperature of 105 °C under dry nitrogen condition. Weight loss-time data generated from the TGA was used to calculate the overall liquid diffusion coefficient during moisture desorption. Results showed that the use of apple juice concentrate as an osmotic solution increased sugar concentration of frozen blueberries to 30.30 °Brix, which was similar to that obtained from the use of sugar-based osmotic solution (32.90 °Brix). Additionally, irrespective of the osmotic solution type, the osmotically dehydrated frozen blueberries reached the desired safe water activity (a_w) range (0.40 – 0.50) in the 600 min drying time during follow-up drying at 74 °C. Moisture desorption characterization using TGA showed that fresh blueberries took about 53% additional drying time compared to frozen blueberries.

Key words: Blueberries, Osmotic Dehydration, Thermogravimetric Analysis, Air Drying, Sugar Solution, Apple Juice

1. Introduction

Blueberries (*Vaccinium Corymbosum* L.) are a high economic value but highly perishable fruit. They are widely consumed for their nutraceutical and nutritive value (Jimenez-Garcia et al., 2013; Martineau et al., 2006; Nile & Park, 2014; Shi et al., 2008a). The United States is by far the largest producer of blueberries, with a total production (wild and cultivated) of 309 kilotons in 2014 (USDA, 2015).

Owing to their short shelf life, blueberries have to be preserved to ensure availability all year round. Direct air-drying, via convection, is one of the widely used methods for preserving blueberries. However, direct air-drying of blueberries requires a prolonged drying time, high energy input, and may lead to non-uniform quality of the final product (Liping, 1998; Vega-Gálvez et al., 2012). To improve the quality, reduce the drying time, and minimize overall energy cost of the dried fruit, direct air-drying is generally preceded by the combination of pre-drying treatments, such as blanching, freezing, osmotic dehydration (OD), enzymes treatments, and chemical treatments (Vega-Gálvez et al., 2012). Osmotic dehydration (OD) is being widely applied as a pretreatment because it minimizes energy consumption and improves the quality of fruit due to reduced heat damage to the fruit's texture, enhanced color, and increased retention of volatiles (Ketata et al., 2013; Azarpazhooh & Ramaswamy, 2010; Shi et al., 2008a). Water removal and sugar uptake in the fruit during OD process are influenced by size of the fruit (Shi et al., 2008b), pretreatments with chemical agents (Ketata et al., 2013; Kucner et al., 2013), contact time and temperature (Nsonzi & Ramaswamy, 2008), and type of osmotic agents (Shi. et al 2009; Kucner et al., 2013; Shi et al., 2009; Yadav and Singh, 2014).

Traditionally, six-carbon dominant sugars (sucrose) are widely used for making osmotic solutions; however, their use renders dried product sugar-rich, a dietary concern among consumers striving to reduce calorie intake from added-sugar. We visualize substituting sucrose with the 5-carbon sugars present in most fruit-juices, for example, apple juice concentrate. Very sparse scientific data is available on the use of apple juice concentrate as an osmotic agent for osmotic dehydration of sour cherries, blackcurrants, and apples (Konopacka et al., 2008; Konopacka et al., 2009) and use of fruit juices during osmotic dehydration of blueberries has not been reported. Konopacka et al. (2008) compared the usefulness of selected fruit juices (concentrated apple juice, mixture of concentrated apple juice and sour cherry juices, and de-acidified concentrated apple juice) with traditional sucrose solution during osmotic dehydration of cherry fruit. They reported that the increase in dry matter content of cherry fruit after 2 h of OD using 60 °Brix osmotic solutions, was statistically similar irrespective of type of osmotic solution (fruit juices or sucrose) (Konopacka et al., 2008). However, types of osmotic solution (sucrose, concentrated apple juice, de-acidified and desalted concentrated apple juices, sorbitol, etc.) and types of fruit (sour cherries, blackcurrants, and apples) have significant effect on water loss and dry matter uptake during 1 h long OD performed using 65 °Brix osmotic solutions (Konopacka et al., 2009). However, it is not clear if contact time of 1 h or 2 h were sufficient to reach equilibrium during the OD processes for sour cherries, blackcurrants, and apples fruits. The purpose of this research was to compare the use of apple juice concentrate and sucrose solutions as osmotic agents during osmotic dehydration of blueberries (fresh or frozen) for contact time equivalent to equilibrium period. The equilibrium period is defined as the time from the start of subjecting fruit to the OD process until no further change in °Brix of either fruit or

osmotic solution takes place. Additionally, the research characterized fresh and frozen blueberries for moisture desorption behavior.

2. Materials and Methods

2.1 Blueberries and Osmotic Agents

Fresh blueberries (Bleuets, distributed by Sunbelle Inc. Miami FL. USA) and frozen blueberries (Blue Crop and Blue Jay varieties, distributed by Kroger Co. Cincinnati OH. USA) were purchased from a retail store. The fresh blueberries were stored below 4 °C in a refrigerator and frozen blueberries were stored in a freezer below -18 °C prior to use. For the purpose of physical characterization and moisture desorption characterization experiments, the frozen blueberries were thawed at room temperature for 2 h and blotted with tissue paper to remove excess water before use. However, for the osmotic dehydration treatment the frozen blueberries were used without thawing.

To make the various osmotic solutions, granular sugar (Pssst brand, distributed by Kroger Co. Cincinnati OH. USA) and apple juice concentrate (Kroger 100% apple juice frozen concentrate, distributed by Kroger Co. Cincinnati OH. USA) were purchased from a retail store. The apple juice concentrate had an initial sugar concentration of 42 ± 2 °Brix. Therefore, osmotic solutions from granular sugar were prepared at the same 42 ± 2 °Brix concentration. About 252 g granular table sugar was thoroughly mixed 348 g of deionized distilled water in a conical flask. For quick solubility of the sugar, the solution was heated to a temperature of 100 °C and then cooled to room temperature prior to use. A cover of poly-film metallic foil was used to prevent solution loss through evaporation during heating.

2.2 Physical Characterization Measurements

The three significant axial dimensions (Fig. 1) of 100 randomly-selected fresh and frozen (thawed) blueberries were measured using an electronic Vernier caliper (least count 0.02 mm). Effective diameter (D_e), sphericity (ϕ), aspect ratio (R_A) and surface area (S_A) were calculated using the axial dimensions according to equations 1, 2, 3 and 4 given below (Jain, 1997; Mohsenin, 1986; Omobuwajo, 1999):

$$D_e = (abc)^{1/3} \quad (1)$$

$$\phi = \frac{D_e}{a} \quad (2)$$

$$R_A = \frac{b}{a} \quad (3)$$

$$S_A = \frac{\pi a^2 (bc)^{0.5}}{2a - (bc)^{0.5}} \quad (4)$$

Where a is the major diameter (mm), b is the intermediate diameter (mm), and c is the minor diameter (mm).

Fig. 1.

Additionally, the fresh and frozen (thawed) blueberries were characterized for moisture content, bulk density, true density, and volumetric moisture compression. The dried samples of fresh and frozen blueberries were also tested for volumetric shrinkage, bulk density and true density. Both fresh and frozen blueberries were assayed for initial moisture content according to the AACC method 44-15A (Moisture – Air-Oven Methods) (AACC, 2000). The bulk density of fresh as well as oven-dried blueberries was measured according to ASTM standard (ASTM C29/C29M – 2009). To measure the true density, the actual volume of the samples excluding pore volume,

was measured using a multipycnometer (Model: MVP.6DC, Quantachrome, Boyton Beach, FL. USA). Volumetric shrinkage was calculated using equation 5.

$$\text{Volumetric Shrinkage} = \frac{V_{\text{Green}} - V_{\text{Oven-dry}}}{V_{\text{Oven-dry}}} \times 100 \quad (5)$$

Additionally, volumetric compression of water present in the blueberries was calculated by following equation 6.

$$\text{Volumetric Compression of Water} = \frac{(m_{\text{Green}} - m_{\text{Oven-dry}}) / \rho_{\text{Water}} - (V_{\text{Green}} - V_{\text{Oven-dry}})}{(m_{\text{Green}} - m_{\text{Oven-dry}}) / \rho_{\text{Water}}} \times 100 \quad (6)$$

In this equation, m_{Green} is the weight of the fresh sample (g), $m_{\text{Oven-dry}}$ is the weight of an oven-dried sample (g), ρ_{Water} is the density of water (g/cm³), V_{Green} is the volume of the green sample (cm³), and $V_{\text{Oven-dry}}$ is the volume of the oven-dried sample (cm³).

2.3 Moisture Desorption Characteristics

To investigate the drying characteristics of the samples, drying of fresh and frozen blueberries samples was carried out in a Thermogravimetric analyzer (TGA) (Model: TGA 701, LECO Corporation, IL) under constant nitrogen flow at the isothermal temperature of 105 °C. About 8 mg of sample, replicated twice, was placed in a TGA crucible. The samples were heated from room temperature to 105 °C at the rate of 16 °C/min under constant dry nitrogen flow to keep the relative humidity zero following an ASTM standard (ASTM D7582, 2015). During the heating as well as during the isothermal drying, the weight of the sample, duration, and furnace temperature were recorded in a computer connected to the TGA. The weight-time-temperature data was further processed to produce drying characteristics curves (rate of weight loss as a function of time, moisture ratio or mean moisture content) and to calculate overall liquid diffusion coefficients.

The moisture content was calculated on a dry-basis as follows:

$$MC_i = \frac{m_i - m_f}{m_f} \times 100 \quad (7)$$

Where MC_i is the dry-basis moisture content at i^{th} time (g/100 g d.b), m_i is the sample weight at i^{th} time m_f is the final (weight of dry solid) sample weight (g). For further calculations, fractional moisture content and drying rate (dX/dt), were calculated according to equations (8 and 9) according to (Geankoplis, 2003).

$$X_i = \frac{MC_i}{100} \quad (8)$$

$$DR = \frac{dX}{dt} = \frac{X_{i-1} - X_i}{t_i - t_{i-1}} \quad (9)$$

Where X_i is moisture fraction at i^{th} time $\left(\frac{\text{g of water}}{\text{g of dry solid}}\right)$, dX/dt is drying rate $\left(\frac{\text{g of water}}{\text{g of dry solids.min}}\right)$ between $(i-1)^{\text{th}}$ and i^{th} time intervals and at mean moisture fraction of $\left(\frac{X_{i-1} + X_i}{2}\right)$.

To compare drying behaviors of samples having different initial moisture contents, the moisture ratio was calculated to normalize moisture content at the i^{th} interval with initial moisture contents, as given below (Chen et al., 2012)

$$MR = \frac{MC_i - MC_e}{MC_0 - MC_e} \quad (10)$$

Where MC_0 is initial moisture content, MC_i moisture content at i^{th} time t , and MC_e is the Equilibrium Moisture Content (EMC), which is zero under the current experiments according to the ASTM standard (ASTM D7582, 2015). The TGA equipment uses a dry nitrogen atmosphere and prescribed temperature of 105 °C to dry any material to bone-dry conditions. However, the bone-dry state of material might not be achieved when it is dried in air of varying humidity and temperatures (60 °C to 80 °C), where only EMC are reached. EMC depends on type of material being dried, air temperature, and relative humidity. To reach bone-dry material all free water and bound water (capillary condensation, polymolecular adsorption, and monomolecular adsorption) must be removed. For example, air-drying of blueberries at 60 °C, 70 °C and 80 °C using inlet relative humidity of $62.0 \pm 5.2\%$ (not inside drying chamber) had resulted in the EMC of 0.031 g water/g dry solid, 0.027 g water/g dry solid and 0.026 g water/g dry solid, respectively (Vega-Gálvez et al., 2009). In this case, relative humidity values in the drying chamber would have been lower than the inlet relative humidity but also different for each drying temperature, probably not sufficient to remove all types of bound water (Vega-Gálvez et al., 2009).

Overall liquid diffusion was assessed from the TGA data according to the procedure outlined by (Geankoplis, 2003). According to the procedure, the drying process of biological materials mostly occurs in the falling rate period. Fick's second law of diffusion (Eqn. 11) (Chen et al., 2012) has been extensively used to describe the drying process and interpret drying data. The mathematical solution of Fick's second law for diffusion for spherical bodies is shown in Equation (12) (Azarpazhooh & Ramaswamy, 2010):

$$\frac{\partial MR}{\partial t} = \nabla [D_{eff} (\nabla MR)] \quad (11)$$

$$MR = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp\left(-\frac{n^2 \pi^2 D_{eff} t}{r^2}\right) \quad (12)$$

Where n is a positive integer, t is the drying time(s), D_{eff} is overall liquid diffusion coefficient (m^2/s), and r is the effective radius of the blueberries (m).

When the sample shrinkage is negligible, initial moisture distribution is uniform, and constant moisture diffusivity is assumed, Equation (12) is suitable for determining the D_{eff} . This equation could be further simplified into Equation 13 by taking the first terms of the series solution for long drying times (Ramaswamy & Nsonzi, 1998; Shi et al., 2008a)

$$\ln(MR) = \ln\left(\frac{6}{\pi^2}\right) - \left(\frac{\pi^2 D_{eff}}{r^2} t\right) \quad (13)$$

Effective liquid diffusivities are typically determined by plotting experimental drying data in terms of $\ln(MR)$ versus time. From equation (13), a plot of $\ln(MR)$ versus drying time, gives a straight line with a slope (K_o)

$$K_o = \frac{\pi^2 D_{eff}}{r^2} \quad (14)$$

2.4 Osmotic Dehydration Experiments

Osmotic dehydration was facilitated by soaking a desired fruit sample in an osmotic solution at a constant incubation temperature of 50 °C. The fruit-to-solution weight ratio was targeted at 1:3. In a typical osmotic dehydration experiment, about 200 g of fruit sample was placed in about 600 g of the hot osmotic solution. The pre-formulated osmotic solution was heated up quickly to a designated temperature (100 °C) before the blueberries were immersed. The fruit-solution mixtures were cooled to the target temperature of 50 °C before transferring to a Lab Line Imperial III incubator (Model: 305, Thermo-scientific, IA. USA). Agitation in the incubator was provided by a platform shaker (Model: EXCELLA E2, New Brunswick Scientific Co. NJ. USA)

set at 50 rpm. Sugar concentrations of the solution and blueberries were measured at intervals of 1 h by means of a Reichert Digital Refractometer, according to the method of Association of Official Analytics Chemist (AOAC) 990.35 (Shi et al., 2009). After the blueberry sugar concentration became constant (equilibrium period), the blueberries were removed from the solution and rinsed with tap water for 30 s on a rubber mesh. They were then blotted with tissue paper to remove excess water. The final weight of the sample was recorded.

Solid gain (SG , g solid/g dry solid), and water loss (WL , g water/g dry solid) were calculated according to (Liping, 1998; Shi et al., 2009) as follows:

$$SG = \frac{W*(1-M)}{W_o*(1-M_o)} - 1 \quad (15)$$

$$WL = \frac{W_o*M_o - W*M}{W_o*(1-M_o)} \quad (16)$$

Where M is the fractional moisture content of the sample after osmotic dehydration on a wet basis (g / g), M_o is the initial fractional moisture content of the sample on a wet basis (g / g), W is the weight of the sample after osmotic dehydration (g), W_o is the weight of the sample before osmotic dehydration (g).

2.5 Air-Drying of Osmotically Dehydrated Blueberry Samples

Osmotically dehydrated fruit samples were placed on tray and further air dried in a fruit dehydrator (Model: 3900T, Excalibur Products, CA. USA) in a climate-controlled room maintained at ambient condition of relative humidity ($43.00 \pm 5.00\%$) and temperature (21.10 ± 2.00 °C). During air-drying, the fruit dehydrator was maintained at 74 °C. Only two of the dehydrator trays were used in the drying for efficient hot air circulation. The trays were placed in the middle of the dehydrator cabinet, directly facing the blower and separated one step

from each other. Osmotically dried blueberries were spread uniformly on the tray and the trays were rotated clockwise at every 1 h. Moisture content and water activity were measured at intervals of 1 h. The water activity was measured using a water activity meter (Model: Aqua Lab Water Activity Meter series 3TE, Decagon Devices, Inc. WA. USA.). Additionally, moisture content was measured by recording the weight loss of a “20 g weighted representative sample”, taken from the bulk sample, during drying. Air-drying was considered complete when the water activity reached a range of 0.45-0.5. Additionally, a representative air-dried sample was further oven-dried at 105 °C for 24 h to measure moisture content of air-dried samples.

2.6 Experimental design

A completely randomized design was applied for the experiment. To compare osmotic dehydration of fresh and frozen blueberries, two treatments of fruit type (fresh and frozen blueberries) were considered with three replications (N=6) for the pre-determined response variables (osmotic dehydration time, solid gain, water loss, and air-drying durations). To compare the influence of osmotic solutions, two treatments of osmotic solution type (sugar-based and apple juice concentrate based osmotic solutions) with three replications (N=6) were considered for the same response variables. Microsoft Excel 2010 was used in summarizing and generating graphs. The *t*-test procedure in SAS statistical software (University Edition) was used in performing a *t* test (at 95% confidence interval).

3. Results and Discussion

3.1 Physical Characteristics of the Blueberries

210 The fresh and frozen blueberry samples were assessed for the following physical
211 characteristics: moisture content, shape characteristics, bulk and true densities. In addition, the
212 same samples were oven-dried, with volumetric shrinkage, moisture compression, and bulk and
213 true densities under dry conditions calculated. Results of all physical characteristics are
214 presented in Table 1. The fresh and frozen blueberry samples used in this study had moisture
215 contents of 5.16 g water/g of dry solids and 5.70 g water/g of dry solids respectively, and upon
216 thawing, the moisture content of frozen berries reduced to 4.30 g water/g of dry solids, a 25 %
217 decrease. Similar values for moisture content were reported by other authors: 5.67g water/g dry
218 solid for both fresh and frozen blueberries (Shi et al., 2008a); 5.28g water/g of dry solid for fresh
219 blueberries (Liping 1998); 4.71g water/g of dry solid for thawed blueberries (Feng et al., 1999).
220 Fresh blueberries had an effective diameter of 13.41 mm and sphericity of 0.89, consistent with
221 ranges reported in literature: 10.40–17.00 mm for diameter and 0.90–0.99 for sphericity for fresh
222 blueberries (Kaleta, 2011). Shi et al (2008b) report that more than 85% of fresh blueberries had
223 diameter within 11.50 – 14.50 mm. They further report that blueberry size correlates with drying
224 rates, for example, drying rates increase with decrease in berry size, although a similar drying
225 rate was recorded for sizes ranging between 13 to 16 mm. Upon thawing, the effective diameter
226 and sphericity reduced to 9.28 mm and 0.76 respectively. The difference in the shape and sizes
227 of the blueberries due to shrinkage that occurred as a result of thawing was observed visually.
228 Shrinkage is essentially due to volumetric water loss and it affects the microstructure of tissue
229 cells (Moreira & Sereno 2003; Allan-Wojtas, et al.,1999). Raghavan & Silveira (2001) reported
230 that shrinkage is linearly correlated with moisture ratio for strawberries. Dimensional
231 characteristic of spherical particles like blueberries increases with an increase in moisture content
232 (Kaleta, 2011). The true densities (density of a single blueberry) of fresh and frozen blueberries

were 1.18 and 1.16 g/cm³, respectively whereas their bulk densities were 0.35 and 0.39 g/cm³, respectively. After drying, the true density increased to 1.72 g/cm³, while the bulk density reduced to 0.20 g/cm³ for fresh blueberries. This is in agreement with the work of (Martynenko, 2014), in which the true density was shown to increase with a decrease in moisture content and bulk density was shown to decrease with a reduction in moisture content at a drying temperature above 40 °C for blueberries. Total volumetric shrinkage was 88.10 % as result of drying. Shrinkage of food matrixes during drying has a direct impact on volume reduction, bulk density and porosity of the food products (Qiu et al., 2015). Moisture loss and volumetric shrinkage data showed that, on average, 69.51 cm³ of water was present in 62.75 cm³ volume space, probably due to compression of water inside the blueberries. Therefore, on average, there was 9.75 % volumetric compression of water inside the fresh blueberry sample. Similarly, after drying of frozen blueberries, the true and bulk densities decreased to 0.96 g/cm³ and 0.19 g/cm³ respectively, the total volumetric shrinkage of 73.52% and on average 79.76 cm³ water was present in only 59.70 cm³ space. Statistically, the compression of water in the thawed blueberries was similar to that of the fresh ones (p-value>0.05).

Table 1

3.2 Moisture Desorption (Drying) Characteristics and Overall Liquid Diffusion Coefficient

Drying characteristics of fresh and frozen (thawed) blueberries were determined in a TGA equipment under isothermal drying at 105 °C under a dry nitrogen atmosphere. To understand how the moisture ratio changes over the drying time, Fig. 2 was plotted for the two samples:

Fig. 2.

The frozen (thawed) blueberries showed a rapid decline in moisture ratio throughout the drying period when compared to the fresh ones. The fresh blueberries took about 260 min to completely dry whereas the frozen blueberries took only 170 min, about 53 % less time. The rapid moisture loss in the frozen blueberries could be attributed to their ruptured surface and internal cell structure as reported in literature. For example, Zielinska et al., reported that frozen blueberries had fractures on their surface and internal tissues, which were not present in fresh blueberries. That is why the frozen blueberries take 29 % less drying time than fresh blueberries (Zielinska et al., 2015). Shi et al. reported a similar trend with the use of NaOH as a pretreatment prior to drying of blueberries (Shi et al, 2008b). They reported that pretreatment with NaOH resulted in an increase in water loss at temperatures above 80 °C due to cracks and deformations made to the fruit tissues, clearly visible under the scanning electron microscope (SEM), when compared to the samples without pretreatment. To better understand how the drying rate changes with moisture content of blueberries, drying characteristic curves were prepared (Fig. 3). Generally, drying of agriculture products has three different periods: 1. an initial heating period, 2. a constant rate period, and 3. a falling rate period (Geankoplis, 2003). The falling rate period progressed in two distinct visible segments (slopes) along with an intermediate transition segment. The first falling rate segment is mainly due to moisture movement controlled by convective flow, which has gentler slope than the latter diffusion flow controlled drying segment (Geankoplis, 2003). Current research made no mention of the observation of a constant rate drying period and the drying proceeded in only heating and falling rate periods. It took about 17 min to reach isothermal conditions of 105 °C. Fig. 3 also clearly shows that the drying rate was higher for frozen blueberries than the fresh blueberries throughout the moisture range.

Fig. 3.

However, the drying rate declined for both samples as the drying progressed. At the start of drying, moisture present closer to the fruit surface quickly evaporated, forming a dry shell encasing a moist core. In this situation, the blueberries took on a core-shell structure. The dried shell continuously grew over time compressing the moist core, which means moisture now had to travel through the dried shell via a “diffusion-in-solid”-dominated process; therefore, the slope of drying curve became less steep. Overall, liquid diffusion coefficients were also calculated for the moisture range of green (fresh) to the moisture content of 15 g/100g (w.b), which is the typical moisture content of commercially dried blueberries. The liquid diffusion coefficients for the frozen (thawed) and fresh blueberries were found to be $8.72 \times 10^{-10} \text{ m}^2/\text{s}$ and $9.11 \times 10^{-10} \text{ m}^2/\text{s}$, respectively, at a temperature of 105 °C. The reported values of overall liquid diffusion coefficients are 0.24×10^{-10} , 1.44×10^{-10} and $12.11 \times 10^{-10} \text{ m}^2/\text{s}$ at temperatures of 40, 60, and 80 °C, respectively for fresh blueberries using a laboratory oven dryer. Additionally, liquid diffusion coefficients for steam-blached blueberries were reported to be 0.49×10^{-10} , $1.93 \times 10^{-10} \text{ m}^2/\text{s}$ and $12.80 \times 10^{-10} \text{ m}^2/\text{s}$ at temperatures of 40, 60, and 80 °C, respectively. There appears to be little difference in the values of liquid diffusion coefficients due to blanching, however, an increase in temperature does have a significant positive effect. (Liping, 1998).

3.3 Comparing Fresh and Frozen Blueberries during Osmotic Dehydration and Subsequent Air Drying

The initial sugar concentration of blueberries was about 10.20 °Brix for fresh and 12.50 °Brix for frozen blueberries. Fresh and frozen blueberries were subjected to osmotic dehydration in a 42±2 °Brix sugar solution at 50 °C. During osmotic dehydration, the sugar concentration was continuously measured both for the blueberries and osmotic solution to determine when it

became constant, as an indication of completion of the osmotic process. As osmotic dehydration proceeds, the sugar concentration of blueberries tends to increase while the sugar concentration of the osmotic solution tends to decrease until an equilibrium is reached between sugar and water concentrations inside blueberries with those in the osmotic solution. Changes in sugar concentration of fresh blueberries and frozen blueberries and their respective osmotic solutions are presented in Fig. 4a and Fig. 4b.

Fig. 4.

A quick observation of these figures indicates that it took an immersion time of about 420 mins for the sugar concentration to stabilize in the blueberries and the osmotic solution. In addition, there was barely any change in the sugar concentration of the osmotic solution for the fresh blueberries (Fig. 4a). However, the sugar concentration of the osmotic solution of frozen blueberries dropped quickly while the sugar concentration of the frozen blueberries increased significantly (Fig. 4b).

The frozen blueberries showed a steady increase in the sugar concentration from 12.50 °Brix to 32.90 °Brix in 420 min (Fig. 5). In contrast, the fresh blueberries showed a small increment of change from 10.20 °Brix to 13.40 °Brix in 200 min and remained at this level even after long hours of immersion (about 10 h). The difference in the increase of sugar concentrations between the fresh and frozen blueberries may be explained by the fractured fruit skin and inner cell membranes of the frozen product, as a result of freezing (Zielinska et al., 2015). Fractured frozen fruit skin and internal cells facilitate two-way diffusion and convective flow (facilitated by mechanical shaking). In the frozen blueberries, ruptured fruit skin does not create an osmotic barrier for sugar flow from the solution to inside the fruit.

Fig. 5.

On the other hand, the fresh blueberries have a strong osmotic barrier due to their continuous skin, which does not permit sugar to flow easily inside of the fruit. Additionally, total solid gain and water loss of blueberries after osmotic dehydration were also calculated related to fruit weight and moisture content before and after the osmotic process (Table 2). It is shown that, three times as much solid was infused in frozen blueberries (23 %) compared to the fresh fruit at the end of the osmotic process, while simultaneously 3.9 times as much water was lost from the frozen berries (181 %). (Kucner et al., 2013) reported a water loss of 0.66 g of water per g of initial dry solid and a solid gain of 0.14 g dry solid per g of initial dry solid for osmotic dehydration of frozen blueberries without pretreatment (at 50 °C, 65 °Brix sucrose solution, fruit to syrup ratio of 1 to 4 for 240 min) (Kucner et al. 2013). It has been generally reported that more water is lost than solid gained during the osmotic dehydration of fruits and vegetables (Azarpazhooh E. and Ramaswamy, 2010). Fruit skin acts as a barrier to the infusion of larger molecular weight sugar like sucrose. Moreover, the simultaneous leaching of the fruit acid into the osmotic solution causes accelerated hydrolysis of sucrose to glucose and fructose and leads to high water loss (Azarpazhooh E. and Ramaswamy, 2010). Another observation illustrated in Table 2 is that frozen berries show a higher yield than fresh ones. A higher sugar gain is a proof of higher yield (Shi et al., 2009).

Table 2

Osmotically dehydrated fresh and frozen blueberries were further air dried in a fruit dehydrator. The fruit dehydrator was set for air temperature of 74 °C, the highest rating of the dehydrator model. During the air-drying, the ambient conditions were 21.10±2.00 °C and relative humidity of 44.00±5.00%. Moisture content and water activity (a_w) of the samples being dried

were continuously monitored at preset intervals of 2 h. Both the frozen and fresh blueberries showed a steady decline in water activity with drying time. Changes in water activity and moisture content during air-drying are shown in Fig. 6. However, for the first 600 min, the a_w and moisture content of the osmotically dried frozen blueberries showed more rapid decline than the osmotically dried fresh blueberries. Another observation shown in Fig. 6 is that the osmotically dried frozen blueberries reached an a_w value of 0.4 in 420 min, which occurred at the moisture content of 0.05 g of water per g of dry solid. On the other hand, osmotically dried fresh blueberries took 600 min to attain the same water activity at the same moisture content of 0.05 g of water per g of dry solid. A closer look at the chart shows that relative to the initial moisture content, osmotically dried frozen berries showed a higher moisture loss to reach the target water activity than the osmotically dried fresh blueberries. This was corroborated by the work of (Shi et al., 2008a). This study observed that reduced drying times of sugar-infused blueberries compared to fresh ones can be attributed to two possible reasons: (1) low initial moisture content of the sugar infused blueberries (2) impediments posed by the infused sugar. Both effects result in lowering the drying rate and thus allowing osmotically dried blueberries to be dried at higher moisture to achieve the same water activity as dried fresh ones.

Fig. 6.

3.4 Comparing Sugar and Apple Juice-Based Osmotic Solutions during Osmotic Dehydration of Frozen Blueberries and Subsequent Air Drying

The frozen blueberries were subjected to osmotic dehydration in an apple juice-based osmotic solution (42.00 ± 2.00 °Brix) and in a sugar-based osmotic solution (42.00 ± 2.00 °Brix) at 50 °C.

Fig. 7 (a and b) shows changes in the blueberry and osmotic solution sugar concentrations as the osmotic dehydration progressed in two situations. In both conditions, the sugar concentration in the fruit as well as in the solutions stabilized before 400 min incubation period. In fact, the osmotic process was completed in just 300 min when an apple juice-based solution was used.

Fig. 7.

Therefore, 400 min is sufficient to complete osmotic process for frozen blueberries in a 42.00 ± 2.00 °Brix osmotic solution at 50 °C, irrespective of solution type. The results of the osmotic dehydration studies indicate that the frozen blueberry sugar concentration increased from 12.50 °Brix to 30.30 °Brix when the apple juice-based osmotic solution was used, and to 32.90 °Brix when the sugar-based osmotic solution was used. There was no statistical difference in the final sugar concentration of the blueberry samples in the two situations ($p\text{-value} > 0.05$) (Fig. 8). While the sugar concentration of the fruit increased, the concentration of sugar in both solutions decreased from an average value of 42.26 °Brix to 38.25 °Brix.

Fig. 8.

It is observed in Fig. 8 that the sugar concentration of frozen blueberries increased in a nonlinear manner with time. The frozen berries pretreated with apple-based osmotic solution, initially showed a lower sugar gain (first 120 min), followed by a higher sugar gain over the next 240 min than that for the fruit pretreated with sugar-based osmotic solution. It has been reported that most water loss and sugar gain are achieved at the first 1 hour of the osmotic process (Azarpazhooh E. and Ramaswamy, 2010). In another study, the highest water loss and solid gain were achieved at a contact time after 180 min (Kucner et al., 2013). Additionally, solid gain and water loss in blueberries during osmotic dehydration are presented in Table 3. The type of

osmotic solution used did not create a significant difference in water loss. The water losses were 1.91 and 1.81 g of water per g of dry solids for apple juice-based and sugar-based solutions, respectively.

Table 3

However, type of osmotic solution used had a significant effect on solid gains. The use of apple juice-based sugar solution showed solid gains of 0.53 g dry solid per g of initial dry solid, which was higher than that shown by the sugar-based solution (0.23 g dry solid per g of initial dry solid). Another observation from the table is that frozen berries immersed in an apple juice solution result in a higher yield (79.31 %) than those immersed in a sugar-based solution (76.33 %). However, the difference was statistically insignificant at (p-value = 0.09). The changes in water activity of osmotically dried frozen blueberries using apple juice and sugar-based osmotic solution, during air-drying in a fruit dehydrator set at 74 °C, are presented in Fig. 9. The data shows that both samples demonstrated a steady decline in water activity with drying time. However, frozen fruit immersed with the sugar-based osmotic solution showed a much more rapid decline for the first 600 min. Moreover, it took a drying time of 420 min to reach a water activity of 0.4 at a moisture content of 0.05 g of water per g of dry solids for the frozen berries immersed in the sugar-based osmotic solution. To reach the same water activity level, it took 480 min at a moisture content of 0.09 g of water per g of dry solids for the frozen berries immersed in the apple juice osmotic solution. The difference in drying time could be attributed to a higher solid gain by the latter sample which slows down the drying rate (Shi et al., 2008a).

Fig. 9.

4. Conclusion

This research establishes that the effect of freezing the fruit prior to hot air drying resulted in a significant reduction in drying time (53% reduction) to reach 0.18 g water /g of dry solid, compared with the similarly dried fresh blueberries. Fresh and frozen berries followed the conventional drying characteristic curves typical of fruits, where only falling rate period but no constant rate regime exist. The drying behavior and a higher drying rate of the frozen berries were facilitated by ruptures in the berries' waxy skin, caused by freezing.

Moreover, the use of osmotic dehydration prior to drying significantly reduces the moisture content of the frozen fruit by about 30 % (from 5.70 - 3.97g of water/g of dry solid) and fresh blueberries by 15 % (5.16 – 4.3997g of water/g of dry solid). Use of an apple juice-based osmotic solution led to a significant higher solid gains in the frozen blueberries than those from a sugar-based osmotic solution. The sugar concentration of frozen blueberries increased from 12.50 °Brix to 30.30 °Brix when the apple juice-based osmotic solution was used and to 32.90 °Brix when the sugar-based osmotic solution was used. On the subsequent hot air drying, the frozen berries treated with sugar-based solution showed a shorter drying time of 420 min than those treated with apple juice-based solution (480 min) to achieve a water activity of 0.4. Therefore, fruit juice concentrates, for example apple juice, may be used as an osmotic solution as a substitute for granular sugar without compromising final yield of the dried blueberries.

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533

List of Tables

- 1- **Table 1.** Physical characteristics, mean (standard deviation) of fresh and frozen (thawed) blueberries.
- 2- **Table 2.** Average moisture content, initial and final sugar concentration of fresh and frozen blueberries and solutions, solid gain and water loss from fruit, osmotic duration and yields during osmotic dehydration at 50°C.
- 3- **Table 3.** Average moisture content, initial and final sugar concentration of frozen blueberries and osmotic solutions (apple juice-based and sugar based), solid gain in fruit and water loss during osmotic dehydration at 50°C.

Table1

Physical characteristics, mean (standard deviation) of fresh and frozen (thawed) blueberries.

	Fresh blueberries	Frozen blueberries (thawed)	Similar data from literatures
Properties	Mean (SD)	Mean(SD)	Mean (SD)
Moisture Content (g water/g dry solid)	5.16 (0.20)	4.30 (0.19)	5.30 (Liping 1998) 4.71 (Feng et al. 1999)
Volumetric Shrinkage (%)	88.10 (1.28)	73.52 (15.07)	90-40 (Martynenko,2014)
Blueberry Moisture Compression (%)	9.75 (1.05)	25.00 (15.48)	This work
Axial dimension (mm)*			
Major diameter, a	15.10 (1.32)	12.26 (1.82)	8.6 - 26.30 (Kaleta 2011)
Interim. Diameter, b	14.77 (1.32)	10.54 (1.83)	11.40-17.80(Kaleta 2011)
Minor diameter, c	10.83 (0.85)	6.29 (1.26)	6.35–17.80(Kaleta, 2011)
Effective diameter, D _e	13.41 (1.04)	9.28 (1.38)	10.40–17(Kaleta 2011)
Sphericity, ϕ	0.89 (0.03)	0.76 (0.06)	0.90-0.99(Kaleta 2011)
Aspect ratio, R _a	0.98 (0.03)	0.92 (0.37)	
Surface area (mm ²)	519.19 (80.52)	235.63 (81.64)	
Densities (g/cm ³)			
True density	1.18 (0.00)	1.16(0.01)	0.70-1.20(Kaleta 2011)
Bulk density	0.35 (0.01)	0.39(0.01)	0.22–0.83(Kaleta 2011)
True density (dried)	1.72 (0.15)	0.96(0.81)	This work
Bulk density (dried)	0.20 (0.00)	0.19(0.00)	0.38(Martynenko,2014)

*Values are averages of determinations for 100 blueberries.

Table 2

Average moisture content, initial and final sugar concentration of fresh and frozen blueberries and solutions, solid gain and water loss from fruit, osmotic duration and yields during osmotic dehydration at 50°C.

Fruit Type	Moisture Content		Solution Sugar		Fruit Sugar		Solid Gain	Water Loss	OD duration	Yield of OD
	(g/g of dry solid)		Concentration		Concentration		(g/g of dry solid)	(g/g of dry solid)	(min)	Fruit (%)
			(°Brix)		(°Brix)					
	Initial	Final	Initial	Final	Initial	Final				
Fresh	5.16	4.39	43.80	44.60	10.20	13.40	0.07	0.47	600	93.51
Blueberries	(0.20)	(0.69)					(0.02)	(0.08)		(1.51)
Frozen	5.70	3.97	41.80	38.60	12.50	32.90	0.23	1.81	420	76.33
Blueberries	(0.57)	(0.14)					(0.02)	(0.04)		(0.77)

Standard deviation in parentheses. Replicate of two results

Table 3

Average moisture content, initial and final sugar concentration of frozen blueberries and osmotic solutions (apple juice-based and sugar based), and solid gain in fruit and water loss during osmotic dehydration at 50°C.

Osmotic solution Type	Moisture Content (g/g of dry solid)		Osmotic Solution Concentration (°Brix)		Frozen Blueberry Sugar Concentration (°Brix)		Solid Gain (g/g of dry solid)	Water Loss (g/g of dry solid)	Yield of OD Fruit (%)
	Initial	Final	Initial	Final	Initial	Final			
Apple-Juice-Based Solution	5.70 (0.57)	2.99 (0.44)	42.70	37.90	13.40	30.30	0.53 (0.01)	1.91(0.01)	79.31(0.21)
Sugar-based Osmotic Solution	5.70 (0.57)	3.97 (0.14)	41.80	38.60	12.50	32.90	0.23 (0.02)	1.81 (0.04)	76.33 (0.77)

Standard deviation in parentheses. Replicate of two results

List of Figures

- 1- Fig. 1. Schematic diagram of a typical blueberry showing the axial dimensions a (x-axis), b (y-axis), and c (z-axis).
- 2- Fig. 2. Relative moisture (moisture ratio) over the drying period performed at isothermal temperature of 105 °C in a thermogravimetric analyzer in dry nitrogen for fresh and frozen (thawed) blueberries.
- 3- Fig. 3. Relationship between drying rate (g of water evaporated per gr of drying fruit per minute) and mean moisture content of fresh and frozen (thawed) blueberries during drying under isothermal temperature of 105 °C in the thermogravimetric analyzer under dry nitrogen.
- 4- Fig. 4. Variation of sugar concentrations over time for (a) fresh blueberries and its sugar-based osmotic solution and (b) frozen blueberries and its osmotic solution during osmotic dehydration performed at 50 °C.
- 5- Fig. 5. Increase in sugar concentrations for fresh and frozen blueberries during their osmotic dehydration with an osmotic solution at 50 °C.
- 6- Figure 6. Decrease in water activity and moisture content (MC) during air-drying of osmotically dehydrated fresh and frozen blueberries in a fruit dehydrator at 74 °C.
- 7- Fig. 7. Variation of sugar concentrations over time for (a) frozen blueberries and its sugar-based osmotic solution and (b) frozen blueberries and its apple juice-based osmotic solution during osmotic dehydration performed at 50 °C.
- 8- Fig. 8. Sugar gained over immersion time for frozen blueberries in (1) sugar and (2) apple juice concentrate osmotic solution.
- 9- Fig. 9. Decrease in water activity of osmotically dehydrated frozen blueberries using sugar-based and apple juice-based osmotic solutions in a fruit dehydrator at 74 °C

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Fig. 2. Relative moisture (moisture ratio) over the drying period performed at isothermal temperature of 105 °C in a thermogravimetric analyzer in dry nitrogen for fresh and frozen (thawed) blueberries

Fig. 3. Relationship between drying rate (g of water evaporated per gr of drying fruit per minute) and mean moisture content of fresh and frozen (thawed) blueberries during drying under isothermal temperature of 105 °C in the thermogravimetric analyzer under dry nitrogen

Fig.4. Variation of sugar concentrations over time for (a) fresh blueberries and its sugar-based osmotic solution and (b) frozen blueberries and its osmotic solution during osmotic dehydration performed at 50 °C

Fig. 5. Increase in sugar concentrations for fresh and frozen blueberries during their osmotic dehydration with an osmotic solution at 50 °C

Fig. 6. Decrease in water activity and moisture content (MC) during air-drying of osmotically dehydrated fresh and frozen blueberries in a fruit dehydrator at 74 °C

Fig. 7. Variation of sugar concentrations over time for (a) frozen blueberries and its sugar-based osmotic solution and (b) frozen blueberries and its apple juice-based osmotic solution during osmotic dehydration performed at 50 °C

Fig. 8. Sugar gained over immersion time for frozen blueberries in (1) sugar and (2) apple juice concentrate osmotic solution

Fig. 9. Decrease in water activity of osmotically dehydrated frozen blueberries using sugar-based and apple juice-based osmotic solutions in a fruit dehydrator at 74 °C