# Experimental values and correlations of some thermal properties of fresh and osmotically dehydrated stone fruits

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**Abstract:** Nectarines, plums and peaches were dehydrated with osmotic solutions (glucose at 60°Brix or sorbitol at 60°Brix) at temperature of 35ºC for scheduled periods (0.5, 1, 2, 4, 6, 8 h). Thermal properties of fresh and osmotically dehydrated fruits were measured by DSC where samples were heated from –40ºC to 40ºC at a rate of 2ºC $min^{-1}$. In this work, we obtained experimental data on the variation of water content and soluble solids during the osmotic dehydration of nectarines, peaches and plums in solutions of glucose or sorbitol; experimental data for enthalpy of fresh and osmotically dehydrated nectarines, plums and peaches as a function of temperature and type of osmotic solute; correlations for initial freezing temperature of osmotically dehydrated stone fruits as a function of water content and type of dehydrating solute and correlations between heat capacity or enthalpy and temperature for some fresh and osmotically dehydrated stone fruits.

**Keywords:** osmotic dehydration; stone fruits; correlations; initial freezing point; thermophysical properties.

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## 1 Introduction

Stone fruits (plums, peaches, nectarines, cherries and apricots) are widely consumed fresh or processed due to their nutritional value and desirable taste. Shelf life of these fresh fruits – even under refrigeration – is limited, so many researches deal with treatments to prolong it. Among these, combined methods that include a first stage of osmotic dehydration (OD) are of interest due to the advantages of OD as a partial dehydration method.

OD is a mild process that consists of immersing fruits or vegetables – entire, peeled, portioned – in sugar/sugar-salt/alcohol concentrated aqueous solutions, where both partial dehydration of the tissue and solids uptake occur. The driving forces for the mass transfer are the differences in chemical potentials of water and solutes between the food tissue and the dehydrating solution (Agnelli et al., 2005). Water removal is carried out in the liquid phase – without phase change – and at almost ambient temperatures, which lowers energy use. During subsequent processing, energy requirements decrease due to the lower water content of the product.

The osmotic process can be used to increase availability of fruits and vegetables and – in some cases – to enhance the soluble solids content in the final product, as a pretreatment prior to air or microwave drying, refrigeration, freezing, preparing of candies and jams or frying (Torregiani, 1993). Moreover, the osmotic process can be employed in the development of minimally processed products, prolonging their shelf life, with a slight reduction in water activity and improving the microbiological stability, without changing considerably the quality characteristics of fresh fruit (Torres et al., 2008). The interest in OD prior to further processing is also due to the enhanced nutritional and sensory properties of the final products. OD, carried out at moderate temperatures, protects thermosensitive compounds such as flavours, pigments and vitamins. Also, as it prevents food from getting in contact with air, oxidation reactions and loss of volatile compounds are limited (Raoult-Wack, 1994).

The wide variety of raw food materials, with their different compositions and structures, sizes and shapes of pieces, and of operating conditions during OD (composition, temperature and agitation of dehydrating solution, contact time) determined the need for extensive experimental work to characterise each case (food, piece, solution, operating condition). Literature presents numerous papers on experimental data of OD of fruits and vegetables (Torregiani, 1993; Ibitwar et al., 2008; Yadav et al., 2012), but no general correlations are given for the variation of thermophysical properties during OD (Tocci and Mascheroni, 2008). It is of high scientific and technical value – when a later stage of freezing or refrigeration is applied – to develop simple equations to predict the variation of heat capacity (Cp) and enthalpy (H) with temperature and water content and initial freezing point (Tcr) with water content, valid independently of the fruit or vegetable used. Only some papers from this research group present results for these properties for some types of fruits and few osmotic agents (Tocci and Mascheroni, 1998, 2001, 2008; Rodríguez and Mascheroni, 2012).

In the case of the stone fruits studied in this work – plums, nectarines and peaches –, several recent research papers studied the application of the OD process to plums (Ibitwar et al., 2008; Koocheki and Azarpazhooh, 2010; Rodríguez et al., 2010a; Tarhan, 2007), nectarines (Araujo, 2004; Pavkov et al., 2011; Rodríguez et al., 2013, 2010b) and peaches (Germer et al., 2010; Mota, 2005; Sahari et al., 2006; Yadav et al., 2012), among others; but none of them dealt with the determination of values of thermophysical properties, except one paper on thermal properties of fresh and air-dried stone fruits (Phomkong et al., 2006).

Therefore, the objectives of this work were to determine enthalpy and heat capacity of fresh and osmotically dehydrated plums, nectarines and peaches with different water contents and soluble solids contents in the range of –40°C to 40°C and obtain correlations for enthalpy and heat capacity as a function of water content and temperature and of initial freezing point as a function of water content for fresh and osmodehydrated stone fruits.

## 2 Materials and methods

### *2.1 Osmotic dehydration*

Nectarines cv August Red, plums cv Larry-Ann and peaches cv O'Henrry, were bought at the market and immediately refrigerated. Dehydrations were performed the next day, after slicing the fruits to 2 mm samples. These were introduced in flasks with the osmotic solution (glucose at 60% or sorbitol at 60%) and shaken at constant temperature of 35°C for the scheduled periods (0.5, 1, 2, 4, 6, 8 h) in a thermostatic bath FERCA, model TT 400 with linear stirring (100 cycles per minute).

Water content (WC) of fresh and OD samples was determined by drying in a vacuum oven (Gallenkamp, Model OVA031 XX1.5, UK) at 70°C + 2°C until a constant weight was reached.

Soluble solids content (SS) was measured in an Abbe refractometer (Bellinham + Stanley Limited N° A77341, England) and read in °Brix.

### *2.2 Differential scanning calorimetry determinations*

Samples (fresh or dehydrated) were mashed and homogenised and small sub-samples (10–25 mg each) were then sealed in the specific sample holders (capsules) for differential scanning calorimetry (DSC) evaluation. Three samples were prepared for each dehydration time and the results of these runs were averaged to get values for data interpretation.

Tests were conducted on a differential scanning calorimeter with automatic data recording and evaluation (DSC model Q100, TA Instruments, New Castle, Delaware USA). Samples were stabilised at –40°C and heated from –40°C to 40°C at a rate of 2°C $\text{min}^{-1}$, as this low heating rate minimises the temperature lags likely to occur in the event of a poor thermal contact of the sample-capsule-base system. The lower temperature level of –40°C was considered sufficient so as to cover the typical temperature range of most industrial processes. Besides, below –40°C, the additional amount of water frozen is negligible. For frozen foods, measurements are always performed heating (thawing) the samples, because during freezing it is common to have subfreezing, which leads to erroneous values in measured properties.

Heat capacity Cp was determined with the Universal Analysis V1.7F software (provided by TA Instruments).

Enthalpy H was calculated by integrating the experimental data of Cp vs. temperature, taking –40°C as the datum $H_{-40} = 0$.

Initial freezing temperature Tcr was determined as the position of the thawing peak in the experimental heat capacity vs. temperature plot, as described in a previous work (Tocci and Mascheroni, 2008). This is probably the less accurate of the measured properties, because this point cannot be determined with good precision even when the DSC was calibrated against pure materials of known melting points (sapphire and bidistilled water).

Enthalpy variation between –40°C and Tcr (Hcr) was determined for each sample. It is a value of technological interest, as can be considered as a characteristic value for the total enthalpy change during freezing or thawing, besides, it also appears as a parameter in some prediction equations.

## 2.3 Prediction equations

### 2.3.1 Enthalpy and specific heat

Most equations from the literature for the prediction of enthalpy and heat capacity in the temperature below Tcr are based on the theoretical Clausius-Clapeyron relationship (Jowitt et al., 1983; Mohsenin, 1980; Rha, 1975). To be able to deal with experimental data, some empirical versions are used because neither the average molecular weight nor the exact composition of food solutes are known, and this relationship also implies ideal behaviour of solutes, which is not true for dehydrated foods. Therefore, the prediction equations used were similar to those proposed by Schwartzberg (1976), adapted by Succar and Hayakawa (1983) for fresh foods and already used by the present authors for OD kiwi and strawberry.

$$Cp = A + B/(-T)^n \tag{1}$$

$$H = A(T+40) + B/(n-1)\left(1/(-T)^{n-1} - 1/40^{n-1}\right) + C \tag{2}$$

for $T < Tcr$ and

$$Cp = E \tag{3}$$

$$H = D + E(T - Tcr) \tag{4}$$

for $T \geq Tcr$,

where $Cp$ is the heat capacity (kJ/(kg °C)); $H$ is the enthalpy (kJ/kg); $T$ is the temperature (°C); $Tcr$ is the initial freezing temperature (°C); $A$, $B$, $C$, $D$, $E$ and $n$ are fitted constants. Under ideal measuring conditions, the value of $D$ should be equal to $Hcr$, that is the enthalpy at $Tcr$.

For high water content foods, such as fresh fruits, vegetables and meats, the values of $n$ and $C$ in equations (1) and (2) should be close to 2 and 0, respectively (Succar and Hayakawa, 1983). Dehydrated and – mainly – osmotically dehydrated foods are expected to have lower values of n due to deviations of the behaviour of the food as compared to that of ideal solutions (Succar and Hayakawa, 1983).

# 3 Results and discussion

## 3.1 Experimental data of SS vs WC

Table 1 presents the experimental data of SS vs WC for nectarines, peaches and plums dehydrated in sorbitol or glucose solutions. Each datum is the average of three determinations.

**Table 1** Experimental values of WC and SS for nectarines, peaches and plums dehydrated in glucose or sorbitol solutions

| *Nectarines* | *Glucose* | | *Sorbitol* | |
|---|---|---|---|---|
| *Time (min)* | *WC (%)* | *SS (%)* | *WC (%)* | *SS (%)* |
| 0 | 87.66 | 10.13 | 87.66 | 10.13 |
| 30 | 79.45 | 20.47 | 70.59 | 27.70 |
| 60 | 71.51 | 25.23 | 62.45 | 31.23 |
| 120 | 65.82 | 27.70 | 54.32 | 37.63 |
| 240 | 61.24 | 35.17 | 46.21 | 45.27 |
| 360 | 54.96 | 41.83 | 41.66 | 52.80 |
| 480 | 51.18 | 46.03 | 40.64 | 56.67 |
| *Peaches* | *Glucose* | | *Sorbitol* | |
| *Time (min)* | *WC (%)* | *SS (%)* | *WC (%)* | *SS (%)* |
| 0 | 85.16 | 13.10 | 85.16 | 13.10 |
| 30 | 79.63 | 19.15 | 73.43 | 21.87 |
| 60 | 74.26 | 28.40 | 68.85 | 28.70 |
| 120 | 71.01 | 27.97 | 53.90 | 39.33 |
| 240 | 63.67 | 30.30 | 44.17 | 42.57 |
| 360 | 52.35 | 36.80 | 42.43 | 51.67 |
| 480 | 46.95 | 40.30 | 42.19 | 56.50 |
| *Plums* | *Glucose* | | *Sorbitol* | |
| *Time (min)* | *WC (%)* | *SS (%)* | *WC (%)* | *SS (%)* |
| 0 | 86.21 | 10.83 | 86.21 | 10.83 |
| 30 | 80.33 | 15.90 | 66.89 | 30.63 |
| 60 | 72.68 | 17.60 | 56.87 | 36.10 |
| 120 | 71.49 | 20.03 | 50.71 | 47.73 |
| 240 | 59.66 | 30.97 | 41.38 | 52.97 |
| 360 | 57.57 | 35.67 | 38.97 | 57.15 |
| 480 | 47.16 | 45.90 | 38.68 | 58.80 |

As can be seen, for all fruits and times WC is always lower when using sorbitol as dehydrating agent, meanwhile SG is always higher for the same solute.

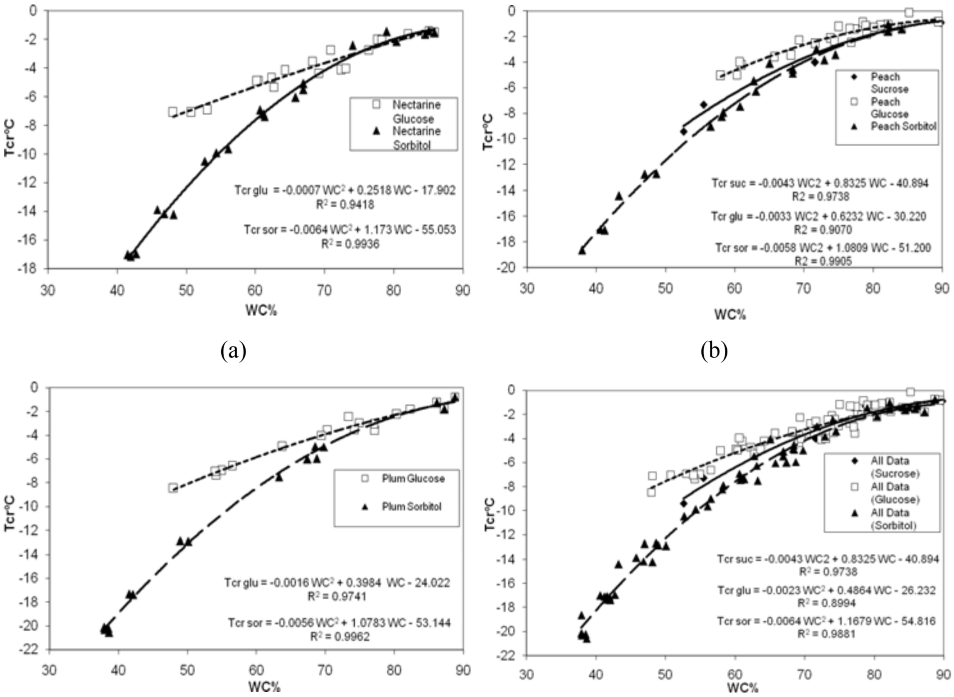
## 3.2 *Initial freezing temperature as a function of water content*

It is to be expected that Tcr is a function of composition, not only of WC but also of SS and of the type of solute. Probably, the dependence on SS is implicit due to its linear relation to WC (Rodríguez and Mascheroni, 2012). As expressed previously there is little information in literature on Tcr of OD fruits, which was summarised in Rodríguez and Mascheroni (2012).

The DSC experiments were also used to determine the initial freezing point (Tcr) of nectarine, peach and plum slices, fresh or osmodehydrated in solutions of glucose or sorbitol. These results are presented in Figures 1(a)–1(c) together with the second order polynomial regressions of Tcr vs. WC, that were those of higher regression coefficients of the different types tested (linear, logarithmic, exponential, polynomial). As can be seen, high accuracy is obtained for each solute, with only one parameter (WC).

As the data of Tcr vs. WC for the three fruits in different solutes followed the same trend found in Rodríguez and Mascheroni (2012), we looked for general correlations of Tcr vs. WC. In this regard, all the available data for the three fruits and different solutes were used, including those of Tocci and Mascheroni (2001) for peaches dehydrated in sucrose and glucose.

**Figure 1** Experimental data (symbols) and regression equations (lines) for the variation of initial freezing point Tcr with WC for different fruits and solutes, (a) nectarines (b) peaches (c) plums (d) all data



(a) (b)

(c) (d)

A careful discrimination of results showed that all the data for different fruits but with the same solute in the dehydrating solution can be included in only one polynomial correlation with high regression coefficient $R^2$ for the cases of sorbitol (0.9881) and sucrose (0.9738) and a little lower for glucose (0.8994). Figure 1(d) presents the experimental data and the polynomial regressions obtained.

These results clearly show that both, water content and type of solute, have direct influence on freezing point depression. Sorbitol – that has similar molecular weight to glucose – has two polar hydroxyl groups, which clearly bound water, provoking a lower water activity and the corresponding descent in Tcr for equal WC.

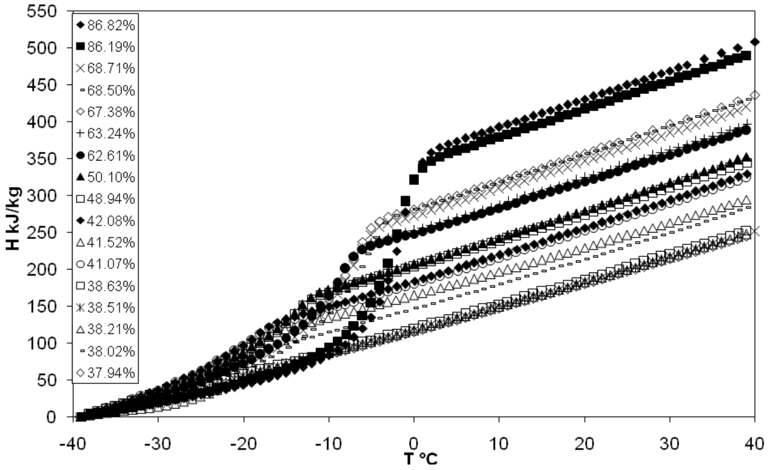
On the other side, fruit type seems to have no discernible influence, which is coherent with similar results for the relation SS vs WC determined in Rodríguez and Mascheroni (2012).

In any case, a much higher amount of experimental data is necessary to produce sufficient information to get more general and accurate relationships.

### *3.3 Heat capacity and enthalpy as a function of water content*

As previously described, we determined the DSC pattern of nectarines, peaches and plums, fresh or osmodehydrated in solutions of glucose or sorbitol, covering the range of WC from that of the fresh fruit (85 to 90%) to 37%, a very low value, below the limit of unfreezability. Examples of the obtained curves of H vs T are presented in Figure 2, which corresponds to plums dehydrated in sorbitol. All the measured data follow the expected trend, with lower H at lower T and WC, with the change of slope (at Tcr) moving to more negative values as WC diminishes, and with no phase change for the lower WC contents.

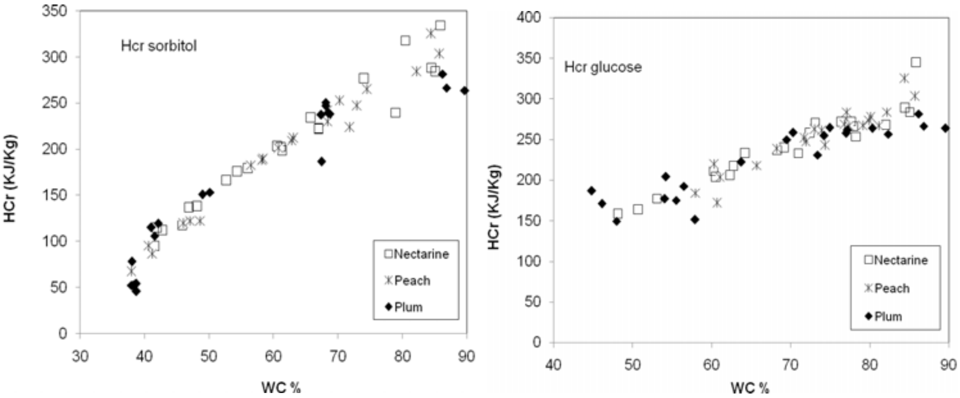
**Figure 2** Experimental values of H vs T for plums with different values of WC, after partial dehydration in sorbitol solution



The experimental values of Hcr (H at Tcr) for each sample, descriptive of the amount of energy needed for freezing the fruit, are given in Figure 3 for fruits dehydrated in glucose or sorbitol solutions. It can be clearly seen from the figure that there is less frozen water in fruits dehydrated in sorbitol than in those treated with glucose (lower Hcr for equal WC), which directly correlates to the previously observed dependence of Tcr with solute type.

As expressed previously we made the regression of experimental data of Cp and H vs T following the procedures of Schwartzberg (1976) and Succar and Hayakawa (1983) [equations (1) to (4)], that were developed for untreated foods. This method already showed to be valid for OD materials, when used for kiwi and strawberry dehydrated in different solutes (Rodríguez and Mascheroni, 2012; Tocci and Mascheroni, 2008).

**Figure 3** Experimental values of Hcr of osmodehydrated nectarines, peaches and plums as a function of WC; solute used, (a) sorbitol (b) glucose



(a) (b)

**Table 2** Regression values and correlation coefficients for the constants in Succar and Hayakawa equations for prediction of enthalpy

| | | | | *Glucose* | | | | |
|---|---|---|---|---|---|---|---|---|
| *WC* | *A* | *B* | *n* | *C* | $R^2$ | *D* | *E* | $R^2$ |
| *%* | *kJ/(kg °C)* | *kJ/(kg* $°C^{1-n}$*)* | --- | *kJ/kg* | --- | *kJ/kg* | *kJ/(kg °C)* | --- |
| 85.90 | 1.434 | 281.622 | 1.577 | –1.336 | 1 | 427.20 | 4.271 | 0.988 |
| 85.03 | 1.030 | 188.058 | 1.463 | –1.046 | 1 | 350.90 | 4.035 | 1 |
| 84.50 | 1.077 | 208.244 | 1.482 | –1.224 | 1 | 354.10 | 4.197 | 0.999 |
| 82.01 | 0.972 | 186.961 | 1.451 | –1.197 | 1 | 334.60 | 3.877 | 1 |
| 78.28 | 1.169 | 249.367 | 1.563 | –1.936 | 1 | 308.70 | 3.683 | 1 |
| 77.97 | 0.351 | 158.058 | 1.278 | –0.581 | 1 | 321.40 | 3.838 | 0.999 |
| 77.68 | 0.026 | 196.464 | 1.364 | 0.828 | 1 | 333.36 | 3.951 | 1 |
| 76.43 | 1.473 | 375.971 | 1.678 | –2.366 | 1 | 325.60 | 4.003 | 0.999 |
| 73.08 | 1.386 | 772.098 | 1.847 | –1.246 | 1 | 323.16 | 4.033 | 1 |
| 72.43 | 1.422 | 436.086 | 1.626 | –2.157 | 1 | 306.80 | 4.057 | 0.999 |
| 70.93 | 0.244 | 297.264 | 1.541 | –0.572 | 1 | 284.28 | 3.509 | 0.999 |
| 69.19 | 1.172 | 346.966 | 1.528 | –1.932 | 1 | 283.00 | 3.691 | 0.999 |
| 68.29 | 0.810 | 251.678 | 1.446 | –2.195 | 1 | 274.80 | 3.564 | 0.999 |
| 64.26 | 0.649 | 487.516 | 1.677 | –1.397 | 1 | 269.04 | 3.523 | 0.999 |
| 62.72 | 0.837 | 308.065 | 1.452 | –2.427 | 1 | 257.40 | 3.579 | 0.999 |
| 62.30 | 0.364 | 191.881 | 1.286 | –2.170 | 1 | 244.10 | 3.316 | 0.999 |
| 60.43 | 0.311 | 197.513 | 1.289 | –2.289 | 1 | 242.00 | 3.310 | 0.999 |
| 60.22 | 0.482 | 451.595 | 1.611 | –1.488 | 1 | 252.48 | 3.524 | 0.999 |
| 53.06 | 0.066 | 201.917 | 1.245 | –2.377 | 1 | 213.80 | 3.198 | 0.999 |

Notes: Fruit: nectarine; solutes: glucose and sorbitol.

**Table 2** Regression values and correlation coefficients for the constants in Succar and Hayakawa equations for prediction of enthalpy (continued)

| *Sorbitol* | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| *WC* | *A* | *B* | *n* | *C* | *R2* | *D* | *E* | $R^2$ |
| *%* | *kJ/(kg °C)* | *kJ/(kg °C*$^{1-n}$*)* | --- | *kJ/kg* | --- | *kJ/kg* | *kJ/(kg °C)* | --- |
| 50.73 | 0.218 | 214.100 | 1.304 | –2.207 | 1 | 195.90 | 3.006 | 0.999 |
| 48.11 | 0.214 | 429.666 | 1.570 | –1.380 | 1 | 192.48 | 3.118 | 0.999 |
| 85.90 | 1.434 | 281.622 | 1.577 | –1.336 | 1 | 427.20 | 4.271 | 0.988 |
| 85.03 | 1.030 | 188.058 | 1.463 | –1.046 | 1 | 350.90 | 4.035 | 1 |
| 84.50 | 1.077 | 208.244 | 1.482 | –1.224 | 1 | 354.10 | 4.197 | 0.999 |
| 80.39 | 1.588 | 342.055 | 1.623 | –2.273 | 1 | 398.76 | 3.956 | 0.999 |
| 78.97 | –1.534 | 110.180 | 0.941 | 0.167 | 0.999 | 281.90 | 3.706 | 0.999 |
| 74.02 | 1.411 | 299.827 | 1.597 | –2.094 | 1 | 337.50 | 4.246 | 0.999 |
| 66.93 | 1.283 | 456.181 | 1.617 | –2.095 | 1 | 257.90 | 3.684 | 0.999 |
| 66.93 | 1.100 | 630.762 | 1.746 | –1.249 | 1 | 257.76 | 3.722 | 1 |
| 65.77 | 1.421 | 511.055 | 1.610 | –2.303 | 1 | 274.90 | 3.965 | 0.999 |
| 61.31 | 1.025 | 394.383 | 1.493 | –1.999 | 1 | 238.80 | 3.415 | 0.999 |
| 61.16 | 1.207 | 1.181.747 | 1.910 | –1.276 | 1 | 246.24 | 3.420 | 0.999 |
| 60.62 | 0.992 | 386.358 | 1.493 | –1.972 | 1 | 242.40 | 3.439 | 0.999 |
| 56.03 | 0.867 | 412.112 | 1.452 | –2.000 | 1 | 221.40 | 3.424 | 0.999 |
| 54.36 | 0.790 | 421.607 | 1.453 | –1.988 | 1 | 218.80 | 3.642 | 0.999 |
| 52.69 | 0.948 | 1.795.580 | 2.002 | –1.573 | 1 | 211.56 | 3.615 | 0.999 |
| 48.14 | –0.805 | 189.916 | 1.063 | –1.876 | 1 | 191.80 | 3.468 | 0.999 |
| 46.83 | –0.443 | 232.293 | 1.150 | –1.940 | 1 | 189.30 | 3.364 | 0.999 |
| 45.79 | 1.240 | 1.527.437 | 1.935 | –1.542 | 1 | 162.24 | 3.169 | 0.999 |
| 42.75 | 0.431 | 483.041 | 1.422 | –1.994 | 1 | 169.00 | 3.164 | 0.999 |
| 41.95 | –3.326 | 101.780 | 0.755 | –1.875 | 1 | 174.60 | 3.312 | 0.999 |
| 41.57 | 0.643 | 610.982 | 1.574 | –1.505 | 1 | 145.32 | 3.264 | 0.998 |

Notes: Fruit: nectarine; solutes: glucose and sorbitol.

Table 2 presents the obtained constants for equations (1) to (4), together with the respective regression coefficients for the case of nectarines. Similar results (not shown due to space restrictions) were obtained for peaches and plums. As can be seen, high coefficients were obtained for all samples, showing that these equations are simple and accurate for prediction purposes and that their theoretical basis is sound, because their use could be extended to products that were partially dehydrated and at the same time enriched in solutes.

At present no simple relations of A, B, C, n, D, E as a function of WC and solute type could be obtained, which would generalise the model and simplify prediction procedures. Much more experimental data on different osmodehydrated fruits (and diverse solutes) is needed if such a general relation is sought.

## 4 Conclusions

Sorbitol and glucose were tested as dehydrating agents for some stone fruits (nectarine, peach, plum). In all cases sorbitol showed a higher dehydrating and absorption capacity for equal dehydrating conditions (sample size, temperature, solute concentration, stirring) and contact time.

General correlations were obtained for the values of Tcr as a function of WC and type of solute, independent of the type of stone fruit. Good accuracy was obtained using quadratic regression equations predicting Tcr as an only function of WC for each solute. Sorbitol showed to have a higher freezing point depression capacity for equal water content, respect to glucose and sucrose.

Measured variations of H with T for samples dehydrated to different WC in sorbitol or glucose showed the expected trend of lower H with lower water content. When using sorbitol, the most dehydrated samples were completely unfreezable – independently of the type of fruit –.

The variation of H with T could be accurately predicted using the model of Succar and Hayakawa, originally not intended to be used with dehydrated and/or infused foods.

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