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Osmo-frozen fruits: mass transfer and quality evaluation

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Abstract

The quality of osmofrozen pears, kiwis, strawberries and apples was studied through measurements of colour, drip-loss and texture after each step of the combined process, that is, after osmotic dehydration for different periods of time and after freezing in a conventional air-blast tunnel at −40 °C. The results were compared to those obtained for fresh products. The effect of the osmotic solution on the whole process has also been investigated using different osmotic solutions (sucrose, glucose and corn syrup mixtures). The evolution of mass transfer was measured through the variation in time of weight loss and the solid gain. The osmotic dehydration prior to freezing demonstrated to be useful for limiting drip loss and, in some cases, to decrease colour change and improve texture. The choice of the dehydrating agent and the usefulness of the pre-treatment depend on the application intended for the final product.

1. Introduction

Osmotic dehydration for partial dehydration of food materials, especially of fruits and vegetables, used previous to freezing leads to substantial energy savings. The osmotic dehydration step consists of a simple immersion of the foodstuff in a highly concentrated solution of salt and/or sugar close to room temperature. As a consequence of the concentration gradient, the water from the food flows towards the solution and, in the inverse sense, the solutes from the solution to the product.

In fact, during dehydration, the water removal is carried out without phase change and, in liquid phase, which enhance heat and mass transfer coefficients (Lenart & Lewicki, 1988). Besides, during freezing, the energy requirements decrease due to the lower level of water in the product.

In addition, the interest of a dehydration step previous to freezing is also related to the nutritional and organoleptic properties of the final products (Ponting, Walters, Forrey, Jackson, & Stanley, 1966). Osmotic dehydration, carried out at moderate temperatures, protects thermosensible compounds as flavors, pigments and vitamins (Ponting et al., 1966; Vial, Guilbert, & Cuq, 1991). Also, as it prevents food from getting in contact with air, the oxidation reactions (Raoult-Wack, 1994) and loss of volatile compounds (Ponting, 1973) are limited.

The type of the concentrated solution plays an important role in the osmotic dehydration affecting its rate and the solid uptake of the food (Garrote & Bertone, 1989; Beristain, Azuara, Cortés, & García, 1990; Rastogi & Raghavarao, 1994). So far, the most frequently used dehydrating agents are salts, sugars and corn syrups. Salts are used mainly for vegetables while sugars and corn syrups, are used for fruits.

The present study investigates the quality of osmotically dehydrated and frozen pears, kiwis, strawberries and apples through measurements of colour, drip loss and texture of the products at different stages of the process, that is, after dehydration for different periods of time and after freezing in a conventional air-blast tunnel at −40 °C. The results were compared to those obtained for fresh products. The effect of the osmotic solution on the whole process has also been investigated using different osmotic syrups.

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2. Experimental

2.1. Dehydrofreezing experiments

Experiments of osmotic dehydration were performed on pears (Packham's), apples (Granny Smith), strawberries (Cv. Selva) and kiwis (Hayward) with the geometry and sample sizes indicated in Table 1.

The samples were submerged in the dehydrating solution at 30 °C for different immersion times. The osmotic concentration agents were sucrose (commercial grade) and the syrups showed in Table 2. The concentrations of the osmotic solutions used in the experiments are presented in Table 3.

The evolution of mass transfer was measured through the variation in time of weight loss (WR), total solids (TS) and soluble solids content (°Brix). From these values, we can evaluate the parameters usually used to follow the dehydrating process:

Water loss:

$$\mathrm{WL} = \left[\left(1 - \frac{\mathrm{TS}^\circ}{100}\right) - \left(1 - \frac{\mathrm{TS}}{100}\right)\left(1 - \frac{\mathrm{WR}}{100}\right)\right] \times 100$$

Solid gain:

$$\mathrm{SG} = \left[\left(1 - \frac{\mathrm{WR}}{100}\right)\frac{\mathrm{TS}}{100} - \frac{\mathrm{TS}^\circ}{100}\right] \times 100$$

where TS° is the initial value of the solid content.

Table 1
Geometry and sizes of the fruit samples treated under dehydrating agents (d = diameter, l = thickness, s = side and w = weight)

Fruit	Geometry	Size or weight
Pear	Disk	$d = 2$ cm $l = 1$ cm
Strawberry	Whole fruit	$w = 10$ g
Apple	Cubes	$s = 1.5$ cm
Kiwi	Disk	$d = 4$ cm $l = 1$ cm

Table 2
Composition of the dehydrating agents (HMW sugars: high molecular weight sugars)

Designation	Glucose	Fructose	HMW sugars
Composition	Glucose (99.5%)	Fructose 19% Glucose 33% Maltose 8% HMW sugars 40%	Glucose 16% Maltose 12% HMW sugars 72%

Table 3
Composition of the osmotic solutions

Solute	Solute/water (wt)	°Brix
Sucrose	2/1	69
Glucose	1/1	47
Fructose	2/1	69
HMW sugars	2/1	69

After the dehydration step, the sample was frozen in a conventional air-blast tunnel at −40 °C. The progress of freezing was followed by the registration of temperature with a thermocouple placed in the centre of the product.

Changes in texture and colour were evaluated for dehydrated samples before and after freezing–thawing processes. Drip loss after thawing was evaluated on dehydrated and fresh samples.

2.2. Drip loss evaluation

Frozen products were laid over an absorbent paper and let to thaw at room temperature. Drip loss was then evaluated by periodically weighing the absorbent paper until a constant value was reached. The results were expressed as drip loss in dry basis as:

$$\mathrm{DL} = \frac{\omega_t - \omega_0}{\omega_\mathrm{S} \times \mathrm{TS}} \times 100$$

where ω_0 is the weight of the dry absorbent paper, ω_t the weight of the wet absorbent paper at time t, ω_S the weight of the sample and TS the total solids of the sample.

2.3. Texture tests

Puncture tests were performed in an INSTRON texturometer. Experiments were run with a metal probe of 2 mm diameter, and a rate and depth of penetration of 30 mm/s and 5 mm, respectively. In this case, the first discontinuity of the force–distance plot was evaluated to characterise the mechanical resistance of the samples.

The penetration force after dehydration and after freezing and thawing was normalised with the puncture force corresponding to the untreated sample (F_0) and depicted as a function of the dehydration time.

2.4. Colour characterisation

For the representation of colour in the three-dimension space, the CIE 1976 $L*$ $a*$ $b*$ system was adopted. Colour difference values $\Delta L*$, $\Delta a*$, $\Delta b*$ were calculated according to

$$\Delta L* = L* - L*_t,\ \Delta a* = a* - a*_t,\ \Delta b* = b* - b*_t,$$

where t represents colour taken as reference.

The total difference of colour is defined by the equation

$$\Delta E*_{ab} = \sqrt{\Delta a*^2 + \Delta b*^2 + \Delta L*^2}$$

Determination of chromatic co-ordinates was done by means of a Minolta CR100 analyser.

3. Results and discussion

3.1. Kiwis

The results of the osmodehydrating process on kiwi disks using the different solutions are presented in Fig. 1

(a) and (b). At the beginning of the process, the WL evolution from tissue (Fig. 1 (a)) with the dehydrating time is fast with fructose, sucrose and glucose solutions. This fact is expected as these solutions contains, totally or partially, sugars of low molecular weight (the so-called fructose solution contains a 52% of a monomer). On the contrary, the dehydrating process is slower with the HMW sugars solution, but always keeps growing. At 24 h, the WL with the glucose solution has reached its highest value but it continues to increase for the other solutions. It must be remarked that the glucose solution is a less concentrated solution as 47 °Brix is the highest concentration that may be achieved, so the dehydrating rate is really very fast. Another thing to be mentioned is that the value of WL with sucrose solution (69 °Brix) is not far from reaching its maximum as its rate has started to decline and the value of WL at the equilibrium would be around 0.56, as estimated from a quick fit of data based on a macroscopic expression of dehydration rate with a first order kinetics (Panatagiotou, Karathanos, & Maroulis, 1998).

The SG curves (Fig. 1(b)) are consistent with the solute molecular weight of the solutions. The solute uptake is significantly higher with glucose solution than with fructose, sucrose, and HMW solutions, being the value of the maximum uptake in that order. Thus, sucrose and HWM sugar solutions would be the recommended osmoactive solutions as they allow high dehydration with a minimum penetration of solids.

The drip loss on dry basis during thawing of the osmotically treated frozen samples is reported in Fig. 2. In this case, the results were normalised with the drip loss corresponding to the fresh untreated sample. It can be noted that the dehydrated samples exhibit an important decrease in drip loss for all osmotic solutions. This reduction increases with the dehydration time and the lower values are obtained for sucrose and fructose solutions at 12 h of process. These results are in agreement with the fact that for 24 h of treatment, these solutions provoked the highest loss of water and, thus, the damage produced by freezing on cell walls is reduced.

Puncture tests on the fresh and dehydrated samples before and after freezing are shown in Fig. 3. In this picture, the mechanical resistances of the samples have been normalised with the corresponding value to the fresh kiwi. A decrease in the penetration force is evident in treated samples, mostly for long dehydration time. Softening is greater for samples treated under glucose. After freezing and thawing, the penetration force diminishes in a very stark amount. Besides, in this case, softening does not depend on the osmoactive solution and it is the same for fresh and dehydrated samples. These results would indicate that, for short dehydration times, the remaining water content is still enough to form big ice crystals during freezing and thus damaging the cell structure. On the other hand, the pre-treatment itself would provoke cellular harm for long dehydration periods.

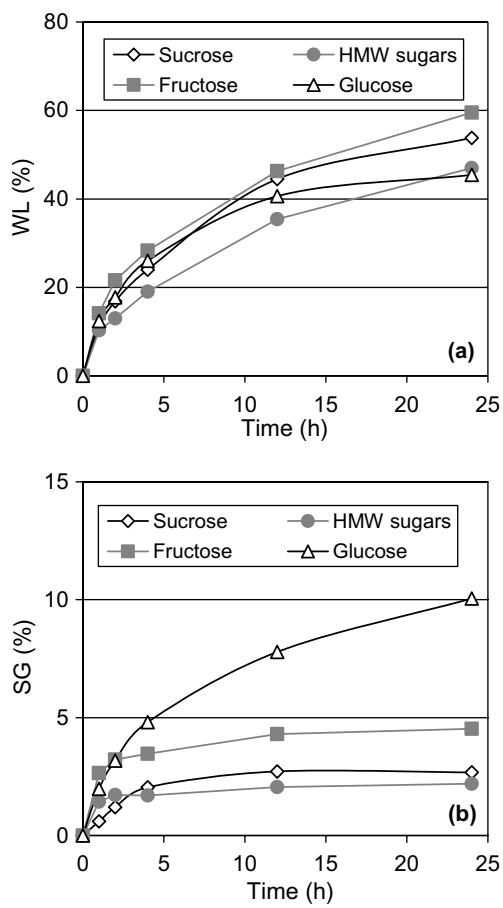

Fig. 1. (a) Water loss (WL) of kiwi slices during osmotic treatments in sucrose, glucose, fructose and HMW sugars solutions, (b) solid gain (SG) of kiwi slices during osmotic treatments in sucrose, glucose, Fructose and HMW sugar solutions.

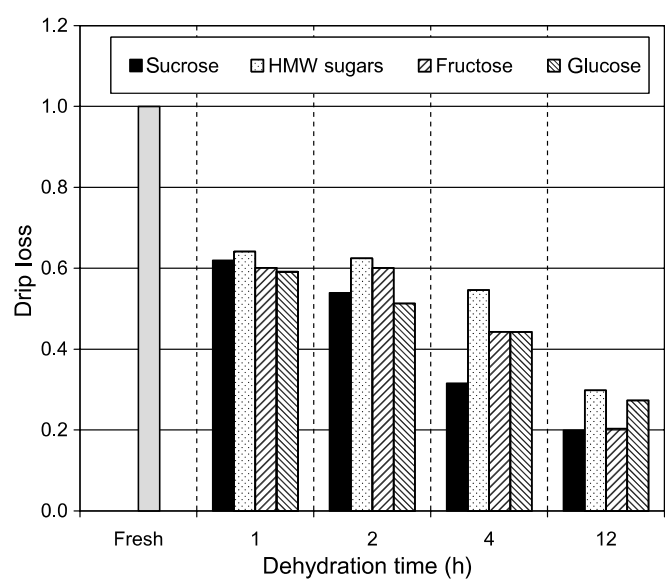

Fig. 2. Drip loss (relative to the fresh sample) during thawing of the frozen dehydrated kiwi slices.

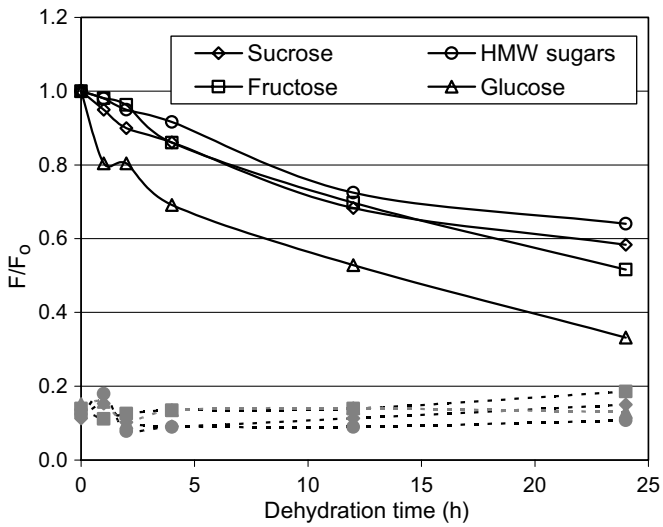

Fig. 3. Normalised puncture force (with the corresponding data to fresh samples) for fresh and dehydrated kiwi slices, before (void symbols) and after freezing (grey symbols).

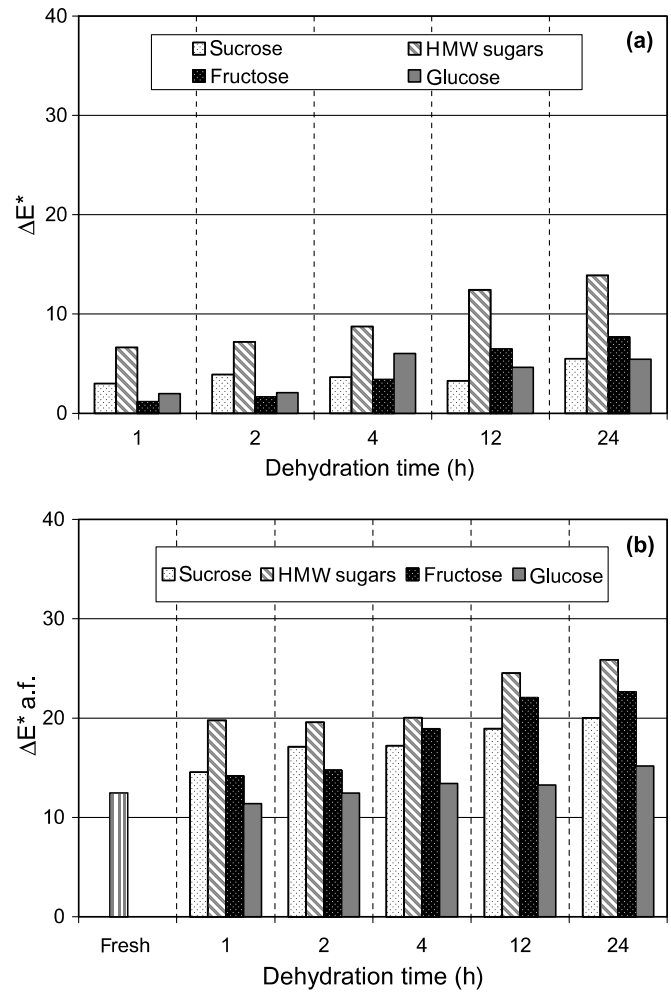

Fig. 4. Total difference of colour ($\Delta E*$) for kiwi slices (a) treated under different dehydrating agents. (b) treated under different dehydrating agents, frozen and thawed ($\Delta E*$ a.f.).

Data of colour tests on the frozen and thawed samples revealed important differences from the simply dehydrated ones. Fig. 4 presents the results of $\Delta E*$ evaluated before and after freezing (a.f.) ((a) and (b), respectively). During the dehydration step the total difference in colour in the treated samples before freezing, shows that the deviations are dependent on the dehydrating agent and operation time. Glucose and fructose are the solutions leading to the least colour change in the two first hours. After that period, the differences with sucrose disappear and only HMW sugars lead to a product with a greater shift in colour. The values of ΔE of frozen and then thawed samples indicate that freezing induces to colour changes by itself as shown for the fresh samples. The total change of colour on dehydrated samples is enhanced by freezing. After freezing and thawing, the samples turn out to be darker and less yellow than after dehydration meanwhile $\Delta a*$ seems not to have changed significantly. Only dehydrated samples under glucose present a similar colour to the non-frozen fresh kiwis (specially, for short periods of dehydration).

3.2. Apples

Fig. 5(a) and (b) presents the results of apple cubes osmodehydration under the different osmotic syrups. The WL and the SG follow, qualitatively, the same tendencies found for kiwis, but, in this case, the rate of both processes is higher. This fact can be ascribed to the differences in structure of each fruit but also to differences in shape and size between kiwi and apple samples. Besides, the lower SG curves obtained for apples as regards kiwi are consistent with the higher dehydration observed for the same period of time.

The normalised drip loss of the frozen and thawed samples, fresh and previously dehydrated, can be observed in Fig. 6. As in the case of kiwi, dehydrated samples show a smaller drip loss than fresh ones and it decreases with the process time, more rapidly than for kiwis. This is in agreement with the WL kinetics.

In contrast with the case of kiwi, puncture tests performed on fresh and dehydrated apples cubes revealed that the osmotic dehydration provokes a moderate hardening of the samples for short times of treatment (Fig. 7). Larger periods of dehydration time provoke a change in the textural characteristics of the samples and they turn to a semi-chewy material type (in agreement with Krokida, Karathanos, & Maroulis, 2000a). Texture improvement can be explained by cell compactness along with the concentration of cell constituents that occurs during osmotic dehydration, leading to a stronger tissue. The subsequent freezing step makes texture to decrease in a small extent after thawing (Fig. 7), nevertheless, the dehydrated samples exhibit, after thawing, a better or equal texture than the fresh untreated sample, except when using fructose solution.

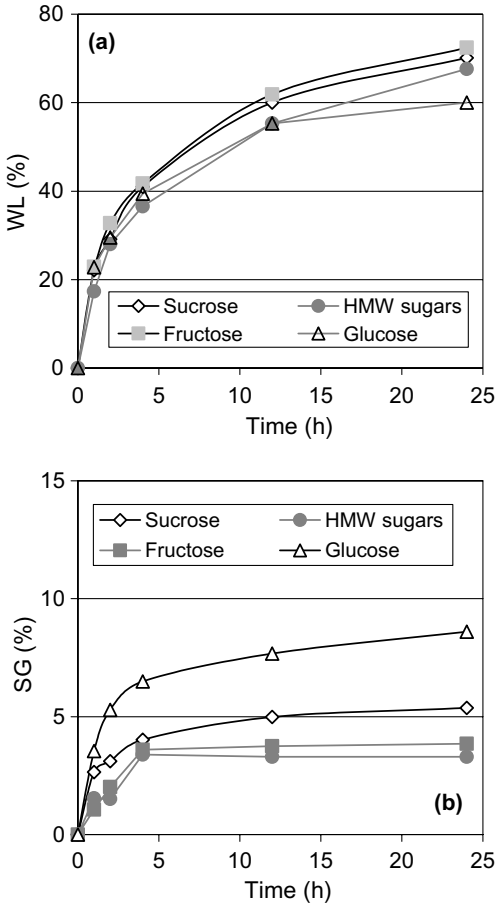

Fig. 5. (a) Water loss (WL) and (b) solid gain (SG) of apple cubes during osmotic treatments in sucrose, glucose, fructose and HMW sugars solutions.

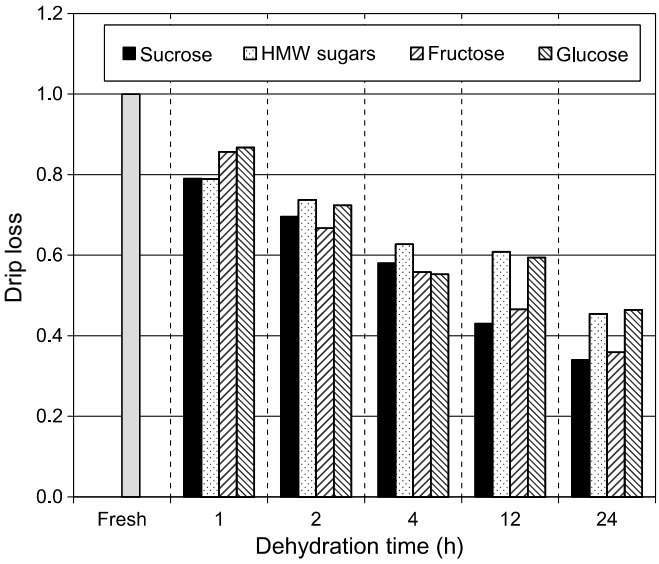

Fig. 6. Drip loss (relative to the fresh sample) during thawing of the frozen dehydrated cubes of apple with different dehydrating syrups.

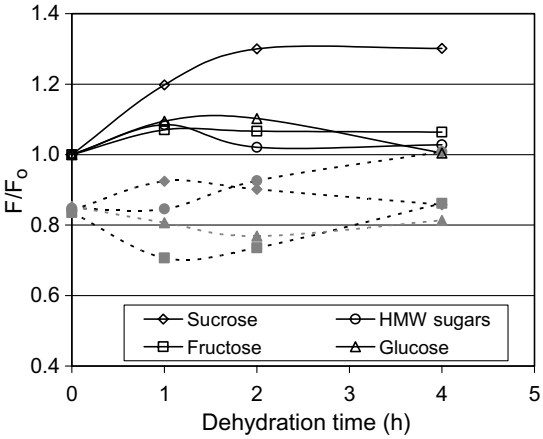

Fig. 7. Normalised puncture force (with the corresponding data to fresh samples) for fresh and dehydrated apple cubes before (void symbols) and after freezing (grey symbols).

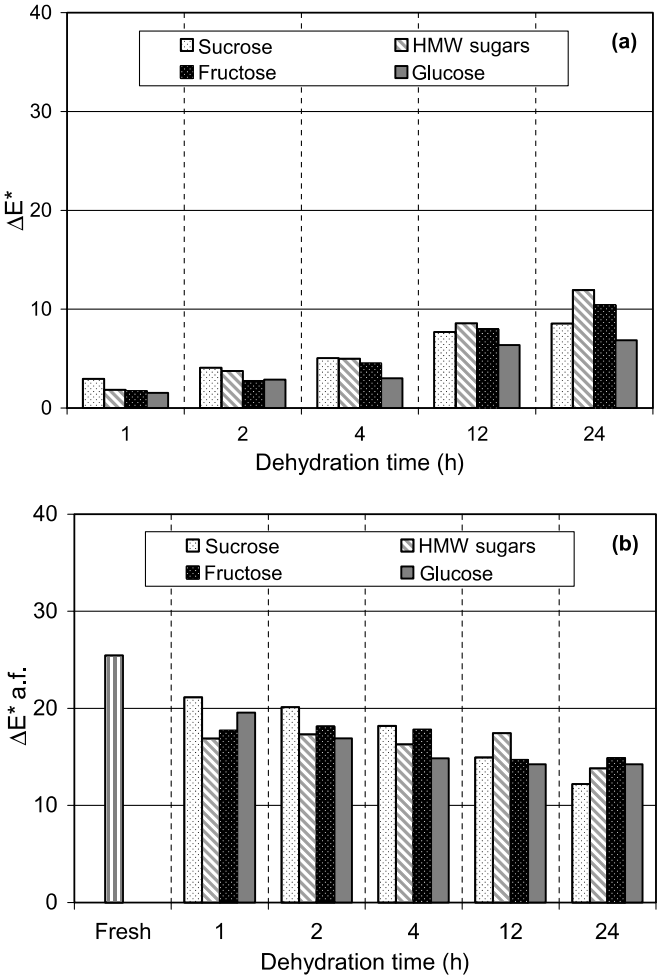

Fig. 8. Total difference of colour ($\Delta E*$) for apple cubes (a) treated under different dehydrating agents; (b) treated under different dehydrating agents, frozen and thawed ($\Delta E*$ a.f.).

Colour measurements on the fresh and dehydrated products before and after freezing are shown in Fig. 8. No significant changes are observed during dehydration with all the solutions. Only a small increase in $\Delta b*$ indicating an incipient yellowish shift was observed. The total difference in colour ($\Delta E*$) of dehydrated apple cubes as

regards fresh samples is presented in Fig. 8(a) confirming the precedent statement. This result is in agreement with Krokida, Karathanos, and Maroulis (2000b).

After freezing and thawing, the fresh samples showed a high decrease in $L*$ and $a*$ and an enhancement in $b*$ revealing an important browning, but, dehydrated samples exhibit browning to a less extent than the fresh one as a function of the increasing dehydration. The values of $\Delta E*$ shown in Fig. 8(b) support this affirmation (in agreement with Krokida et al., 2000b). All the solutions lead to dehydrated products that after freezing and thawing exhibit a lower total change of colour than the fresh apple.

3.3. Pears

Fig. 9 (a) and (b) present the WL and SG, respectively, during pear disks dehydration using different sugar solutions. The rate of WL from tissue presents a similar trend to the preceding fruits as regards the molecular weight of the dehydrating agent. The rate of WL is slightly lower to the correspondent for apple. SG, on the contrary, indicates an important increase in the penetration of solids in relation to the previous cases, especially for glucose.

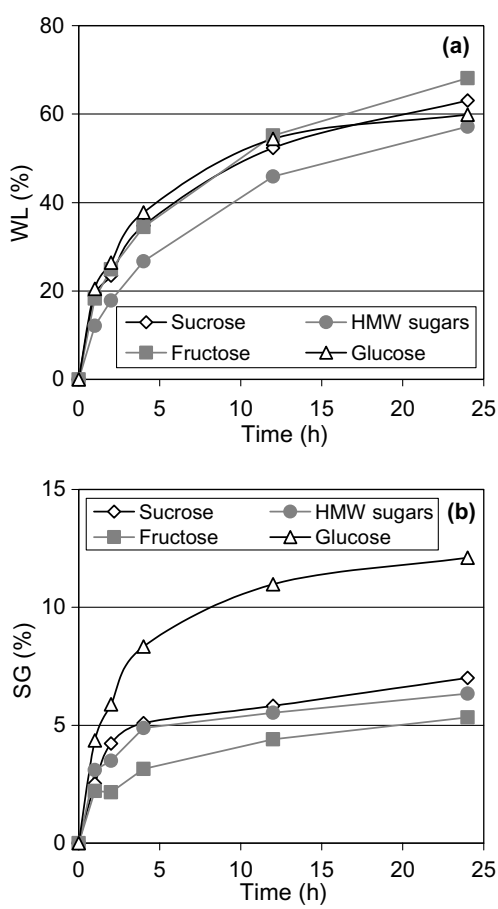

Fig. 9. (a) Water loss (WL) and (b) solid gain (SG) of pear disks during osmotic treatments in sucrose, glucose, fructose and HMW sugars solutions.

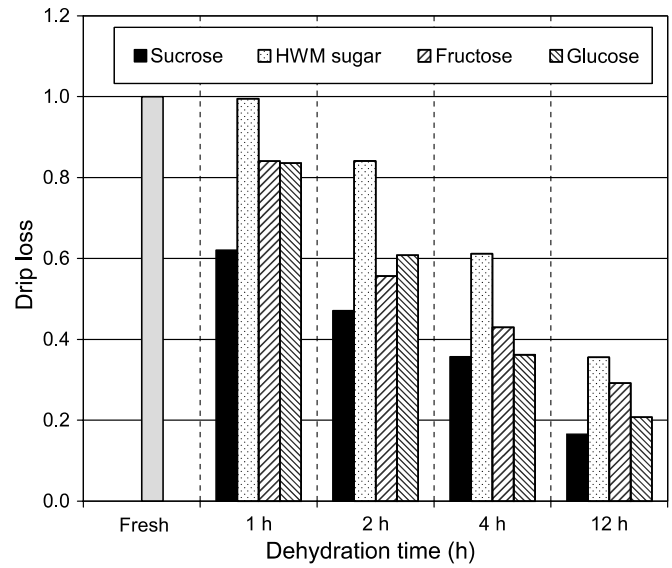

Fig. 10. Drip loss (relative to fresh sample) during thawing of the frozen dehydrated disks of pear with different dehydrating syrups.

Fig. 10 reports the drip loss during thawing of the osmotically treated frozen samples (normalised with the corresponding values to the untreated frozen one). Drip loss decreases with the dehydration time for all the samples. The lower value corresponds to the sucrose solution which presents an intermediate WL/SG rate. The lowest WL/SG value is for fructose and the highest for HMW sugars. So, it would seem that sucrose retains water during thawing. The same phenomenon has been observed in the former fruits as well, even if the final water content with other dehydrating agents has reached a similar level.

Puncture tests on the fresh and dehydrated samples before and after freezing are shown in Fig. 11. Up to 4 h of treatment, the osmotic dehydration does not really affect the hardness of the samples, except for those treated with sucrose solution, which presented a rapid and progressive decrease in the penetration force for short times of treatment. Larger periods of dehydration provoke a change in the textural characteristics of the samples and they turn to a semi-chewy type as for apples (in agreement with Krokida et al., 2000b).

The penetration force after freezing and thawing is very low, although higher than for kiwi but lower than for apples, and the same for untreated and treated samples. These results can be ascribed to the formation of ice crystals during freezing, damaging the cell structure.

Total change of colour of fresh and dehydrated products before and after freezing are reported in Fig. 12. No significant changes are observed during the dehydration step for all the solutions in agreement with Krokida et al. (2000a).

After freezing and thawing, the fresh samples showed a high decrease in $L*$ and an enhancement in $a*$ and $b*$ revealing an important browning. In turn, dehydrated samples present a similar browning after freezing and thawing, and have a light dependency with the increasing

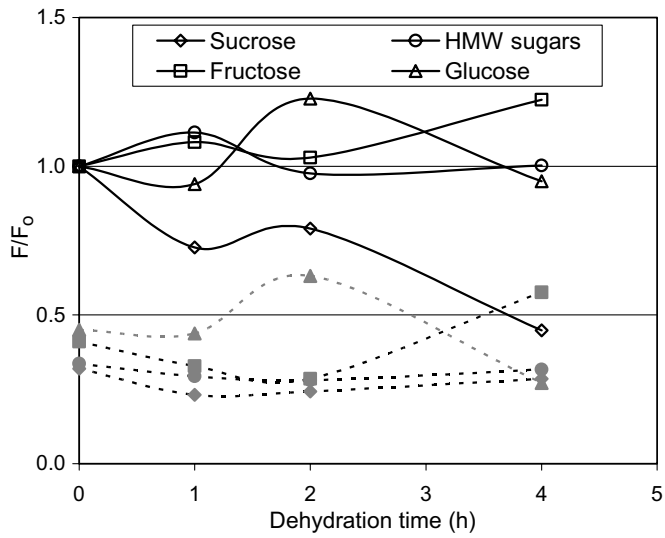

Fig. 11. Normalised puncture force (with the corresponding data to fresh samples) for fresh and dehydrated pear disks, before (void symbols) and after freezing (grey symbols).

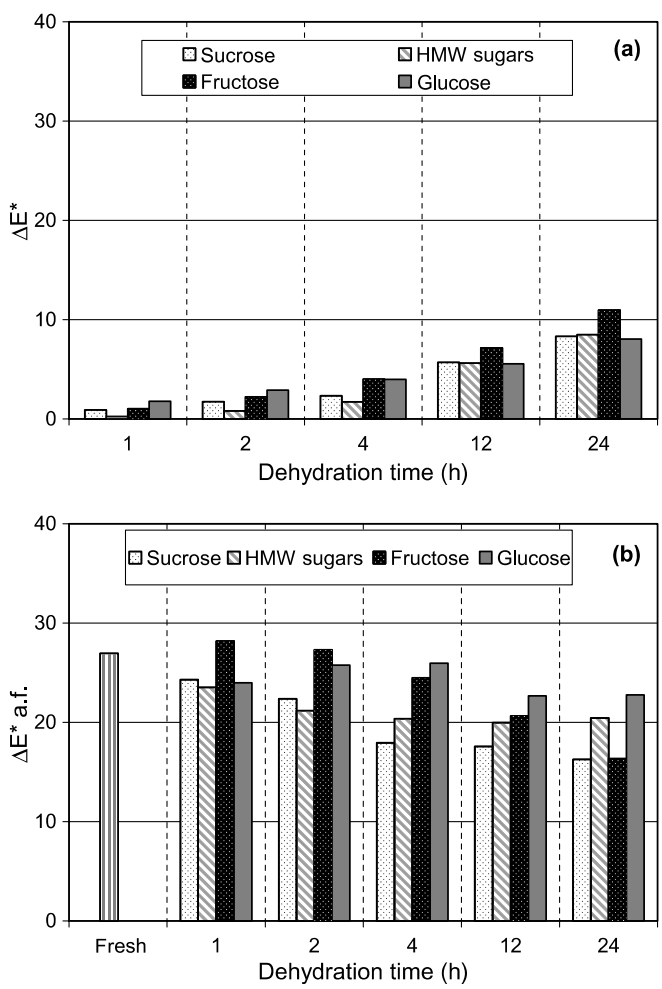

Fig. 12. Total difference of colour ($\Delta E*$) for pear disks (a) treated under different dehydrating agents. (b) treated under different dehydrating agents, frozen and thawed ($\Delta E*$ a.f.).

dehydration. The values of $\Delta E*$ a.f. (Fig. 12(b)) support this assertion (in agreement with Krokida et al., 2000a).

3.4. Strawberries

The results of the osmodehydrating process on strawberries using the different solutions are presented in Fig. 13. The WL evolution from tissue (Fig. 13(a)) with the dehydrating time is in agreement with the expected trend as regards the molecular weight of the solutions. These values represent the lowest rates found for the fruits considered in the present work, implying that whole strawberries are very hard to dehydrate due the resistance of their skin and seeds surrounding the pulp.

The SG curves (Fig. 13 (b)) reveal also very low rates of solid uptakes. Besides, they are not consistent with the solutes molecular weight of the solutions. At short times, the solute uptake is significantly higher for HMW sugars and fructose solutions, that is, the solutions containing solutes with higher average molecular weight. In contrast, the gain of solids shows a low rate of penetration for sucrose and glucose solutions. One possible explanation may be that the volumetric flow of the water leaving the product could

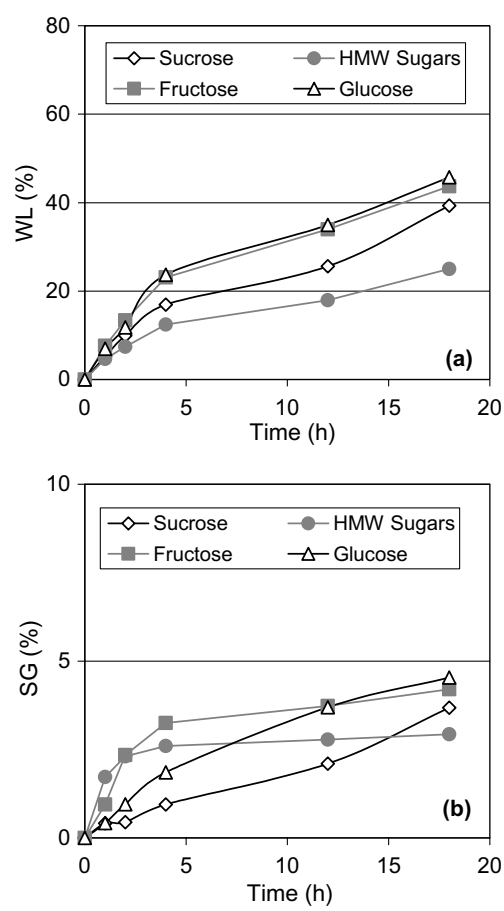

Fig. 13. (a) Water loss (WL) and (b) solid gain (SG) of strawberries during osmotic treatments in sucrose, glucose, fructose and HMW sugars solutions.

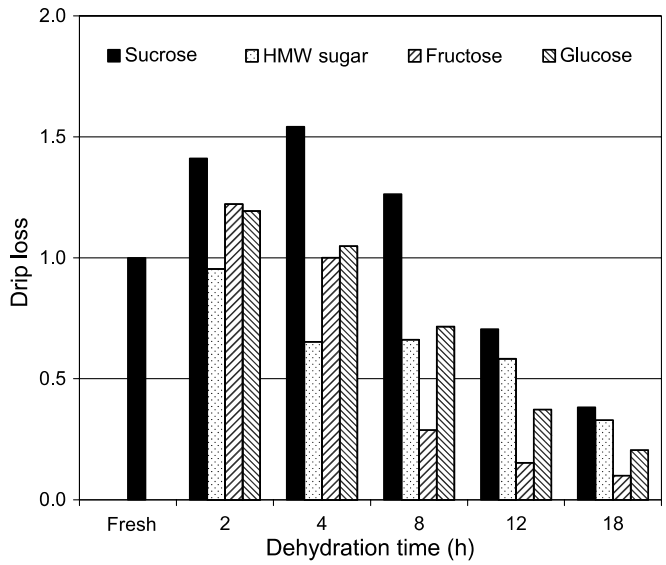

Fig. 14. Drip loss (relative to fresh sample) during thawing of the frozen dehydrated strawberries with different dehydrating syrups.

carry a part of the inner and/or the incoming solutes, partly compensating the entrance of solutes by the very slow diffusive process. Nevertheless, the solute uptake in the case of the HMW sugar solution reaches approximately its utmost value in a short time (4 h) and is kept low.

The drip loss during thawing of the osmotically treated frozen samples is reported in Fig. 14. It can be noted that, except for the samples treated under the HMW sugars solution, the dehydrated samples exhibit an important increase in drip loss for short dehydration times. The increment is enhanced first with the dehydration time and passes through a maximum, then is rapidly reduced to lower values than those for the untreated samples. The best result for 4 h dehydration is obtained with the HMW sugars solution. Longer periods of pre-treatment lead to a constant decrease in drip loss with the dehydrating time. After the maximum, fructose and glucose solutions conduct to products with the lowest drip loss. This is consistent with the fact that the penetration of glucose and fructose keep always growing meanwhile that of HMW sugars has stopped after the first hours (it must be remembered that drip loss is expressed in dry basis).

Puncture tests on the untreated and dehydrated samples before and after freezing are shown in Fig. 15. A decrease in the penetration force is evident in treated samples before freezing. Besides, no matter the treatment, the penetration force after freezing and thawing is low and the same for untreated and treated samples. These results can be ascribed to the fragility of the strawberry pulp towards both processes (freezing and dehydration) damaging the cell structure even though, the temperature of the immersion osmotic solution is set up in the vicinity of room temperature. The total difference in colour in the treated samples before freezing ($\Delta E*$) presented in Fig. 16(a) shows that the deviations are dependent on the dehydrating agent.

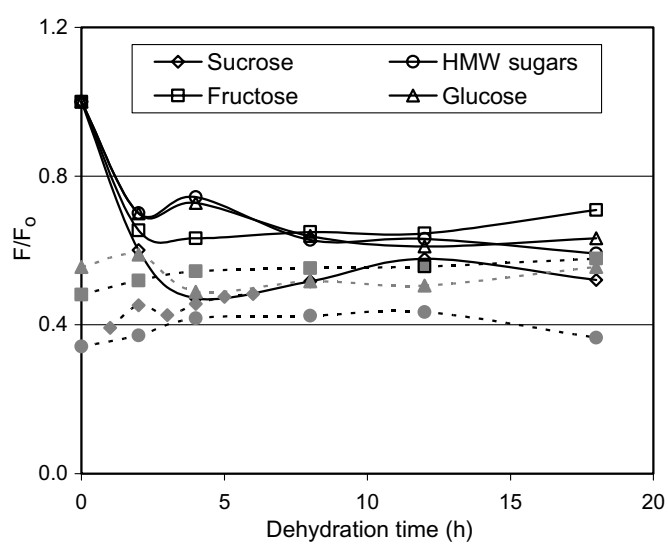

Fig. 15. Normalised puncture force (with the corresponding data to fresh samples) for fresh and dehydrated strawberries, before (void symbols) and after freezing (grey symbols).

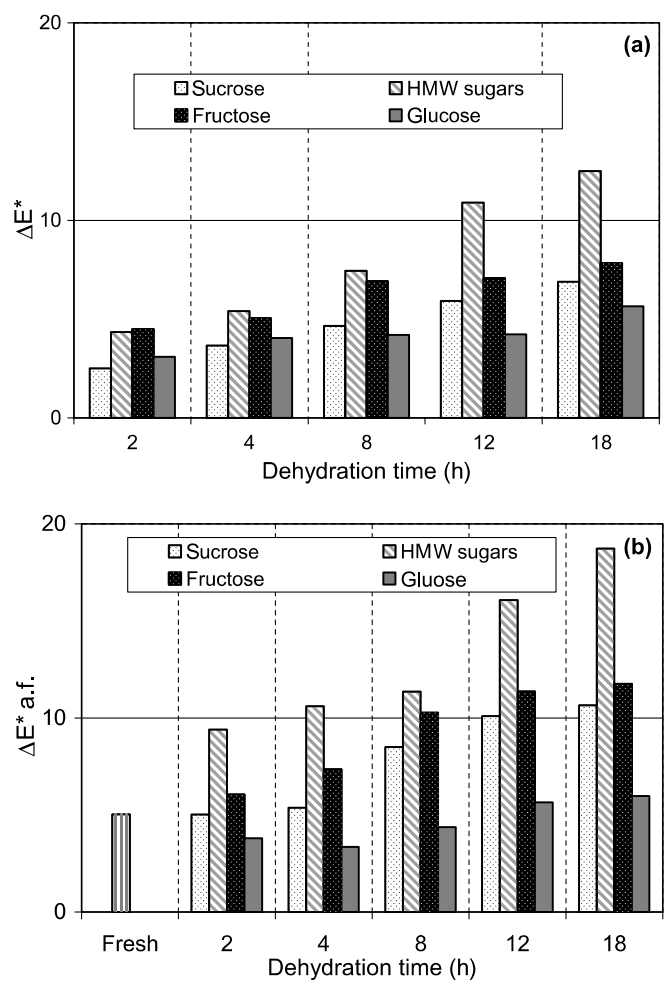

Fig. 16. Total difference of colour ($\Delta E*$) for strawberries (a) treated under different dehydrating agents. (b) treated under different dehydrating agents, frozen and thawed ($\Delta E*$ a.f.).

Glucose and sucrose are the solutions leading to the least colour change.

On the contrary, data of colour tests on the frozen and thawed samples revealed more important differences. For all samples, a more marked decrease in $a*$ and $b*$ is observed, indicating that samples become to a bluer red. Fig. 16(b) reports the values of $\Delta E*$ of samples after being frozen and thawed. Samples dehydrated under glucose present a colour very close to non-frozen fresh strawberries.

4. Conclusions

The improvement caused by the osmotic dehydration prior to freezing on the quality of frozen fruits depends on both the fruit tissue and the dehydrating agent as it was shown in this work through colour, texture and drip loss parameters.

In the case of kiwis, except for drip loss, an osmotic pre-treatment turn samples to a worse quality, at least, as regards the other quality parameters studied in this work.

Apples, on the contrary, exhibit a decrease in drip loss, a better texture and less browning (only, fructose and glucose solutions do not provoke the last two mentioned benefits, respectively). The choice of the dehydrating syrups is decisive to improve quality after freezing and thawing.

The osmotic dehydration prior to freezing on pears has demonstrated to be useful for limiting the drip loss and the colour change of frozen pears. Drip loss decreases with the dehydration time for all the samples, mainly with the sucrose solution. The same phenomenon with sucrose has been found in the former fruits as well, even if the water content with other dehydrating agents has reached similar levels as if sucrose retains water during thawing. In contrast, no improvement was found as regards texture. Thus, the usefulness of the dehydrating pre-treatment depends on the application of the final product.

Dehydrated strawberries, in contrast, show less drip loss after thawing for long dehydration time. Only HMW sugars solution present a lower drip loss at the beginning of the pre-treatment. Texture and colour after freezing are not changed by the osmotic pre-treatment.

As regards mass transfer rates, the structure of the tissue is a very important parameter. The dehydration rates increase in the following order: strawberry, kiwi, pear and apple. The solid penetration follows the same trend.

Concerning the size of the solute molecules, the divergence in dehydration rates for the different tissues is bigger when the molecular weight of solutes is bigger.

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