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Food Research International, Vol. 29, Nos 5–6, pp. 471–479, 1996

Printed in Great Britain
PII: S0963-9969(96)00056-7
0963-9969/96 $15.00+0.00

Osmodehydrofreezing of apples: structural and textural effects

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Osmodehydrofreezing refers to the combined process of partial drying in a concentrated solution (osmotic dehydration) followed by freezing. The physical properties of osmodehydrofrozen apple slices were determined. Granny Smith apples slices were dehydrated to the point of a 50% weight reduction using concentrated sugar solutions (sucrose, sorbitol, or 42 DE corn syrup solids [CSS]) and were frozen at −35°C. Rheological testing (force deformation and small scale oscillatory deformation) indicated that the presence of sugars increased firmness of thawed/rehydrated tissue compared with apples treated in the same manner without sugar, however, untreated frozen controls were firmer than osmodehydrofrozen samples after thawing/rehydration, indicating that conditions used during osmotic dehydration (50°C for 40 min) were too severe. Cryo-scanning electron microscopy revealed a smoothness of the middle lamella of cryoprotected cells and cracking in that of untreated, frozen cells. Conductivity measurements of rehydrating apple tissue indicated that much of the damage done to osmodehydrofrozen fruit membranes was done during pre-freezing treatment.

Keywords: apples, osmotic dehydration, freezing, osmodehydrofreezing, microstructure, rheology.

INTRODUCTION

Freezing of fruits can result in reduced quality, due mainly to inferior texture and enzymatic action. The use of osmotic dehydration as a pre-freezing treatment has been shown in the past to decrease enzymatic browning (Conway *et al.*, 1983) and to reduce structural collapse and drip during thawing (Forni *et al.*, 1990). Osmotic dehydration is the partial concentration of food by immersion in a concentrated solution. It can be followed by further drying or, as in this study, by freezing (LeMaguer, 1988). One of its main benefits is that it allows rapid removal of water from the cell without the disturbance of phase change experienced during air dehydration.

During freezing/thawing of plant cells, it is thought that membranes, particularly the plasma membrane, play a key role in the changes that occur within the tissue; the plasma membrane is considered the primary site of freezing injury (Partmann, 1975; Burke *et al.*, 1976; Steponkus, 1984; Salisbury & Ross, 1985; Webb *et al.*, 1994). Damage to the membrane can be attributed to different events within the tissue, depending on the rate of freezing. Commercial freezing of foods would generally be considered "rapid" freezing. Damage in this case can take different forms, for example, auto-oxidation of lipids (Joslyn, 1966), toxicity due to concentration effects (Heber *et al.*, 1981), or damage to structures within cytoplasm. Membranes of cytoplasmic organelles are vulnerable to loss of membrane functionality due to the formation of irreversible endocytotic vesicles (Dowgert & Steponkus, 1984), lamellar to hexagonal$_{II}$ phase transitions (Wolfe & Bryant, 1992), membrane aggregation due to a reduction in charge density (Morris, 1981), and a reduction in free sterol content of membrane lipids (Uemura & Yoshida, 1986).

In addition to removal of water and a subsequent reduction in ice formation, osmotic dehydration using

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selective solutes can allow cryoprotection of the cell during freezing/thawing. The high concentrations of membrane toxic solutes can be compensated for when protective compounds such as sugars are present to "dilute" potentially toxic solutes (Grout, 1987). Cryoprotectants may also allow the membrane to undergo a rapid change in fatty acid composition in the cell, mimicking the molecular rearrangements of a plasma membrane that is adapted to freeze dehydration stress (Watanabe *et al.*, 1990). Cryoprotective agents, especially high molecular weight compounds, are also thought to lower intramembrane stresses (Wolfe & Bryant, 1992; Skaer, 1987), inhibit protein denaturation in membranes (Burke *et al.*, 1976), and, in the case of sugars, increase hydrophobic interactions in the membrane, increasing stability. Sugars are the solutes of choice for osmotic dehydration of fruits. Sucrose is most common, due to its acceptable flavour, accessibility, and oxygen barrier properties. However, other agents have shown potential. Sorbitol as a dehydration agent for fruits has been shown to have efficient water removal properties (Erba *et al.*, 1994) and to give acceptable flavour (Bolin & Huxsoll, 1983). Corn syrup solids (CSS) were used on apples by Contreras & Smyrl (1981), who proposed that solute uptake was substantially lower than for sucrose treated apples due to high molecular weight compounds. Flavour and texture (evaluated organoleptically) were acceptable.

Much of past work on osmotic dehydration has focused on the kinetics of solute/water exchange in and out of tissue foods with little quantitative evaluation of end product. The purpose of this work was to investigate the quality of apples after osmodehydrofreezing, on a microstructural and rheological level. The physical properties of the apple tissue were investigated after thawing of frozen apple slices that had been osmotically dehydrated using sucrose, sorbitol, or corn syrup solids. Rheological studies included small scale dynamic oscillatory measurements and large scale force deformation testing. Microstructural examination was performed to give insight into the effects of treatments on the cell. Further information on cellular integrity was obtained by measuring conductivity of apples in solution; high conductivity is indicative of leakage of intracellular ions, and therefore, damage to membranes (Willis & Teixeira, 1988; Grout, 1987).

MATERIALS AND METHODS

Processing

Peeled, cored apples (Malus domestica Borkh. cv. Granny Smith, Washington grown) were sliced equatorially to a thickness of 5 mm; 1.5 cm from each end was discarded. Rings were halved and held in a solution of 0.5% ascorbic acid (Stanley Pharmaceuticals, N. Vancouver, BC) and 0.5% citric acid (Fisher Scientific, Fair Lawn, NJ) in tap water for 1 h to allow infusion of the surface of the fruit for the prevention of browning. Apple slices (1.8 kg) were placed between two connected metal sieves in a glass tank, through which solution (30 l) was pumped at 50°C and at 12 l min^{-1}. The target net weight reduction during osmotic dehydration was 50%, reported by Bolin & Huxsoll (1993) as the point at which improvement to texture was observed in pear quarters. Concentration of solutions were modified among trial runs to give this reduction; for a 40 min dehydration at 50°C, the target of 50% weight reduction (±3%) was achieved with 52% sucrose (Redpath Sugars, Toronto, ON), 75% CSS, 42 DE (Casco Inc., Etobicoke, ON) and 50% sorbitol (Debro Chemicals, Mississauga, ON) on a w/w basis. A vacuum of 20 kPa was applied at the beginning of the run for 15 s to increase initial solute/water transfer. A control termed "no treatment" was run by freezing freshly sliced apples. A second control, termed "blanch", was produced by exposing apple slices to tap water at 50°C for 40 min in the absence of sugar. After treatment, the slices were rinsed with cold tap water, patted dry with cheesecloth, and frozen at −35°C in a single layer on metal trays. All samples (three treatments and two controls) were prepared in triplicate. Freezing rates, determined with thermocouples from 5°C below crystallization points on the freezing curves, were found to be between 2°C min^{-1} and 4°C min^{-1}. Apples were stored at −35°C in plastic 1 l containers until use.

Compositional analysis

Compositional analysis was performed on three osmodehydrofrozen samples of each treatment or control, in duplicate, both before and after rehydration.

Moisture levels were determined using a vacuum oven at 70°C for 18 h. Net weight reduction (WR) was calculated as:

$$WR = 1 - \frac{(\%\ \text{total solids fresh})}{(\%\ \text{total solids dehydrated})} \quad (1)$$

Moisture was determined directly after osmotic dehydration and again after thawing and complete rehydration. Rehydration was done at room temperature using tap water at a fruit:water ratio of approximately 4:1 and was maintained for 15 min, which was determined to be the point at which weight gain ceased.

Sugar levels were determined using a Waters 600E HPLC equipped with a Waters 700 Intelligent Sample Processor and a Waters 410 Differential Refractometer Detector (Millipore Corp., Milford, MA). Tissue (10 g) was mixed with 20 ml of HPLC grade methanol (Fisher Scientific, Fair Lawn, NJ); the mixture was held overnight at room temperature, then centrifuged at 1000×*g* and filtered through #2 filter paper (Whatman International Ltd., Maidstone, England). Prior to injection the

liquid was filtered through a 0.45 μm filter (Micron Separations Inc., Westboro, MA).

Colour

Thawed apples (200 g), rehydrated for 15 min in tap water and allowed to drain for 20 min, were blended with 50 ml of tap water ($n=3$). The mash was exposed to air for 20 min and placed in a colorimeter (Agtron Inc., Sparks, NV) where red light was used for the measurement of the L value; a scale of 0 to 90 Agtron units was used, where 0 represents black and 90 represents white.

Conductivity

The conductivity of the rehydration solution of the apples was measured using a portable conductivity meter (Hanna Instruments, Laval, QB) for determination of membrane damage during freezing. Treated and control apples were investigated before and after freezing ($n=3$). Fifteen disks with a diameter of 7 mm (partly thawed in the case of frozen tissue) were weighed and placed into test tubes containing 35 ml of 0.4 M mannitol solution made with deionized, double-distilled water. Tubes were held in an agitated water bath at 25°C and agitated with a vortex mixer prior to readings. Upon completion (3 h), the samples were autoclaved for 30 min at 121°C for complete disruption of cells and a final reading was taken, representing 100% electrolyte leakage.

Dynamic oscillatory measurements

Frozen samples from the three osmotic dehydration treatments and the two related controls were thawed/rehydrated in tap water until fully rehydrated (15 min). Using a 1.8 cm cork borer, disks were removed from the apple slice. An attempt was made to lower the cork borer at a consistent rate, avoid vascular tissue, and take the disk from the centre of the slice. Small scale deformation was performed on twelve replicates for three samples of each treatment using a Carri-Med CSL 100 Controlled Stress Rheometer (Carri-Med, Dorking, Surrey, UK). Apple disks were oscillated between parallel plates, which were covered with fine (150) grade sandpaper for prevention of slippage; the lower plate was raised until the sample touched the upper plate, and 50 μm (1%) of compression was added to ensure adequate contact with the apple. The stainless steel upper plate was 2 cm in diameter. After determination of the linear viscoelastic range (LVR), a constant stress of 2.0 Pa was chosen (common to the LVR of all samples). Frequency sweeps were performed from 25 to 0.02 s^{-1} at 10°C on 12 disks for each treatment. ANOVA was performed on the following parameters: resonance frequency (f_r), maximum % strain (σ_r), storage modulus (G') at 10 Hz, and loss modulus (G'') at 10 Hz.

Force deformation

Force deformation curves were generated by an "M" series texture measuring apparatus (J.J. Lloyd, Omnitronix, Mississauga, ON) equipped with a 50 N load cell (a 500 N load cell was used for fresh apples). A Warner–Brazler type shear apparatus was used to deform the sample lengthwise across the slice. A test speed of 10 mm min^{-1} was employed. Deformation at maximum force (D_m) was determined for ten replicates of three samples for each treatment.

Scanning electron microscopy

All samples were examined in duplicate by cryo-SEM after 1–2 weeks and after 3 months of storage, in the following conditions: fresh-frozen, osmodehydrated (not frozen), osmodehydrated (frozen), and rehydrated. For all frozen samples, cubes (approximately 3 mm×3 mm×8 mm) were cut from the apple slices while immersed in liquid nitrogen to maintain the frozen state. For non-frozen samples, cubes were quench-cooled in nitrogen slush under vacuum (−210°C) within the preparation chamber of the SEM. All cubes were cut and positioned longitudinally so that air pockets would be seen only cross-sectionally. A copper holder was used to contain the samples; it had two 4.5 mm diameter holes drilled into its base in which samples were held with Tissue Tek (Miles Scientific, Naperville, IL). After immersion in nitrogen slush, the holder was delivered under argon to the Emscope SP2000A Cryogenic Preparation System (Emscope Ltd., Ashford, Kent, UK), where a fresh sample surface was fractured using the moveable blade. The specimens were sublimated for 30 min at −80°C and sputter coated with gold. The holder was transferred (under argon) to the cold stage of the Hitachi S570 SEM (Hitachi Ltd., Tokyo, Japan) at −150°C and viewed using 10 kV of accelerating voltage.

Data were analysed using the REGWQ multiple range test from Statistical Analysis Systems (SAS Institute, Cary, NC) at the 5% level.

RESULTS AND DISCUSSION

Fresh apples contained an average of 87.6% moisture (±1.2, $n=10$) and this increased to 89.7% (±0.9, $n=10$) after soaking in the citric/ascorbic acid solution. Osmotic dehydration reduced moisture content in apples to 75%, corresponding to a 50% net weight reduction. Sugar levels obtained by HPLC are shown in

Table 1. Sugar levels of osmodehydrofrozen apples before rehydration, determined by HPLC. Percent sugars and normalized solids ratio (NSR) are given for each sugar

Treatment	% H_2O	Fructose		Glucose/sorb		Sucrose		Total sugar[a]	
		%	NSR	%	NSR	%	NSR	%	NSR
Fresh	87.2	5.6 (0.93)	1.0	3.6 (0.91)	1.0	2.2 (0.57)	1.0	11.4	1.0
Suc (OD)	75.4	8.10 (2.92)	0.76	5.84 (2.23)	0.84	3.82 (1.77)	0.91	17.8	0.81
CSS (OD)	76.0	4.64 (1.37)	0.44	6.09 (1.00)	0.90	1.58 (1.24)	0.39	12.3[b]	0.58
Sorb (OD)	74.9	12.6 (1.17)	1.14	7.63 (2.52)	1.08	3.60 (1.53)	0.85	23.8	1.06

The NSR is a ratio of experimental % sugar to normalized % sugar (corresponding to dehydration with no sugar migration).
Values are the average of three samples, each done in duplicate.
Numbers in brackets are standard deviations.
[a]Sum of fructose, glucose/sorbitol and sucrose.
[b]Levels may be higher than indicated due to presence of maltose, maltotriose, and other higher molecular weight saccharides.

Table 2. Sugar levels/rehydratability of osmodehydrofrozen apples after full rehydration (three samples, $n = 2$)

Treatment	% Fructose	% Glucose	% Sucrose	% Total sugars	% Rehydratability	% Water
Fresh	5.6	3.6	2.2	12.8		
Sucrose	2.95	2.12	1.71	6.78	72.0[a,b]	84.8
CSS	3.54	4.39	0.100	8.02	54.7[a]	84.9
Sorbitol	5.67	3.48	1.57	10.7	84.0[b]	84.8

Values with the same letter are not significantly different ($\alpha = 0.05$).
[a]Rehydratability $= \frac{\%\text{ weight regained (rehydration)}}{\%\text{ weight lost (dehydration)}}$ ($n = 3$ for each of two replicates).

Table 1. As previously demonstrated by Giangiocomo *et al.* (1987), results indicate an outward migration of sugars from the tissue. The results have been expressed on a dry weight basis, as normalized solids ratio (NSR); this is a ratio of experimental % sugar to normalized % sugar (corresponding to dehydration with no sugar migration). In a separate experiment, apples immersed in sugar solutions at room temperature overnight with no agitation had a NSR of approximately twice that of the osmodehydrated apples (Tregunno, 1995). This indicates that the heat and/or circulation applied during processing facilitates water removal with a lower corresponding sugar increase. CSS treated apples show values for total sugars lower than suspected true values; this is because CSS contains not only glucose (15%) but maltose (24%) and maltotriose (14%) (Goff *et al.*, 1990). After rehydration, all sugar levels returned to original (fresh) levels or below, as shown in Table 2.

Conductivity

Conductivity of rehydrating apple disks increased sharply initially, levelling off at 2–3 h. The curves generated, shown in Figure 1, were fit to the following first-order equation:

$$C(t) - C_o = (C_f - C_o)(1 - e^{-kt}) \quad (2)$$

where $C(t)$ is maximum conductivity at a given time t, C_f is the final conductivity level, k is the rate of ion loss, and C_o is the initial conductivity value, or offset. Results are shown in Table 3. Values for C_f indicate that significant damage was inflicted upon cell membranes of blanched and osmotically dehydrated apples before freezing was undergone. This is most likely due to the heat applied during treatments; in addition, the presence of sugars seemed to cause additional damage (although not significant at $\alpha = 0.05$). It is possible that dehydration effects, such as lamellar to H_{II} phase transitions, may have compromised membrane quality. The formation of the H_{II} phase facilitates introduction of openings in the membrane.

Table 3. Initial and maximum conductivity (C_o and C_f) values of rehydrating solution of apple tissue. Frozen samples were partially thawed prior to time 0

Treatment	Before freezing		After freezing	
	C_o	C_f	C_o	C_f
Fresh	0.240[a]	0.317[a]	0.561[a]	0.908[a]
Sorbitol	0.250[a]	0.845[b]	0.484[a]	0.926[a]
Sucrose	0.250[a]	0.845[b]	0.435[a]	0.932[a]
CSS	0.307[a]	0.869[b]	0.511[a]	0.956[a]
Blanch	0.335[a]	0.735[b]	0.771[b]	0.955[a]

Values in the same column with the same letter are not significantly different ($\alpha = 0.05$) $n = 3$.

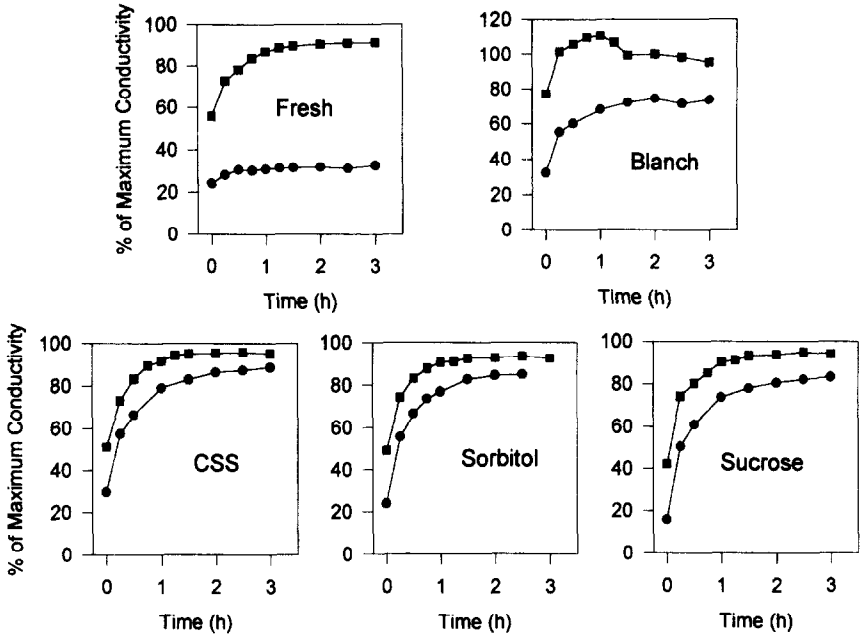

Fig. 1. Percent maximum conductivity over time of rehydrating solution for untreated, blanched and osmodehydrated apple plugs before and after freezing. ●, Unfrozen; ■, frozen ($n = 3$).

After freezing, there was no significant difference between C_f values, however, C_o values for blanched samples were significantly higher than for untreated/frozen and osmodehydrofrozen tissue. This indicates extensive damage to blanched tissue during freezing. In addition, the untreated frozen samples had a higher C_o value than osmotically dehydrated apples, although the difference was not significant. Thus, data imply that although the osmotic dehydration process itself caused damage to tissues, the osmotically dehydrated tissues were protected during freezing.

Colour

Browning was inhibited by all three treatments (Fig. 2). A significant improvement in colour (as measured by the L value) was seen after thawing of osmodehydrofrozen apples when compared with either untreated/frozen or blanched/frozen apples. Treated samples compared closely with fresh apples; in fact, treatment with either sorbitol or CSS gave a higher L value than for fresh samples (not significantly different when $\alpha = 0.05$). It is apparent that tissues frozen with no protection or with a 40 min blanch at 50°C are subject to severe enzymatic browning, which is highly unappealing.

Cryo-scanning electron microscopy

Tissue that was frozen at −35°C with no former treatment maintained intercellular spaces but the middle lamella appeared to have lost its smoothness and had a brittle appearance, indicative of damage during freezing. Chunks of ice and a small crack are visible and cellular material appears to have pulled away from the middle lamella, indicating that the plasma membrane has most likely separated from the cell wall (Fig. 3a). Treated samples prior to freezing showed some loss of "round" appearance and a filling of intercellular space. However, frozen treated samples also showed a smoother middle lamella and less apparent shrinkage of cell contents away from the cell wall, compared with untreated frozen samples (Fig. 3b).

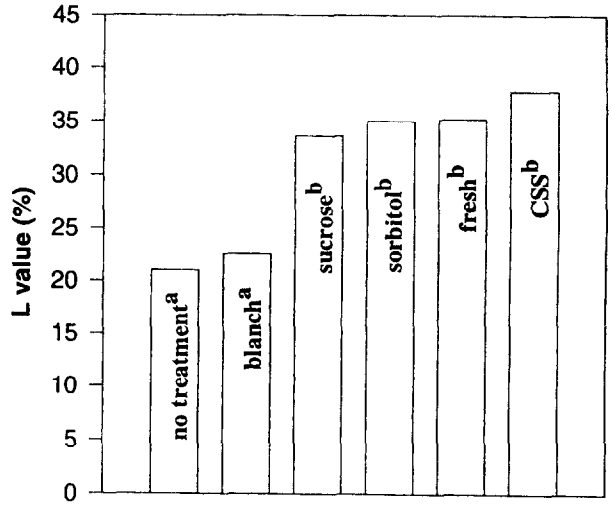

Fig. 2. L values of blended apples (thawed and rehydrated except in the case of "fresh") after exposure to air for 20 min ($n = 3$). Colour is measured using red light on a scale of 0 to 90, where 0 is black and 90 white. Values with the same letter are not significantly different ($\alpha = 0.05$).

Upon comparison of untreated cells and cells treated with sucrose, sorbitol and CSS (Fig. 4), it can be noted that ice crystal formation did not follow the same mechanism in all cases. Untreated, frozen cells show evidence of ice formation outside the cell and inside the cell, indicating that perhaps extracellular ice formation (EIF) was initiated, seeding intracellular ice across the membrane. For tissues treated with sucrose and sorbitol, it appears that IIF occurred, meaning that the rate of ice crystal propagation was more rapid than the rate of water flux from inside to outside the cell. CSS treated samples show evidence of primarily EIF (Fig. 4e), meaning that water was able to exit the cell and condense onto extracellular crystals. Although the location of ice is generally a function of freezing rate, other factors such as nucleation may play a role, since freezing rate was similar for all samples. The differences seen may reflect the location of solute within the tissue; solute acts as a nucleating agent for ice, thus, IIF is kinetically favoured if solute exists inside the cell. In the case of CSS, molecules are large, therefore may remain outside the cell, causing ice to preferentially form there.

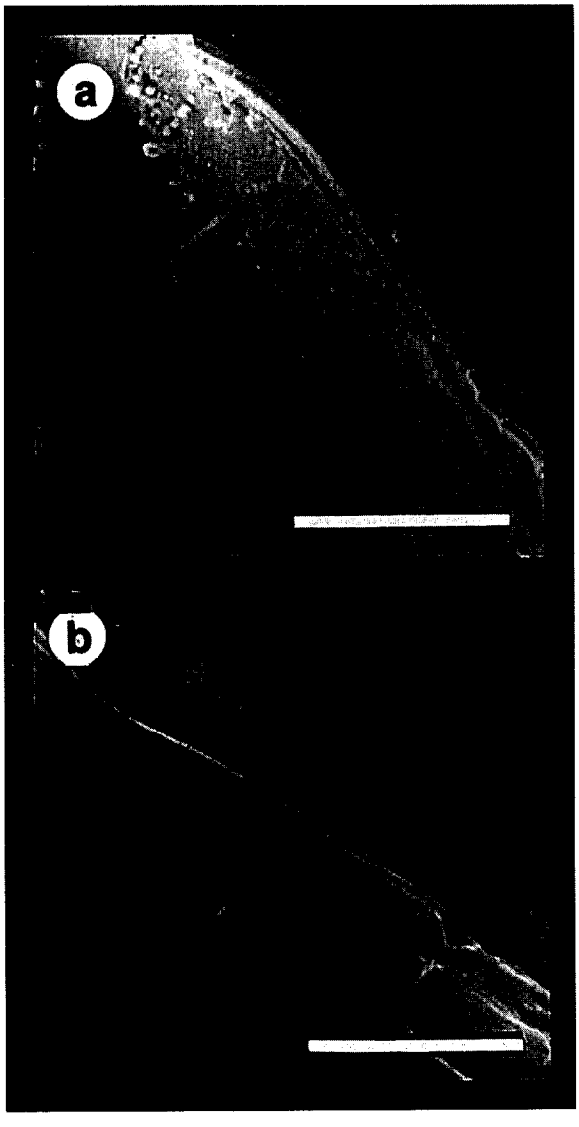

Fig. 3. Cell wall of apple cells from (a) untreated, frozen apple (−35°C) and (b) apple osmodehydrofrozen using sorbitol. Bar, 12 μm.

Rheology

Rheological testing by the various methods used showed similar trends. When subjected to small scale oscillatory strain, all samples gave a constant G' throughout the frequency sweep, while G'' showed a slight linear decrease as frequency was lowered. All samples exhibited a peak in strain, characterized as the resonance frequency (Jackman *et al.*, 1996). Figure 5 compares values for resonance frequency (f_r), resonance strain (σ_r), storage modulus (G') at 10 Hz and loss modulus (G'') at 10 Hz for osmodehydrofrozen and control apples after thawing/rehydration. It can be seen that untreated, frozen apples had a firmer texture (corresponding to a higher G', G'' and f_r and a lower σ_r) than osmodehydrofrozen apples. However, apples which were treated with water in the absence of sugar appeared to be the least firm.

Similar to these trends, the large scale deformation data indicated a significant loss of firmness for apples treated without sugar. Deformation at maximum force (D_m) increased with decreasing firmness. As shown in Figure 6, D_m values indicate that all samples were significantly "harder" than the apples frozen after treatment without sugar. Samples were significantly different from one another excepting sorbitol and sucrose. The order of firmness as measured by D_m was: fresh > no treatment > sorbitol = sucrose > CSS > blanch. These data indicate that the three osmodehydrofrozen samples were slightly less rigid than the untreated, frozen apple, but all were superior in firmness to the control blanched without sugar.

Rheological data indicate that heat treatment was too severe during osmotic dehydration, causing damage to the cell. This was most likely manifested as injury to membranes and alterations of cell wall material. However, it also indicates that cryoprotection occurred in the sugar treated samples; in the absence of sugar, blanched samples were shown to be significantly less rigid than all others.

CONCLUSIONS

The presence of sugars in the apples provided protection during freezing, allowing firmer tissue than for fruit treated in the same manner without sugar. Osmodehydrofrozen apples treated with CSS were less firm than those treated with sucrose or sorbitol (differences were significant for D_m values). Blanched samples were least firm and sucrose/sorbitol were not significantly different from each other. Untreated, frozen samples were firmer

than osmodehydrofrozen apples, indicating that temperatures were too severe during treatments.

SEM revealed that cells protected by sugars exhibited less damage to the middle lamella and less severe shrinking away of cell contents. It appears that ice crystal formation occurred extracellularly for CSS treated samples and intracellularly for other treatments. Conductivity data indicated that prior to freezing,

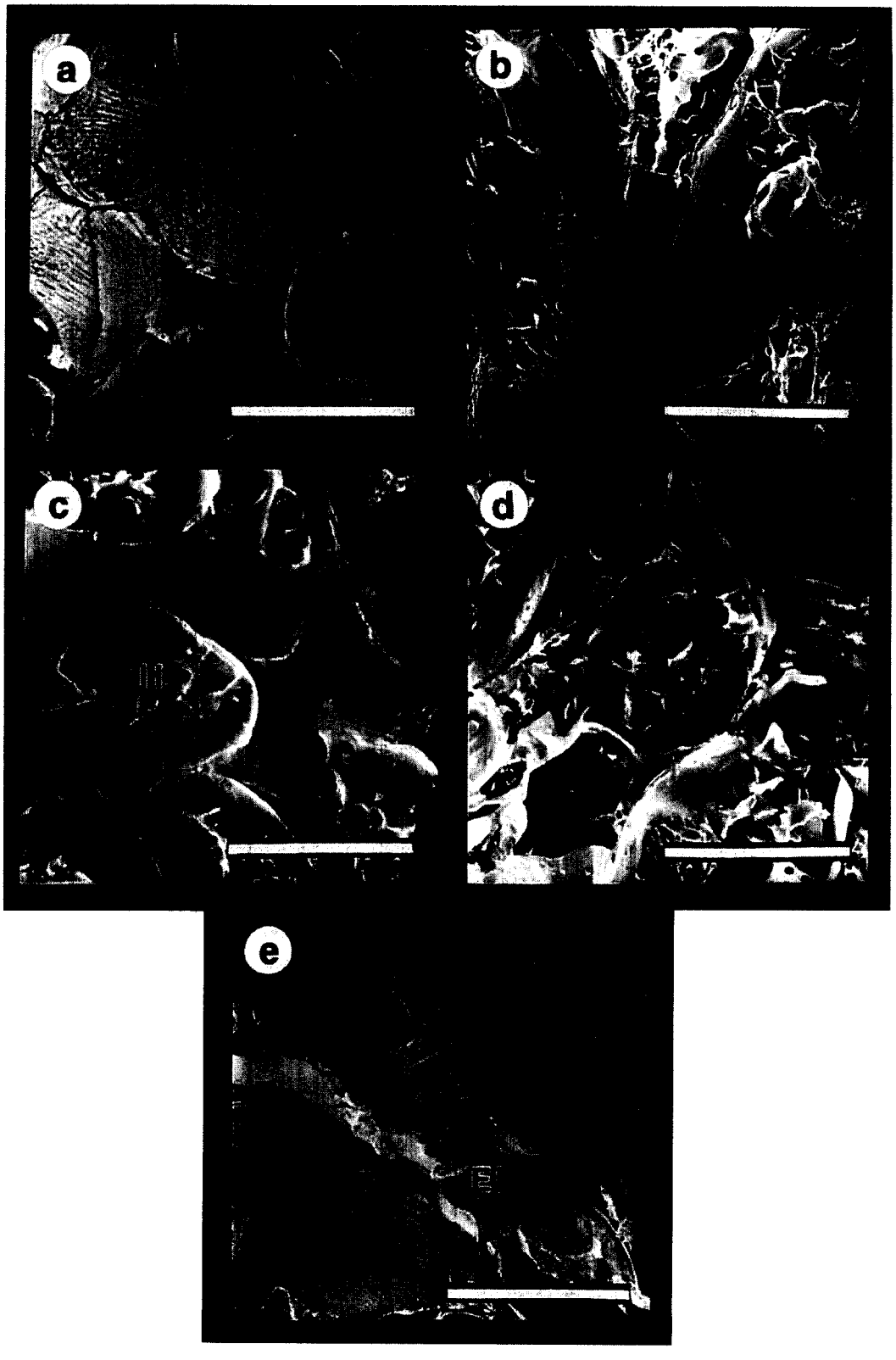

Fig. 4. Cells of (a) fresh apple, (b) untreated/frozen apple, (c) sorbitol treated, (d) sucrose treated, and (e) CSS treated apples. All samples except (a) were frozen at −35°C. CSS treated apples show signs of extracellular ice formation (EI), whereas the other samples show pockets left by intercellular ice (II). Bar, 150 μm.

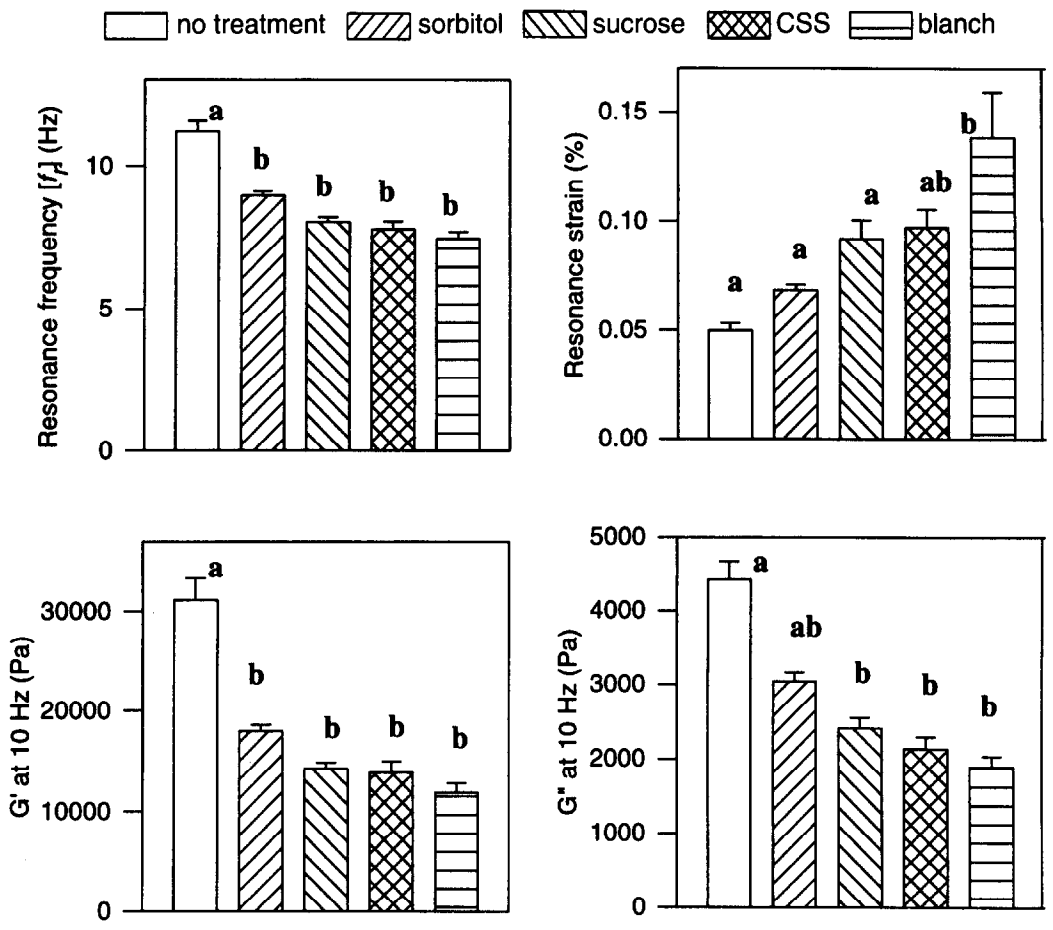

Fig. 5. Comparison of resonance frequency (f_r), resonance strain (σ_r), storage modulus (G') at 10 Hz and loss modulus (G'') at 10 Hz for osmodehydrated apples after thawing and full rehydration during an oscillatory frequency sweep with a stress of 2.0 Pa. Values with the same letter within a histogram are not significantly different ($\alpha = 0.05$); $n = 12$ for each of three replicates.

damage to membranes was severe for osmotically dehydrated samples. All treatments showed higher final conductivity readings than the blanched sample despite better texture upon thawing/rehydration. This may imply that although membrane integrity plays a role in contributing to the textural properties of the tissue, it is not the only consideration; the cell wall may have equal or greater influence on the physical properties of the final product in this situation.

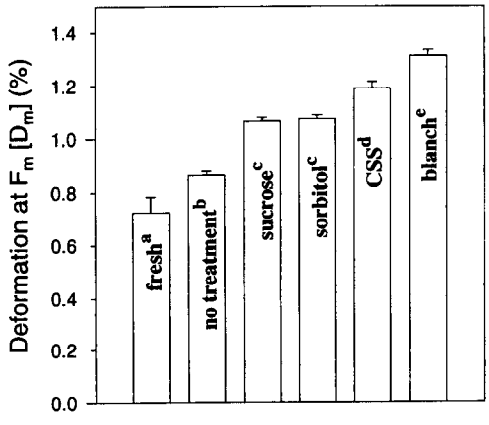

Fig. 6. Deformation at maximum force, D_m, for dehydrofrozen apple slices after thawing and full rehydration, deformed using a test speed of 10 mm min^{-1} with a Warner–Bratzler type shear. Values with the same letter within a histogram are not significantly different ($\alpha = 0.05$).

ACKNOWLEDGEMENTS

The authors wish to acknowledge the financial assistance of the Natural Sciences and Engineering Research Council of Canada and the Ontario Ministry of Agriculture, Food and Rural Affairs.

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(Received 19 April 1996; accepted 29 July 1996)