Immersion Chilling and Freezing: Phase Change and Mass Transfer in Model Food

T. LUCAS and A.-L. RAOULT-WACK

ABSTRACT

The influence of immersion chilling and freezing process variables (temperature, concentration, agitation) on phase change and salt gain, was studied on a gelatin gel dipped into a NaCl solution, with particular emphasis on unsteady state (first hour). Under freezing conditions, water loss was <2 g/100g initial gel and salt gain remained low (<1 g/100g initial gel). Salt penetration was hindered by formation of an ice barrier, which was favored by high concentration and low temperature. Under chilling conditions, salt gain was higher (up to 2 g/100g initial gel), and was enhanced when temperature or concentration increased.

Key Words: chilling, freezing, mass transfer, heat transfer, salt impregnation

INTRODUCTION

IMMERSION CHILLING and freezing consists of directly contacting food with a chilled aqueous solution (<0°C). Binary brine solutions are generally used, most often sodium chloride (eutectic ≅ −20°C) or calcium chloride (eutectic ≅ −50°C) (Robertson et al., 1976; Ogawa, 1988). Ternary solutions (e.g. water + NaCl + ethanol, or sugars) have also been studied (Noyes, 1942; Holston and Pottinger, 1954; Cipoletti et al., 1977).

Immersion chilling and freezing has been studied for whole or pieced foods, e.g.: carrots and peas (Cipoletti et al., 1977; Robertson et al., 1976; Noyes, 1942), potatoes (Stanley, 1978), fish and crabs (Holston and Pottinger, 1954; Hansen, 1981; Anonymous, 1979), fruits (Noyes, 1942), pork and poultry (Brown et al., 1988; Lentz, 1969; Anonymous, 1971), and packaged liquids, such as fruit juices (Cornier and Groussiaut-Monboisset, 1983). Immersion freezing has been used extensively for on-board freezing of fish (Jul, 1986; Möller, 1969), but its industrial use on other products has been limited.

There is a twofold advantage of immersion freezing over air-blast freezing. Overall, energy consumption could be reduced by ≥25% with immersion freezing (Robertson et al., 1976). It is one of the fastest freezing techniques, because heat transfer coefficient is at least 10-fold higher in the liquid phase than in air (Robertson et al., 1976). It takes 15–20 min to freeze peas in an air blast tunnel (Fellows, 1990), vs <2 min by immersion in a ternary solution (Cipoletti et al., 1977). Immersion freezing causes less product dehydration (Mafart, 1991; Robertson et al., 1976), and a higher quality final product is obtained (Coleman et al., 1986).

Cross transfer of water and solute is a unique feature of immersion chilling and freezing, as compared to other freezing processes. Solute transfer leads to two major disadvantages to immersion chilling and freezing: (1) degradation of solution, due to release of solutes and food fragments, microbial growth, oxidation of fats, and foaming (Mafart, 1991; Lenhart and Cosens, 1949; Anonymous, 1979), (2) uncontrolled uptake of solutes by the food, which may be minimized by pre-treatments (e.g. packaging), or post-process treatments (trimming solute impregnated areas). However, packaging hinders heat transfer, and trimming may not always be effective. Solute gain may also be minimized by controlling temperature, agitation, time, solution concentration and composition (Holston and Pottinger, 1954; Robertson et al., 1976; Cipoletti et al., 1977; Poulsen, 1982). Results have not indicated any general trends for effective control. Solute gain can be decreased by formation of an ice barrier (Ottesen, 1915; Noyes, 1942; Holston and Pottinger, 1954; Robertson et al., 1976; Poulsen, 1982). However, how and where such ice barrier may be formed in the product have not been defined. This barrier may be a solid frozen layer integrated within the material or located around the outside surface.

Ottesen (1915) proposed that only an ice barrier around the product could limit solute uptake. The ice barrier would be formed by water migration from the core to the outside of the product. This water could be transformed into an ice barrier (phase change) only when the working temperature was equal, or lower than, the initial freezing point of the solution. Otherwise the water would dissolve, without phase change. Conversely, Noyes (1942) reported that an ice barrier formed regardless of the solution.

The physical state of the surface also determines mass transfer. Robertson et al. (1976) stressed that adherence of solutes on the product surface was the principal source of solute uptake. Methods to reduce it include rinsing, centrifugation, or soaking in dilute solutions (Robertson et al., 1976). In addition, product cracking due to high thermal stress promotes deep solute penetration into the product. Ogawa (1988) advised using a ''thermal equalizing'' stage during low temperature immersion to reduce stress and product deformation. Precoating the food in a sucrose solution has reduced salt uptake (Cipoletti et al., 1977).

The cited examples illustrate the highly empirical current state of immersion freezing. Our objective was to study simultaneous heat and mass transfer during immersion chilling and freezing, with particular emphasis on unsteady state. Experiments were carried out using a model food (gelatin gel cylinders) and binary solutions of NaCl, the most commonly used solute for this purpose (Cipoletti et al., 1977). Variables were temperature, concentration and agitation. Simultaneous time-course changes in mass transfer (with particular emphasis on salt gain) and phase change (cross-sectional observations of freezing changes) were studied.

MATERIALS & METHODS

Solution preparation

Solutions were prepared by completely dissolving commercial grade NaCl in demineralized water at room temperature. Solutions were then stored at 4°C. The NaCl mass fraction and initial freezing temperature of the solution were denoted x_j and T_j, respectively (Table 1).

Solutions (A, B, C, D and E) were at their initial freezing temperature, in the agitated mode. For some solutions, we modified the solution temperature (conditions B' and E') or phase contacting mode (conditions E_s and E_c). All experimental conditions were placed on a simplified water/NaCl solution phase diagram (Fig. 1). The different NaCl crystal forms were eliminated on the right side of the phase diagram. All experimental conditions were on the freezing curve, except for B' (x_B = 0.15, T_A = −2.9°C) and E' (x_E = 0.23, T_A = −2.9°C). The gel freezing point range (−14°C/−15°C) was within the experimental domain. Hence, freezing conditions (C, D, E, E_s, E_c) and chilling conditions (A, B, B', E') could be studied.

Author Lucas is with CEMAGREF, BP 121, 92185 Antony, France. Author Raoult-Wack is with CIRAD-SAR Food Technology & Engineering Division, 73 Rue J.F. Breton, BP 5035, 34032 Montpellier, Cedex, France.

Table 1—Characteristics of solutions used

	NaCl mass fraction x_j	Initial freezing temp (°C) T_j
A	0.05	−2.9
B	0.15	−10.9
C	0.19	−15.3
D	0.21	−17.8
E	0.23	−20.6

Gel preparation

Gels (75% w/w water and 25% w/w gelatin) were prepared by mixing gelatin powder (Sanofi B250) with blue colored demineralized water, heated in a sealed beaker in a 60°C controlled bath, and maintained at that temperature for about 1.5 hr. Foam was skimmed off, and the sol was poured into cylindrical molds (36 mm i.d.). They were left to gel at room temperature for 2 hr. The gel cylinders were then stored in hermetically closed molds for about 17 hr in a ventilated room at 5°C.

Two hours before processing, gel cylinders were removed from the chill room and separated from the mold. A sample was manually cut from one end for moisture determination. The initial gel mass, denoted m(0), was determined. At this stage, the gel was $11 \pm 0.5 \times 10^{-2}$ m long, and weighed 118 ± 3g. The gels were then wrapped in stretchable plastic film (food-grade PVC) and stored back in the chill room. Gel water loss after removing from the mold was previously found to be negligible during storage.

In experiments using solutions A, B and E, one thermocouple (T-type, protected with a kapton sheath, 1 mm o.d.) was placed at the geometrical center of the sol (±1 mm precision) before gelling. The gel freezing point was −14°C/−15°C.

Experimental apparatus and procedures

Two different experimental apparatus and procedures were used, for kinetics over 1 hr, and 12 days, respectively. For water loss and solute gain kinetics over 1 hr, a stainless steel container (255 × 150 × 22 mm) was filled with 6L solution and placed in a Lauda RKS 20 cryostat container. Rapid flow of freezant fluid (glycoshell-ethylene glycol based) around the solution container and the high thermal conductivity of steel resulted in <1°C difference between the solution and the freezant fluid. The solution temperature was measured with a Pt100 probe mounted on the gel carrier (Fig. 2). Gels were placed in a stainless steel screen (4 mm mesh) basket (234 × 134 × 40 mm). A removable cover kept the gels submerged. Gel cylinders were prevented from contacting each other during treatments.

Three agitation modes were studied. In 'agitated' mode, the carrier underwent agitation cycles as follows: the gel carrier was pneumatically raised or lowered with 7 cm amplitude; it was lowered to the bottom within 2 sec, held there 1 sec, then raised within 1 sec and held at the top 3 sec and repeated. In 'stationary' mode, the basket was submerged to and kept at mid height in the solution. The 'combined' mode involved an initial 5 min agitated phase followed by a second stationary phase. For each type of solution (A, B, C, D, E), the agitation mode was specified using subscripts: ''s'' for stationary, ''c'' for combined, and no subscript for agitated.

Immediately prior to treatment, three gels were placed in the carrier and plunged into the solution. The high solution to gel volume ratio (>50) prevented solution temperature from increasing more than 1°C. At 5, 30 and 60 min, a gel was removed from the carrier, rinsed and blotted. The mass of gel, denoted m(t), was determined. Water loss, denoted WL(t) and salt gain, denoted SG(t) were then measured.

For solute gain kinetics over 12 days, hermetically closed plastic boxes (350 × 150 × 170 mm) were filled with 6L solution E (x_E = 0.23), which were placed in a freezer at −20°C ± 3°C, with no agitation. At t = 0, the carrier was submerged to mid-height in the solution. The gels were taken out of solution after 1, 6, and 12 days treatment; rinsed, blotted and weighed [m(t)]. Water loss and salt gain were then measured.

Measurement and analysis procedures, data expression

Experiments involving thermal and mass measurements were done separately to prevent thermocouples from altering mass transfer. Thermocouples were fixed in the gel during gel preparation, connected to a data logger (Campbell CR 10 KD), and temperatures were recorded at 15 sec intervals. Core temperatures of three different gels processed together in the same experiment were averaged. Time-temperature curves obtained at the core of the gel were used to establish thermal equilibrium and the initial freezing temperature of the gels. The gels were considered to be cooled when core temperatures equalled brine bulk temperature. The time from onset of cooling until the core temperature reached −18°C was the freezing time.

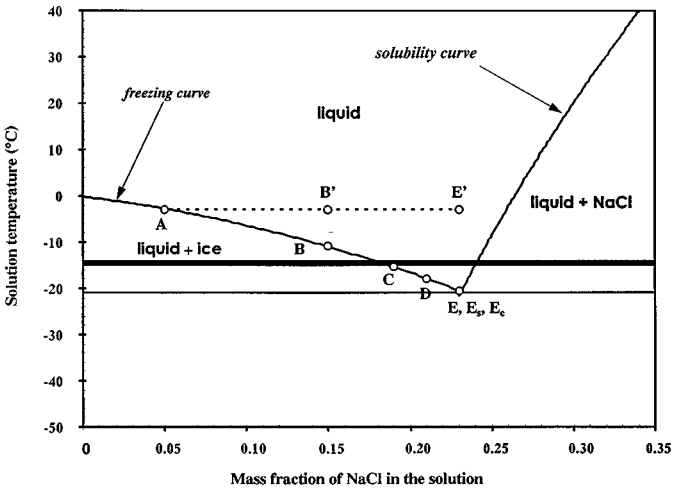

Fig. 1—Phase diagram for the water/NaCl solution (at atmospheric pressure) showing all experimental conditions used in this study: A, B, B', C, D, E, E', E_S, E_C, ▩ gel freezing temperature range: −14°C to −15°C. The experimental values for liquids plotted on the curves were derived from the *ASHRAE Handbook* (1983), *Handbook of Chemistry and Physics* (1975), and Cocks and Brower (1974).

Staining of gels made it easy to visualize freezing because the frozen zone was white and the unfrozen zone was blue. Extent of freezing was observed on cross-sections cut at the center of the gel where edge effects were lowest. An appendix includes two series of photographs showing appearance of these cross-sections, which were drawn as schematic diagrams. Frozen zone thickness was measured within ±1 mm.

Overall water loss and salt gain measurements were determined by blending the whole gel sampled at time t (Moulinex blender) 30 sec to 1 min, depending on thickness of the frozen zone. Salt distribution was determined on the central cylinder section (h = 45 mm) by cutting it out into one central cylinder (12 mm radius) and two concentric cores (16 mm and 18 mm external radius) with a core borer. Each of these sections was blended separately. 10-g samples were used to determine water mass fraction, denoted $\omega_w(t)$ (oven-drying at 104°C for 24 hr). Salt mass fraction, denoted $\omega_{NaCl}(t)$, was determined on free chloride ions. Extraction was carried out with a 2–5g sample dissolved in 50 mL of 0.3N nitric acid, followed by potentiometric titration (Corning 926 chlorometer). Water loss [WL(t)] and solute gain [SG(t)] expressed in g/100g initial gel, were expressed by equations (1) and (2), respectively:

$$WL(t) = 100 \left(\omega_w (0) - \omega_w (t) \left(\frac{m(t)}{m(0)}\right)\right) \quad (1.)$$

$$SG(t) = 100' \left(\omega_{NaCl} (t) \frac{m(t)}{m(0)}\right) \quad (2.)$$

WL and SG results were means of three replications with standard deviations. The relative error, defined as ratio of mean to standard deviation, was <12% for SG.

When comparing three curves, significant differences in SG were assessed by analysis of variance (95% significance) and a Tukey range test. When comparing only two curves, Student test (95% significance) was used.

RESULTS & DISCUSSION

Overall behavior of gel through immersion chilling and freezing

Simultaneous time-course changes were followed (Fig. 3) in the freezing and thawing phenomena (3.a) and salt gain (3.b) of gels immersed over 12 days in nonagitated solution E, at −20°C. In the progress of a freezing front at the first stages (within 2 hr, Fig. 3.a), the gel core was frozen and reached thermal equilibrium after 2 hr immersion in the stationary mode. For longer

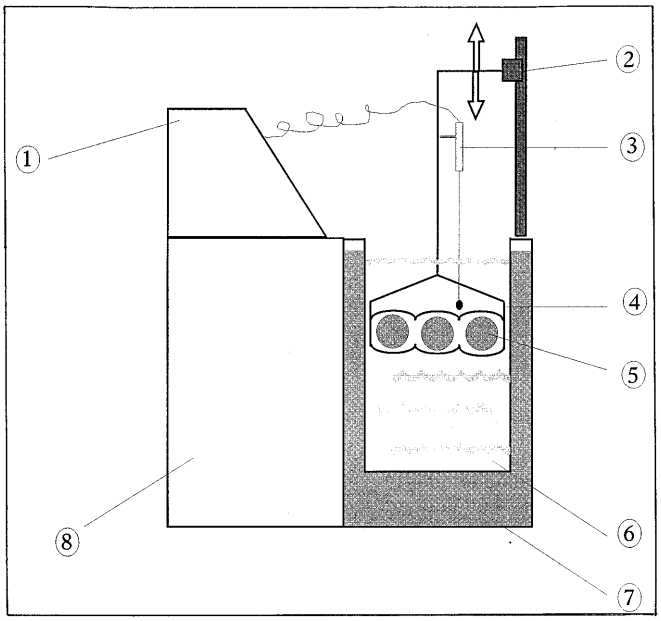

Fig. 2—The experimental system for immersion chilling and freezing. ① thermoregulator; ② pneumatic lifter; ③ Pt100 temperature probe mounted on the immersion basket; ④ stainless steel immersion basket with gel grooves and cover; ⑤ gels for processing; ⑥ internal container filled with concentrated aqueous solution; ⑦ external container filled with freezant fluid; ⑧ cryostat.

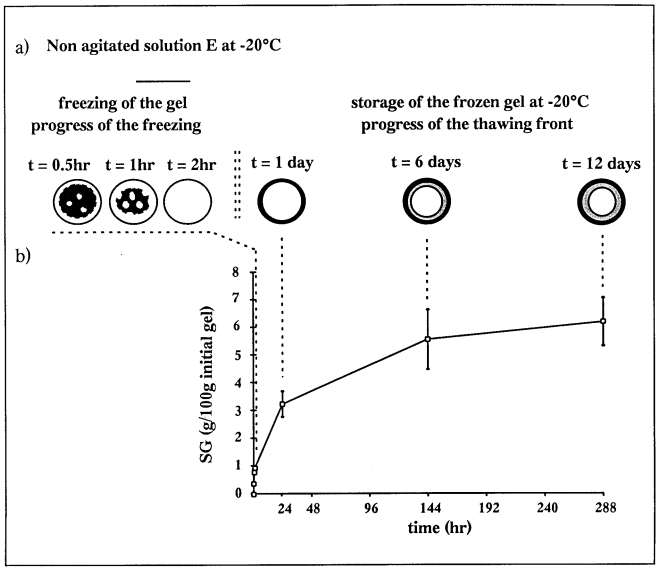

Fig. 3—Immersion of gels in solution E at −20°C in the stationary mode. (a) Cross-sectional comparison of the freezing and thawing fronts over time. (b) Time-course of salt gain (SG).

times, once frozen state and thermal equilibrium were reached at core (for t >2 hr in this case), peripheral thawing was observed as shown by differences in gel staining in the frozen and unfrozen states. At t = 1 day, a completely thawed zone (dark blue) was noted in the gel, the thickness of which remained constant over 12 days storage. A second thawing zone became evident at t = 6 and 12 days. This zone remained white but had a soft texture and was clearly separated by a line from the internal hard frozen zone.

The gel continued gaining salt (Fig. 3.b), even after the frozen state was reached, which we termed the ''secondary'' penetration. During the first stage of the process (freezing not initiated yet), SG levels remained quite low (0.92 g/100g initial gel after 1h treatment for instance). But SG level was much higher at t = 1 day (SG = 3.22%) and plateaued after 6 days (SG = 5.5%). Due to denaturation of the gel network, this experiment was not extended beyond 12 days, although mass equilibrium had not been reached.

Although low SG levels were obtained while chilling or freezing (first stage of the process), salt uptake should be quite important from a sensory point since most of the salt was concentrated on the surface layer. Other results have shown that salt was concentrated in a 2 mm layer on the gel surface (salt mass fraction = 2%) after 1 h immersion in experimental condition E. Also the salt could increase surface thawing by depressing the freezing point, which was in accordance with previous results on fish (Holston and Pottinger, 1954). Core temperature was measured for experimental conditions A (chilling condition), B, E, and E_s (different freezing conditions), i.e. at the boundaries of the studied experimental temperature range. Thermal equilibrium was respectively reached within 28 min, 35 min, 1 hr 26 min, and 1 hr 54 min. Hence, the first 2 hr of processing corresponded to non-steady state period during which thermal equilibrium was reached, well before mass equilibrium, as shown by kinetics carried out over 12 days in freezing conditions.

As a whole, the behavior of the gelatin gel as a model food was satisfactory. Salt content results were close to other reported results (Robertson et al., 1976). Poulsen (1982) observed a 1.45% NaCl mass fraction for 1.5% agarose gel dice (initial side dimension being 2.5 cm) treated for 30 min in a 19.9% sodium chloride solution at freezing point −16.5°C. The mechanical behavior of the gel was also satisfactory (no bursting or swelling) and freezing changes were clearly visible. The gel freezing point (−14°C/−15°C) was relatively low as compared to high moisture content foodstuffs, e.g. initial freezing points for meat are −0.8/−1°C, and for fruits and vegetables −0.5/−3°C (Cheftel et al., 1977). This relatively low freezing point allowed us to carry out detailed studies under both chilling (0 to −15°C) and freezing conditions (−15 to −20.6°C), and to investigate basic mechanisms governing heat and mass transfer that occurred at the first stages, when the gel was immersed into the solution for 1 hr.

Effects of concentration, temperature, and agitation of solution

Water loss. After 1 h of processing (Table 2), WL levels were low, <1 or 2%, except for experimental condition E' wherein 4% WL was reached. The lowest WL was obtained in freezing conditions (E and E_c). Variability was partially due to the low WL levels, but also to uncontrolled ambient condensation on gels during cutting and blending. The low degree of product dehydration during processing is a feature of immersion chilling and freezing. Results were close to those obtained with liquid nitrogen freezing (<2% water loss), and much lower than those obtained with air-blast freezing, where up to 6% weight loss has been reported (Cheftel et al., 1977).

Salt gain and phase change. Simultaneous time-course changes were compared (Fig. 4 a,b) in the freezing and salt gain of gels immersed in solution E at eutectic temperature for various agitation modes (E, E_s, E_c). A freezing front was observed in all cases. In agitated mode (E), the thermal treatment was homogeneous, with a continuous freezing front with axial symmetry and uniform thickness of frozen zone. The front was formed (1 mm thick) after 5 min processing. In the stationary mode (E_s), the frozen front was discontinuous at t = 5 min. Ice began forming in the lower part of the gel and progressed upwards. The thickness of the ice frozen zone varied according to position (due to natural convection) and was denoted when observed in the upper (b_a) or lower (b_b) part of the cylinder. At t = 30 min and t = 60 min, the frozen front was continuous but non-symmetrical (frozen zone thicker in the lower part of the gel cylinder than in the upper part) and the freezing front was

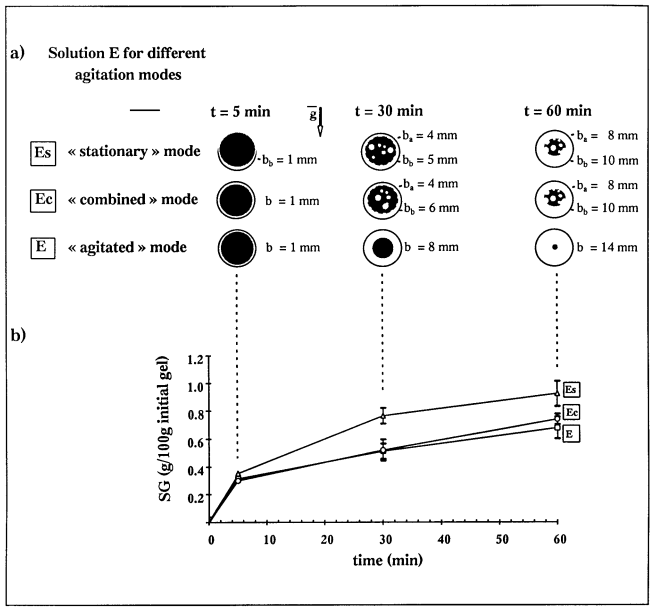

Fig. 4—Immersion of gels in solution E under different agitation modes: E in the agitated mode (□), E_s in the stationary mode (△), and E_c in the combined mode (○). (a) Cross-sectional comparison of the different freezing processes over time: □ frozen state, ▩ unfrozen state, g: gravitational force ($m \cdot sec^{-2}$), b = thickness of the frozen area (mm). (b) Time-course of salt gain (SG).

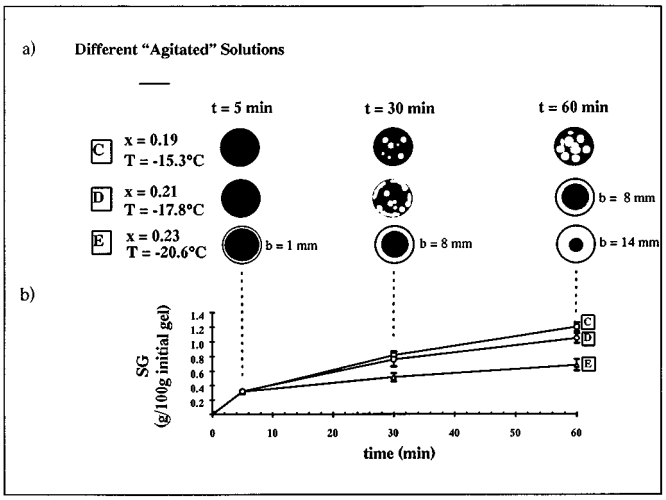

Fig. 5—Immersion of gels in different solutions plotted on the freezing curve: C (□), D (○), and E (△). (a) Cross-sectional comparison of the different freezing processes over time: □ frozen state, ▩ unfrozen state, b = thickness of the frozen area (mm). (b) Time-course of salt gain (SG).

Table 2—Immersion of gels in different solutions. Changes in water loss over time[a]

Experimental conditions	t = 5 min $\overline{WL} \pm \sigma$	t = 30 min $\overline{WL} \pm \sigma$ g/100g of initial gel	t = 60 min $\overline{WL} \pm \sigma$
A	0.40 ± 0.34	−0.56 ± 0.18	−0.71 ± 0.77
B	0.40 ± 0.10	0.81 ± 0.07	1.22 ± 0.17
C	0.39 ± 0.04	1.23 ± 0.04	1.85 ± 0.25
D	0.92 ± 0.72	0.89 ± 0.38	1.19 ± 0.20
E	0.79 ± 0.41	0.40 ± 0.30	0.19 ± 0.22
Ec	0.48 ± 0.16	0.23 ± 0.16	−0.31 ± 0.76
Ei	0.67 ± 0.14	1.34 ± 0.45	1.28 ± 0.27
E'	1.20 ± 0.42	2.50 ± 0.13	3.77 ± 0.20
B'	0.31 ± 0.17	0.96 ± 0.20	1.43 ± 0.27

[a] $\overline{WL}$: mean water loss in g/100g initial gel; σ: standard deviation in g/100g initial gel.

unclear. We noted frozen clusters around the frozen front and near the unfrozen core of the gel cylinder. Thus freezing began by nuclei which gradually grew into frozen clusters. In the combined mode, freezing behavior was the same as observed in the agitated mode at first (continuous 1 mm thick freezing front at t = 5 min). However, for longer processing times, at t = 30 and 60 min (stationary carrier), the freezing profiles did not differ markedly from those obtained in the stationary mode.

There were no significant differences in SG results at t = 5 min, regardless of agitation mode. From t = 30 min, SG levels obtained in the agitated and combined modes were not different, but they were lower than those obtained in the stationary mode (about 30% lower after 1 h processing). Hence, formation of an ice barrier (continuous frozen front) at the first stage of the process probably hindered further salt transfer, and the agitation mode of the solution determined the early formation of the ice barrier. Such an ice barrier differs from that described by Ottesen (1915), Noyes (1942) and Poulsen (1982) (ice barrier around the material surface but outside). This also contrasted with the hypothesis of Ottesen (1915), who stressed that only an external ice barrier on the food product would affect mass transfer.

Simultaneous time-course changes were compared in freezing (Fig. 5.a,b) and salt gain of gels immersed in different solutions in the agitated mode (C, D, E). Freezing with a front was observed in E and D. These two differed in the rate of formation of the continuous frozen front (in <5 min for E, 30 to 60 min for D). Surface freezing was not observed for C. As the solution temperature was close to the gel initial freezing point, freezing occurred slowly, starting around impurities in the gel (ice nucleation).

In all experimental conditions (C, D, E), SG increased over time but remained low (<1.2%) after 1 hr. SG was similar for all experimental conditions (C, D, E) at t = 5 min when no front had completely formed. For t > 30 min, salt uptake was much lower in condition E than in C and D. In condition D (freezing with late continuous front) and C (freezing without front), the SG levels were identical as long as the gel surface was not completely frozen in condition D (for t < 60 min). Once a continuous freezing front had formed, (i.e. at t = 60 min), SG in condition D was lower that in condition C (surface still unfrozen) (1.2g vs 1.05 g/100g initial gel). Thus, even late formation of an ice barrier could hinder further salt gain.

Effects of solution concentration were determined (Fig. 6.a) on the SG kinetics at a given solution temperature under chilling condition in the agitated mode (A, B', E'). SG increased with concentration; after 1 hr (thermal equilibrium reached), SG results for experimental conditions B' (SG = 1.1%) and E' (SG = 1.9%) were respectively 2.5- and 4-fold higher than those for condition A (SG = 0.45%). Effects of solution temperature on salt gain (Fig. 6.b) at given solution concentrations, i.e. x_B = 0.15 and x_E = 0.23, showed that when temperature increased from −10.9°C to −2.9°C (Δ T = 8°C) for x_B = 0.15, SG increased (by 20%) after 1 hr immersion (thermal equilibrium reached). For x_E = 0.23, when temperature increased from −20.6°C to −2.9°C, SG was twofold higher (from 0.7g to 1.9 g/100g initial gel) after 1 hr immersion. However, in that case, gel freezing (which occurred in condition E but not in condition E') was an additional factor which could influence SG variations. Hence in that case, effects of solution temperature and ice barrier could not be separated.

Results from chilling conditions confirmed that either a temperature increase at a given concentration, or a concentration increase at a given temperature, increased salt penetration. This confirmed results obtained with immersion processing at higher temperatures (Raoult-Wack et al., 1991; Lenart and Lewicki, 1990a,b). Choosing specific solutes and controlling cross-flow interactions in ternary or more complex solutions, as well as using variable temperature, may permit effective control (limit or pro-

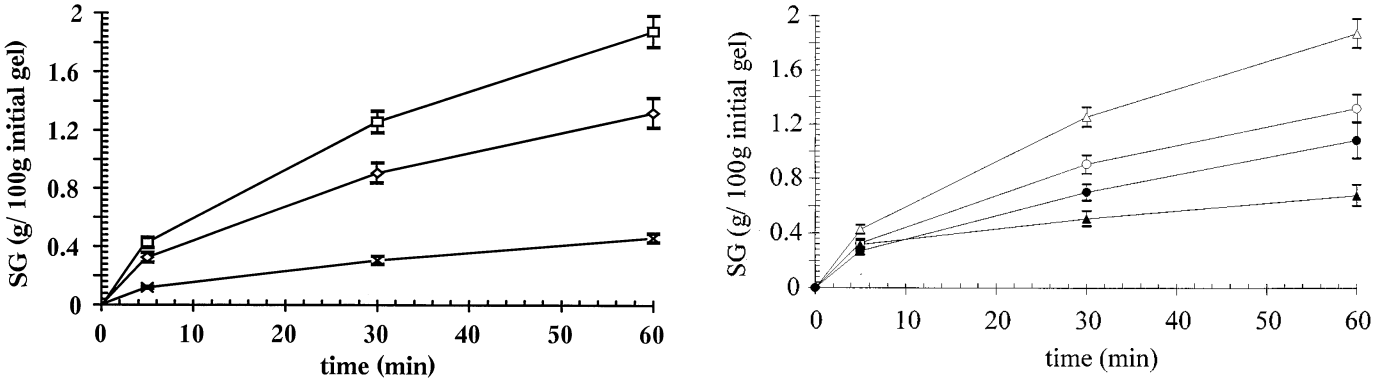

Fig. 6—Time-course of salt gain on gels immersed in different solutions (A, B, B', E, E') (a) at constant temperature (−2.9°C) and for varying concentrations: A (x_A = 0.5) (x); B' (x_B = 0.15) (◇); E' (x_E = 0.23) (□); (b) at constant concentration and for varying temperatures (experimental conditions B (●); B' (○); E (▲); E' (△)).

mote) of solute impregnation (Raoult-Wack, 1994). This indicates the possibility of direct formulation of foods with solutes present in the solution (cryopreservatives, antioxidants, flavorings, etc.) before freezing. It was previously noted by Holston and Pottinger (1954) and Cipoletti et al. (1977) that salt gain was reduced by presence of an additional solute at low temperatures.

CONCLUSION

IMMERSION CHILLING and freezing process variables (e.g. concentration/temperature of solution, agitation mode) could be modified to control salt penetration during unsteady state. Under chilling conditions, SG could be up to fourfold higher when the temperature or concentration of solution increased. Under freezing conditions, SG was mainly controlled by formation of an ice barrier, inside the material. The ice barrier formed sooner as the solution temperature was low and agitation was increased. There was competition between heat transfer (including phase change) and mass transfer at the surface layer of the material. In nonsteady state, salt uptake occurred, but the formation of an ice barrier impeded salt penetration. In transient state, salt uptake persisted even after the gel was frozen and an ice barrier had formed. Further studies on mechanisms of heat (including phase change) and mass transfer requires further analysis of competitive heat and mass transfer kinetics.

LIST OF SYMBOLS

b	thickness of frozen or thawed zone	m
$\vec{g}$	acceleration of gravity	m·sec^{-2}
h	height	m
SG	salt gain (kg/100 kg of initial gel)	
m	mass of gel	kg
WL	water loss (kg/100 kg of initial gel)	
r	radius	m
T	temperature	°C
T_p	temperature of gel core	°C
t	time	sec
x	sodium chloride mass fraction in binary solution (%)	
ω_{NaCl}	sodium chloride mass fraction in gel (%)	
ω_w	water mass fraction in gel (%)	
a	refers to upper part of gel cylinder	
b	refers to lower part of gel cylinder	
c	refers to combined mode of agitation	
s	refers to stationary mode of agitation	
j	refers to solution, characterized by concentration/temperature (A,B,B',C,D,E,E')	

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APPENDIX

Fig. a—Immersion of gels in solution E. Cross-section showing the freezing front after 5 min treatment. Scale in cm.

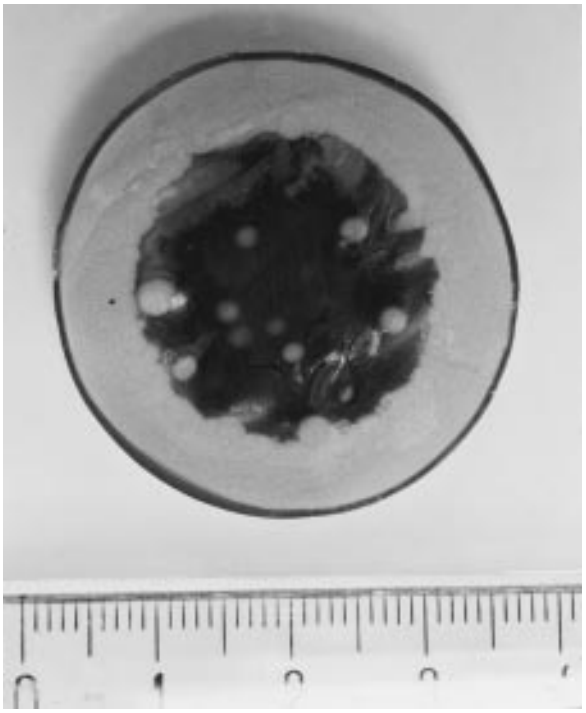

Fig. b—Immersion of gels in solution E. Cross-section showing the freezing front after 30 min treatment. Scale in cm.

Fig. c—Immersion of gels in solution E at −20°C in the stationary mode. Cross-section showing the thawing front after 1 day treatment. Scale in cm.

Fig. d—Immersion of gels in solution E at −20°C in the stationary mode. Cross-section showing the thawing front after 6 days treatment. Scale in cm.