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# PHASE TRANSITIONS OF ADSORBATES: VI, EFFECT OF DEICING AGENTS ON THE FREEZING OF CEMENT PASTE

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## LES TRANSITIONS DE PHASES DES ADSORBATS

### VI. L'effet des Dégivreurs sur la Congélation de la Pâte de Ciment

#### SOMMAIRE

Des échantillons de ciment hydraté (rapport eau/ciment ordinaire de 0.4, 0.6, 0.8 et eau/ciment aéré de 0.5) imprégnés de solutions de NaCl de 0, 5, 9, 13, 18 et 26 % sont soumis à des cycles de refroidissement-réchauffement et on enregistre en même temps les changements de dimensions et de teneur calorifique. De plus des échantillons e/c de 0.6 sont imprégnés de solutions d'urée de 0, 5, 10, 20 et 40 % et mis à l'essai dans des conditions semblables. On mène des expériences afin de préciser l'effet de la vitesse de refroidissement et des dimensions de l'échantillon.

Les résultats s'expliquent par le mécanisme déjà proposé des transitions de phases des adsorbats. En présence de sels, la congélation et la fusion du liquide sortant des pores lors du refroidissement se produit conformément au diagramme de phase en bloc, entraînant des sommets doubles dans les thermogrammes sauf à des concentrations extrêmes. L'effet aggravant des dégivreurs est attribué surtout au degré élevé de saturation par suite de la faible pression de vapeur des solutions. Un avantage est l'élargissement de la gamme de températures auxquelles se produisent les transitions. Les deux effets contraires produisent les pires conditions à une faible concentration (5 % de NaCl) et les meilleures conditions à une concentration moyennement élevée (13 % de NaCl). Les dégivreurs ont un effet de nature physique qui devrait être commun à tous les produits chimiques. L'entraînement d'air, bien que favorable dans la plupart des cas, est parfois défavorable. La meilleure protection contre "l'écaillage dû aux sels" semble être une diminution de la porosité.



# Phase Transitions of Adsorbates: VI, Effect of Deicing Agents on the Freezing of Cement Paste\*

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Changes in the dimensions and heat content of hydrated cement specimens were determined as a function of temperature and concentration of deicing agent in cooling-warming cycles between  $+15^{\circ}$  and  $-70^{\circ}\text{C}$ . The concentration of the polar deicer (NaCl) solution varied from 0 to 26% and that of the nonpolar (urea) solution from 0 to 40%. The  $w/c$  ratios were 0.4, 0.6, and 0.8 plain and 0.5 air-entrained. Experiments were also conducted to clarify the effect of cooling rate and sample size. The observations can be explained by the mechanism previously proposed for phase transitions of adsorbates. In the presence of salts, freezing and melting of liquid exuded from the pores on cooling proceed according to the bulk phase diagram, producing double peaks in the thermograms except at extreme concentrations. The detrimental effect of deicers is attributed mainly to the high degree of saturation, a consequence of the low vapor pressure of the solutions. A beneficial aspect is the widening of the temperature range in which transitions occur. These opposing effects result in the worst conditions at a low deicer concentration (5% NaCl) and optimum conditions at a moderately high concentration (13% NaCl). Since the effect of deicers is physical, it should be common to all chemicals. Air entrainment, although beneficial in most circumstances, can be detrimental. The best protection against "salt scaling" appears to be reduction of porosity.

## I. Introduction

THE "bare pavement" policy of road maintenance has resulted in the use of deicing chemicals in very large and ever increasing quantities. Concurrently, deterioration of concrete pavements, bridge decks, and embedded steel elements has accelerated alarmingly. Disintegration resulting from the action of deicing agents is a serious technical and economic problem<sup>1,2</sup> for which no satisfactory solution has been found. The present work, a continuation of previous studies,<sup>3-5</sup> was conducted to identify the causes of the aggravating effect of deicers on frost action in hydrated cement paste.

## II. Experimental Procedure

The cement paste specimens, which were similar to those investigated previously,<sup>4</sup> were  $\geq 4$  years old. The cylinders were fabricated and the samples cut by the method described in Ref. 4. The air-entrained paste had a spacing factor of 0.48 mm (0.019 in.) and an air content of 5.0%. The water-saturated specimens were immersed in an NaCl or urea solution of known concentration and stored submerged in a de-aired desiccator at room temperature for  $> 70$  days. The diffusion coefficient,  $D$ , of Cl ions in cement paste with a water/cement ratio ( $w/c$ ) of 0.4 was taken as  $2.51 \times 10^{-8} \text{ cm}^2 \text{ s}^{-1}$ .<sup>6</sup> According to the approximation  $X = 4\sqrt{Dt}$  (where  $X$  = distance between the sample surface in contact with the solution and the point in the paste where the concentration of solution is 0 and  $t$  = time), a cement slab 3 cm thick is completely impregnated in 70 days, so that the soaking time for specimens 0.127 and 0.318 cm thick is more than adequate. The experimental apparatus and procedure have been described.<sup>4,5</sup>

## III. Results and Discussion

Changes in the length and heat content of cement specimens 0.127 cm (0.050 in.) thick ( $w/c = 0.4, 0.6$ , and  $0.8$ , fully saturated with NaCl solutions of various concentrations) were observed during a cooling-warming cycle (Figs. 1 and 2).<sup>†</sup> Results obtained under similar conditions with 0.5  $w/c$  air-entrained paste are pre-

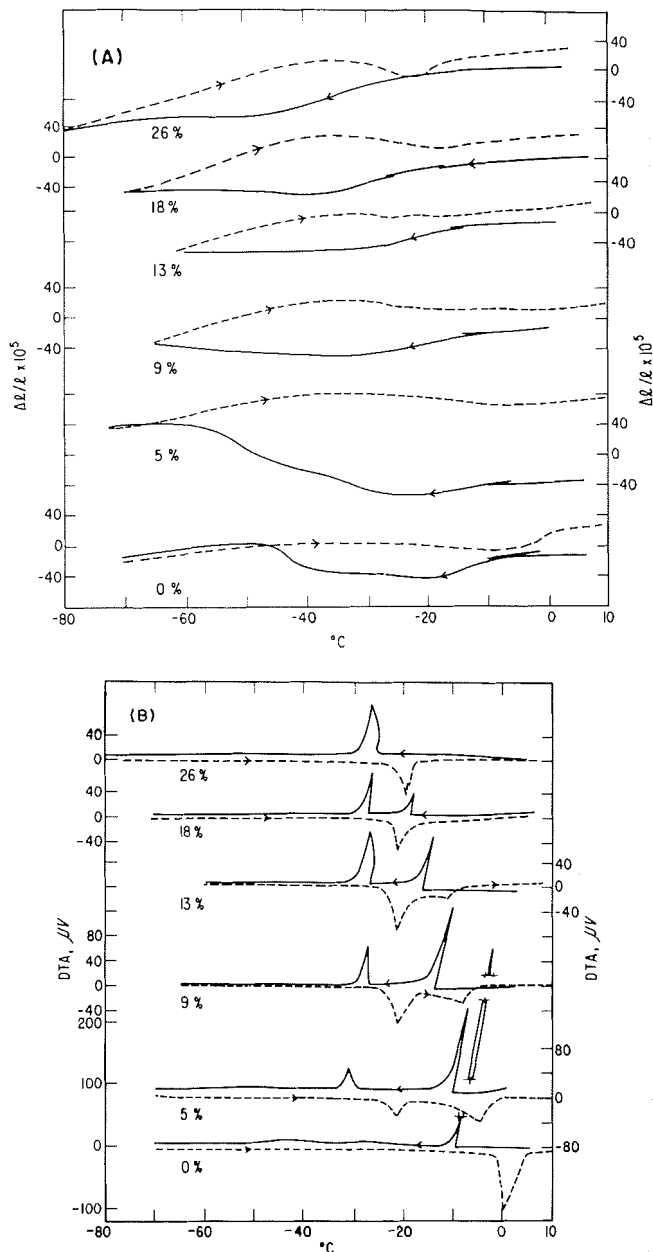


Fig. 1. (A) Fractional length changes and (B) thermograms for 0.4  $w/c$  paste saturated with NaCl solutions of concentrations indicated during temperature cycles ( $0.33^{\circ}\text{C}/\text{min}$ ). Specimens 1.27 mm thick.

sented in Fig. 3. As a result of the constancy of e.g. sample size and shape and instrument geometry, the  $\mu\text{V}$  output of the differential thermocouple serves as a basis for estimation of the observed heat effects.

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† For brevity, the results obtained with the 0.6  $w/c$  samples, although analyzed and discussed in the text, are not presented; they are available from the author.

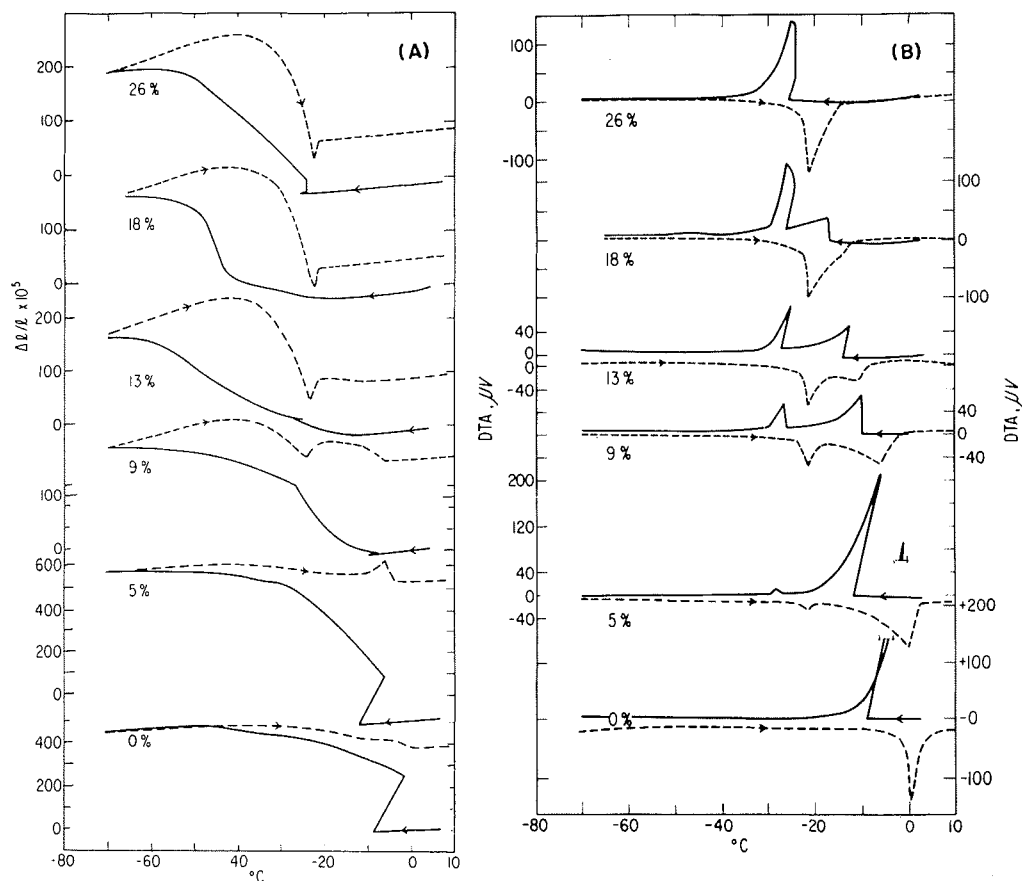


Fig. 2. (A) Fractional length changes and (B) thermograms for 0.8 w/c paste saturated with NaCl solutions of concentrations indicated during temperature cycles ( $0.33^{\circ}\text{C}/\text{min}$ ). Specimens 1.27 mm thick.

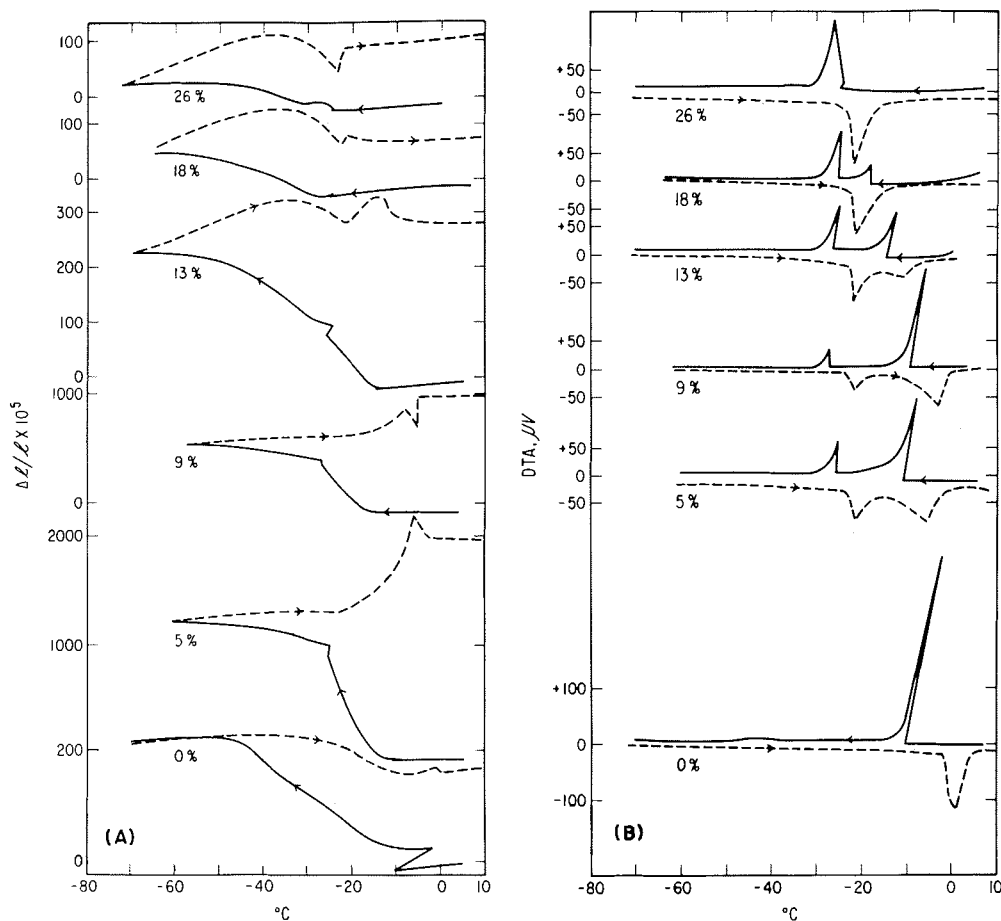


Fig. 3. (A) Fractional length changes and (B) thermograms of 0.5 w/c paste, air-entrained, saturated with NaCl solutions of concentrations indicated during temperature cycles ( $0.33^{\circ}\text{C}/\text{min}$ ). Specimens 1.27 mm thick.

### (1) Heat Effects

During cooling, the thermograms indicate (1) exothermic heat effects in all 4 series 3° to 5°C below the bulk freezing point of the impregnating solution and (2) associated anomalies in length changes. One peak is exhibited in the DTA curves when the adsorbate is pure water or saturated salt solution (0 or 26% NaCl); a doublet appears when the adsorbate is of intermediate concentration. The high-temperature peak of the doublet shifts to a lower value and decreases in magnitude with increasing concentration. The lower-temperature peak always occurs at  $\approx -25^\circ\text{C}$ , and its magnitude is directly proportional to salt concentration. The shapes of the peaks reveal abrupt onset of the process at the higher temperature; at the lower temperature, the heat evolves more gradually.

It has been suggested that frost action originates because the vapor pressure of bulk ice outside a porous body is lower than that of the nonfreezable liquid adsorbed in the pores.<sup>4</sup> When a body is saturated with pure water, this inequality arises just below  $0^\circ\text{C}$ , but with a mixed adsorbate this temperature is depressed because of the lower partial pressure of water in the solution. After instability has been created, equilibrium is restored by desorption, which lowers the adsorbate vapor pressure by the meniscus effect. In this respect, the mechanism is similar to that for systems containing a single-component adsorbate. The exudate on the external surface reduces its vapor pressure by freezing and, in solutions, proceeds according to the phase diagram valid for bulk constituents. The first DTA peak observed on cooling signifies the beginning of the crystallization of ice and the second the solidification of the eutectic mixture.

The temperature of the first peak (the incipient freezing point) of the samples is shown in Fig. 4. The values are understandably lower than the bulk freezing point because of the time lag in migration to the external surface and because of supercooling. The spread of values decreases with increasing concentration, presumably as a result of the greater probability of nucleation at low temperatures. On the other hand, this solid, in the bulk state, is expected to melt at the normal melting point on warming. Since a solution melts over a range of temperatures rather than at a single temperature, the criterion of melting must be chosen with care. The temperature of the endothermic peak is not suitable for this purpose because it is displaced to higher temperature in a warming system. An apparently reliable indicator of the completion of the fusion process is the resumption of linear expansion of the system. Figure 5 illustrates the deviation from the bulk melting point of the temperature at which normal behavior resumes for 30 samples. Since the variance in 26 cases is  $\pm 1^\circ\text{C}$ , the results are judged consistent with the proposed mechanism. Only the samples at 18% concentration and one at 26% seem to deviate significantly. On further cooling to lower temperatures, no heat effects are exhibited despite the anomalous changes in length. This behavior is in agreement with the finding that solidification in this region is continuous<sup>4,5</sup> and the conclusion that the product is semicrystalline.<sup>7,8</sup>

The measured dynamic mechanical response (elastic modulus and internal friction) of water-saturated porous bodies has also indicated a sharp transition just below  $0^\circ\text{C}$ , with another, broader transition at  $-90^\circ\text{C}$ .<sup>9,10</sup> The high-temperature peak in the curve was attributed to "capillary transition," but previous results<sup>3</sup> suggest that it results from freezing of capillary water which has migrated to the external surface.

Glass transition<sup>9</sup> and proton movement in the semicrystalline adsorbate<sup>10</sup> have been suggested as causes of the low-temperature peak. Wittmann<sup>11</sup> found that the behavior of cement paste on cooling was anomalous to  $-90^\circ\text{C}$ , where normal behavior resumed.

These observations are not inconsistent with the concept of continuous formation of semicrystalline ice after initial freezing. The only discrepancy is with regard to the value of the transition temperature, but this disagreement is probably a result of the measuring techniques applied in the present experiments. Gradual transitions cannot be detected by DTA, particularly those of the anomalous first- or second-order type and, similarly, after even partial solidification has occurred in the pores, the dimensions of the system are little affected by adsorption processes. It is therefore believed that the contradiction is only apparent.

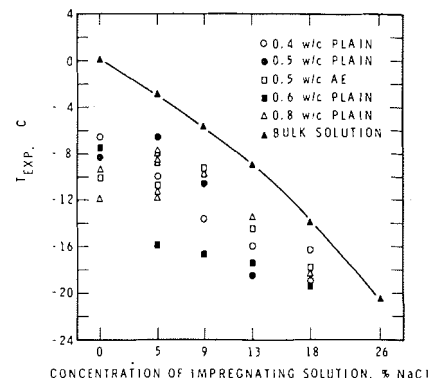


Fig. 4. Incipient freezing point (determined from heat effect) as a function of concentration.

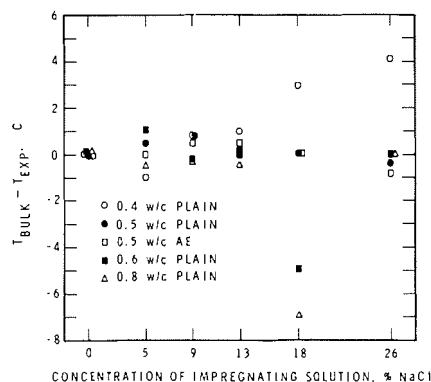


Fig. 5. Deviation of experimental melting point from bulk value.  $T_{\text{EXP}}$  is indicated by feature in expansion curve.

According to DTA, 2 processes occur on warming, i.e. one endothermic peak, whose magnitude increases with increasing salt concentration, appears at  $\approx -21^\circ\text{C}$ , and another, which increases in magnitude as concentration decreases, appears at higher temperatures approaching  $0^\circ\text{C}$ . These peaks are caused by melting of the eutectic mixture and of pure ice, respectively.

### (2) Dimensional Changes

On cooling, all samples first contract linearly at an average rate of  $\Delta l/l = 8.0 \times 10^{-6} / ^\circ\text{C}$ , a value very similar to the coefficient of thermal expansion for water-saturated normal portland cement,  $10.2 \times 10^{-6} / ^\circ\text{C}$ , given by Powers and Brownard.<sup>12</sup> In this phase of the cycle, length changes are evidently caused by thermal effects.

At and below the temperature of the first exothermic peak, the 0.4 w/c sample contracts because the quantity of the pore-held liquid decreases. For the porous-silica-glass-brine system, which is more amenable to quantitative analysis, the amount of liquid desorbed could be estimated; on this basis, the expected contradiction agreed well with that observed.<sup>3</sup>

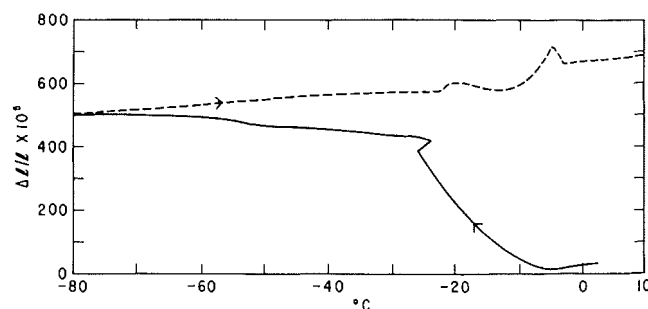


Fig. 6. Fractional length changes of 0.4 w/c paste saturated with and immersed in 5% NaCl solution (specimen 3.18 mm thick, heating rate  $0.33^\circ\text{C}/\text{min}$ ).

When, during cooling, the unstable liquid in the pores is prevented from reaching the external surface in its migration, the sample expands. Such a condition can be created by a high degree of saturation, low permeability, and/or blockage of the passageways. Figure 6 demonstrates the effect of blockage by ice. When a cement sample of 0.4 w/c was saturated with and immersed in 5% NaCl and cooled to  $<0^{\circ}\text{C}$ , a fractional expansion of  $400 \times 10^{-5}$  occurred, instead of contraction (Fig. 1(A)). The increased residual expansion,  $700 \times 10^{-5}$  instead of  $120 \times 10^{-5}$ , clearly indicates the added severity of the damage inflicted.<sup>13</sup>

When they were cooled below the eutectic temperature ( $-21.1^{\circ}\text{C}$ ), all the specimens expanded because, at this stage, all are encased in ice and migration is hindered. However, the resulting dilation is rather small and persistent, even during rewarming from  $-60^{\circ}$  to  $-40^{\circ}\text{C}$ , as if completion of the expansion were delayed until after the incipient temperature had been reestablished.

This interpretation is supported by the fact that, on very slow cooling, salt-impregnated porous glass expands greatly and does not dilate on rewarming.<sup>3</sup> It is therefore assumed that the expansion during warming at low temperatures is part of the expansion associated with cooling. Accordingly, to assess expansion, the difference between minimum length on cooling and maximum length on heating is plotted in Fig. 7 as a function of salt concentration; overall expansion generally increases with increasing w/c and is greatest at 5% concentration. The 0.5 w/c air-entrained sample is an understandable exception because it has the largest porosity and liquid content by virtue of the incorporated air. (A long period of immersion in liquid water would fill some, if not all, of the air-entrained bubbles.) On the other hand, the rather small expansions of the 0.8 w/c series cannot be readily explained; until further investigations prove otherwise, this behavior should probably be attributed to incomplete saturation.

Notwithstanding the exceptions, the overall expansion values are consistent with those expected, supporting the assumption that expansion on cooling cannot proceed to completion, presumably because the viscosity of the adsorbate is very high at the temperatures in question. The solidified liquid apparently lends rigidity to the system, and expansion is stored until after the "reinforcing" links melt on rewarming. The substantial increase of the elastic modulus at low temperatures<sup>9,10</sup> is in harmony with this view. On warming, specimens impregnated with highly concentrated salt solutions contract distinctly at  $-21^{\circ}\text{C}$ , the melting point of the eutectic mixture. With more dilute solutions, length anomalies are shifted to higher temperature and are especially pronounced when the w/c ratio is large.

### (3) Effect of Variables

(A) *Urea*: To gain information on the effect of the chemical composition and polarity of the adsorbate, samples saturated with urea solutions were investigated. The expansion and DTA curves

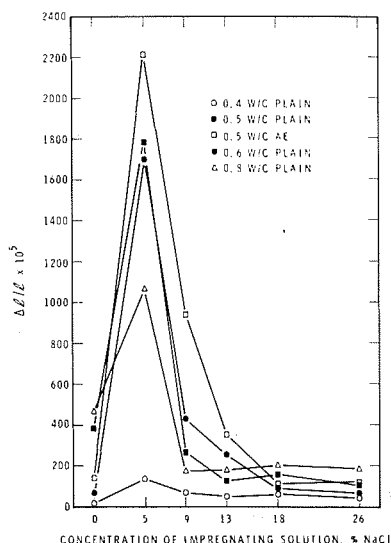


Fig. 7. Overall expansion (difference between minimum on cooling and maximum on warming) as a function of concentration.

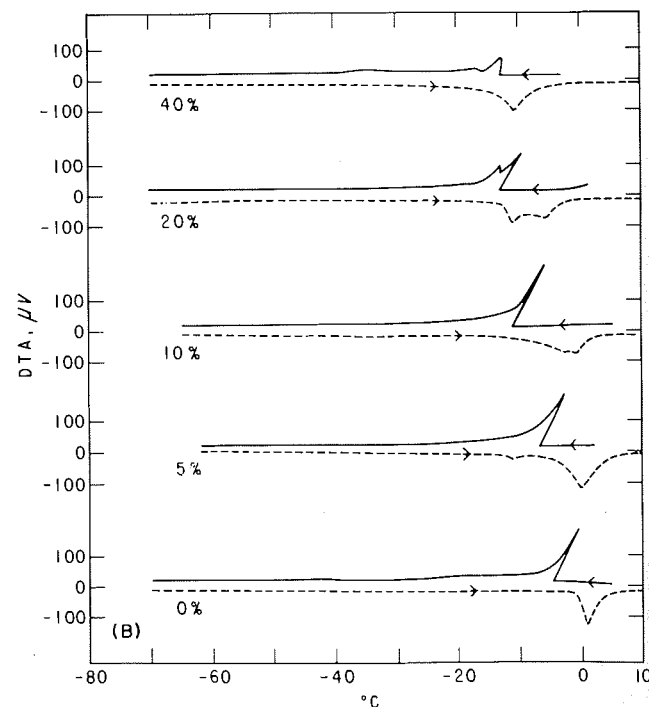
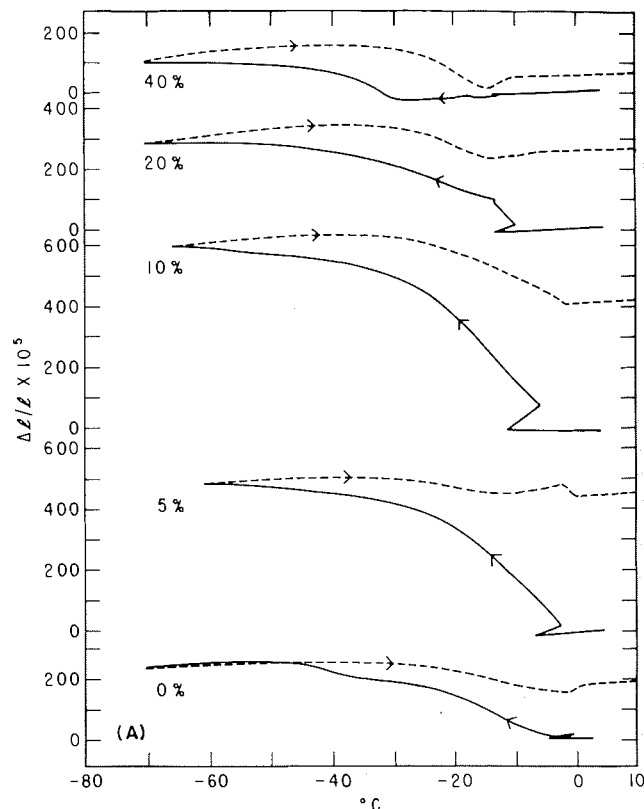


Fig. 8. (A) Fractional length changes and (B) thermogram of 0.6 w/c paste saturated with urea solutions of concentrations indicated during temperature cycles ( $0.33^{\circ}\text{C}/\text{min}$ ). Specimens 3.18 mm thick.

(Fig. 8) strongly resemble the corresponding diagrams for NaCl adsorbate and can readily be interpreted in terms of the conclusions of Sections III(1) and III(2). Verbeck and Klieger,<sup>14</sup> who studied deicers other than chlorides, including urea, found that their action was similar to that of NaCl and  $\text{CaCl}_2$ . The present results confirm their conclusions.

(B) *Cooling and Warming Rate*: A rate of temperature change different from the standard  $0.33^{\circ}\text{C}/\text{min}$  was used in a few instances.

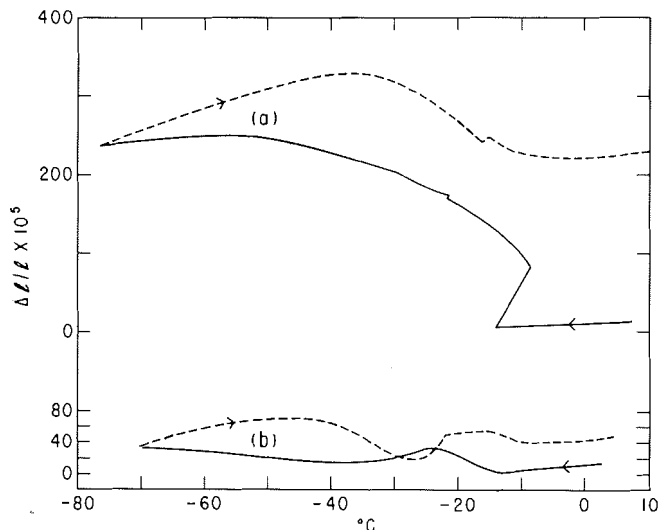


Fig. 9. Length changes of 0.6 w/c paste saturated with 13% NaCl solution during (a) rapid (1.25°C/min) and (b) slow (0.0417°C/min) temperature cycles.

When a 0.6 w/c paste saturated with 5% NaCl solution was cooled at 0.0417°C/min, the length changes diminished dramatically compared with those resulting at the more rapid (by a factor of 8) rate. The residual length change was  $650 \times 10^{-5} \Delta l/l$  compared to  $2000 \times 10^{-5} \Delta l/l$ , and no dilation at low temperatures was observed on warming. This behavior is consistent with the assumption that expansion is stored when cooling is rapid. The curves of Fig. 9 prove that the rate effect is not restricted to the extremely large expansions of samples containing dilute solutions and that a higher-than-standard cooling rate increases the magnitude of dilation.

(C) *Size*: As in the case of pure water as the adsorbate,<sup>4,5</sup> extensions along the longitudinal axis of the specimen are magnified when its thickness is increased. The length change curves for 0.8 w/c paste samples 2.0 and 3.2 mm thick saturated with 18% NaCl solution (Fig. 10) illustrate this point.

(D) *The w/c Ratio*: As the value of w/c increases, the features of the expansion curves become more pronounced (Figs. 1-3) because less cement and more adsorbate are present in a unit volume. The adverse effect of these parameters can be appreciated.

(E) *Air Entrainment*: Incorporation of air bubbles in a cement mix is a well-proved method of minimizing and even avoiding freezing damage. In the presence of deicing agents, however, air entrainment does not always give complete protection.<sup>14</sup> Large voids are beneficial because they serve as a refuge for liquid that, on cooling, has become unstable in the pores. The "excess" liquid can be accommodated, however, only when the air voids are empty. Large pores become full only after prolonged exposure to 100% rh and empty on the slightest decrease below this value. When the pores contain soluble salts, filling is much more probable because of the low vapor pressure of the resulting solution. For example, with a 26% NaCl solution all the voids will be full at 77% rh, a very common climatic condition. After it is completely saturated, air-entrained cement suffers greater freeze-thaw damage than comparable plain cement because its porosity is substantially larger. This behavior explains the results obtained with the 0.5 w/c air-entrained samples (Figs. 3(A) and 7).

(F) *Concentration*: As stated, the largest expansion occurs at the relatively low concentration of 5% (Figs. 1-3, 8), in agreement with the findings of Verbeck and Klieger<sup>14</sup> and Arnfelt.<sup>15</sup> Similar behavior of the NaCl-porous-glass system has been attributed to an increased degree of saturation and broadening of the temperature range of freezing.<sup>3</sup> These effects oppose one another and result in the most severe conditions at low (5%) concentrations and the least severe at moderately high (13%) concentrations. This argument im-

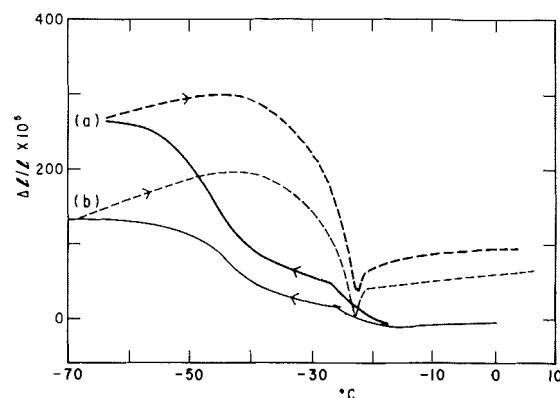


Fig. 10. Length changes of 0.8 w/c paste saturated with 18% NaCl solution for specimens (a) 3.2 mm and (b) 2.0 mm thick.

plies that dimensional changes with extreme concentrations (0 and 26%) of NaCl should be very similar in a given system. Although this expectation is qualitatively true, it is not realized quantitatively, except in the 0.4 w/c series; the samples showed significantly greater expansion when water was the saturating medium. A probable explanation for the reduced length change in the 26% NaCl-cement system is that, at the low temperature at which anomalies occur, length stabilization resulting from the cementing action of the solidified adsorbate causes a significant increase in elastic modulus. Additional experiments will be necessary to verify this hypothesis.

#### IV. Conclusions

The action of deicing agents can be rationalized on the basis of the theory previously proposed for "freezing" of adsorbates. The phenomenon in cement paste is similar to that found in porous silica glass. These conclusions lend further support to the theory because they imply that essentially the same mechanism is valid for water and nonaqueous adsorbates, with and without deicers, in cement paste and porous silica glass.

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