



Wetting and drying of concrete using aqueous solutions containing deicing salts

Robert P. Spragg^{a,*}, Javier Castro^{a,1}, Wenting Li^{a,b,1}, Mohammad Pour-Ghaz^{a,1}, Pao-Tsung Huang^{a,2}, Jason Weiss^{a,3}

^aSchool of Civil Engineering, Purdue University, West Lafayette, IN 47907, USA

^bSchool of Civil Engineering, Southeast University, Nanjing 211189, China

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ABSTRACT

A series of wetting and drying tests were performed on concrete using different aqueous solutions containing deicing salts. The rate of fluid absorption was generally lower for aqueous solutions containing deicing salts than it was for water. In addition, less fluid was absorbed for samples exposed to aqueous solutions containing deicing salts than for samples exposed to water. The change in the rate of aqueous fluid absorption was proportional to the square root of the ratio of surface tension and viscosity of the absorbed fluid. Concrete that has been exposed to solutions containing deicing salts showed less mass loss during drying. Measures of equilibrium relative humidity over the salt solutions are used to interpret drying behavior. Experimental data indicates that concretes that had previously been exposed to deicing solutions can also exhibit reduced rate of absorption, even if water is the fluid being absorbed.

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1. Research need and significance

Some jointed plain portland cement concrete pavements in freezing prone climates have shown premature deterioration at the longitudinal and transverse joints. While some have attributed this damage to a chemical attack, inadequate air entrainment, poor mixture design, inadequate constituent materials, or poor construction practices, it is the hypothesis of the authors of this paper that this joint deterioration may be attributed, at least in part, to preferential absorption of fluid at joints. This hypothesis was developed based on observations from the field that show these deteriorated locations frequently occurred at low spots in the pavement, where water has accumulated on the pavement, where joint sealers were damaged, or where the joint does not appear to have opened thereby trapping water [1]. Preferential fluid ingress at joints could increase a variety of damage mechanisms including deleterious chemical reactions, crystallization pressure, or freeze

thaw damage that may degrade the concrete. To fully evaluate fluid ingress at the joints it is essential that the wetting and drying behavior of concrete is evaluated using aqueous solutions containing deicing salts.

This work is limited in scope as it considers only the ingress of aqueous solutions over short time periods and does not explicitly consider any chemical reaction that occurs between the aqueous solution and the concrete. This information is intended to provide reference for those developing tests to evaluate potential deicer-concrete interactions [2], for developing tests on fluid absorption, for evaluating fluid absorption in concrete [3], for input parameters in computer simulation of fluid ingress at joints [4], and for potential approaches to limit joint deterioration like penetrating sealers for possible use in concrete pavements [5].

2. Research objectives

The objective of the present paper is to evaluate: (1) the effect of initial equilibrium moisture content and sample conditioning on the rate of fluid absorption in concrete, (2) the wetting and drying behavior of concrete in the presence of deicing solutions, (3) the effect of change in fluid properties due to presence of deicing salts on the fluid absorption and drying rate of concrete, and (4) the effect of residual salts on the re-absorption of water. The

* Corresponding author. Tel.: +1 765 494 7999.

E-mail addresses: rspragg@purdue.edu (R.P. Spragg), jcastro@purdue.edu (J. Castro), li448@purdue.edu (W. Li), pourghaz@purdue.edu (M. Pour-Ghaz), huang69@purdue.edu (P.-T. Huang), wjweiss@purdue.edu (J. Weiss).

¹ Tel.: +1 765 494 7999.

² Tel.: +1 765 494 6242.

³ Tel.: +1 765 494 2215.

findings of this paper have direct implications toward predicting the long-term performance of concrete pavements and provide insight to the underlying mechanism of premature joint deterioration.

3. Fluid absorption in porous materials

Fluid absorption is a frequently used test to provide an indication of the durability of hardened concrete, as it is simple to perform. Several standard tests exist for measuring water absorption including ASTM C 1585-04 [6], BS 1881-99 [7], and ASTM D6489-99 [8]. While the concept behind these tests is very similar, there are differences in how the samples are conditioned, treated, and tested. In each of these tests water is typically used as the fluid that is being absorbed. Hall [9] discusses that water can interact with the cement matrix adding complexity to the interpretation of results. To overcome some of these limitations or to indicate how absorption can be reduced by fluid composition other solutions have been tested [9–14].

MacInnis and Nathawad [15] assessed the absorption of an aqueous solution consisting of a NaCl deicing salt and reported a decrease in absorption. Sutter et al. [16] reported that sorptivity decreased from highest to lowest in the order of water, NaCl, CaCl₂ and MgCl₂. Similar data has recently been observed by Janusz [17]. As a result, it can be observed that concrete exposed to deicing salt solutions absorb fluid at a slower rate than they would absorb water; however the previous work has not related this behavior to the fluid properties or described the influence of salt concentration or properties of the aqueous solution.

The results of one-dimensional fluid absorption tests (assuming negligible gravitational effects) are typically reported as the cumulative water absorbed per surface area (surface from which water is absorbed) versus the square root of wetting time. Eq. (1) is frequently used to describe the water absorption (total volume of fluid absorbed) and the sorptivity (related to the rate of absorption) [18].

$$i = S\tau^{1/2} \quad (1)$$

where i (mm³/mm²) is the cumulative water absorption, S (mm/s^{1/2}) is the sorptivity, and τ (s) is the elapsed time. It should be noted that additional equations have been proposed to account for time dependent properties [19].

Hall et al. [9] proposed that the diffusion would scale proportionately with the ratio of surface tension (γ) and viscosity (η) of the fluid. Hall further related this to sorptivity since sorptivity is related to the square root of diffusion. Kelham [20] derived an expression for fluid absorption (Eq. (2)) that shows the relationship between depth of penetration and the square root of the ratio of surface tension and viscosity.

$$x(\tau) = \sqrt{\frac{4k\gamma \cos(\theta)\tau}{p\eta r}} \quad (2)$$

where $x(\tau)$ (mm) is the penetration depth, γ (N/mm) is the surface tension, θ (rad) is the liquid–solid contact angle, p (Dimensionless) is the porosity of the medium, r (mm) is the pore radius, k (mm²) is the intrinsic permeability of the material, and η (Pa·s) is the viscosity of fluid. An expression similar to Eq. (2) was derived by Scherer and Wheeler [21] for stone consolidates.

Previous research using organic fluids has shown an absorption rate that scales proportionally with the square root of the ratio of surface tension and viscosity of the fluid $((\gamma/\eta))^{1/2}$. This work will use this approach to attempt to interpret results from absorption tests that used aqueous solutions containing deicing salts.

4. Properties of deicing salt solutions

Physical properties of pure solutions were gathered from literature and compared with measured values for the industrially available deicing solutions tested in this research, and they are provided here for convenience in one location. The properties of the deicing solutions will be used in interpreting the wetting and drying results, discussed later in this paper. This section is divided into four sections. The first three sections describe the influence of the deicing solutions in terms of surface tension, viscosity, and equilibrium relative humidity over the aqueous solution. Section 4 describes the specific gravity of the solution as a function of concentration as this is used to determine the volume of solution absorbed during the absorption test.

4.1. Surface tension of deicing salt solutions

Fig. 1a shows surface tension measurements at different concentrations for the three solutions used in this research: NaCl, CaCl₂, MgCl₂. The surface tension for NaCl was obtained from [10], CaCl₂ from [22] and MgCl₂ from [23]. A ring tensiometer was used with a resolution of 0.1 mN/m for the industrial deicers tested in this study. The tensiometer was cleaned between measurements following ASTM D971-04 [25]. The tensiometer was first calibrated using de-ionized water, which provided a value of 71.0×10^{-6} N/mm. A series of three measurements were performed for each solution, with the average reported.

The closed points in Fig. 1a are the values measured for the industrially available solutions. The lines represent values taken from literature for pure salt solutions at different mass concentrations. While the general trends are consistent, differences between the industrial deicing salts and literature values may be due to impurities or other additives. However, further work is needed to examine this in greater detail.

4.2. Viscosity of deicing salt solutions

Fig. 1b shows a comparison of the viscosities for the solutions used in this research between pure solutions taken from literature and measurements of the deicing solutions. Viscosity measurements for the industrial deicers were performed using a rheometer. The rheometer kept the solution being tested at 23.0 ± 0.02 °C and from the torque applied to the fluid that causes a shear from which the viscosity can be found. Calibration of the device was performed using a reference standard.

The dashed lines presented are viscosities at different concentrations and are taken from literature [10,22–24], while the points represent measured viscosities of the industrially available solutions. Again, differences between literature values and those of the solutions measured can be explained by differences in possible additions or chemistries of the industrial deicers.

4.3. Relative humidity of deicing salt solutions

Relative humidity measurements were performed on the salt solutions using a combination relative humidity and temperature sensor ($\pm 0.8\%$ RH at 23 ± 0.1 °C). The relative humidity probes were mounted in a 75 mm × 68 mm stainless steel cylinder that was placed over a water jacketed sample cup holder. The water jacket was connected with a water bath at a constant temperature of 23.0 ± 0.1 °C.

Fig. 1c shows the relative humidity measured over salt solutions for a wide range of solution concentrations. As the concentration increased the relative humidity over the solution decreased. The measured relative humidities of these unsaturated salt solutions

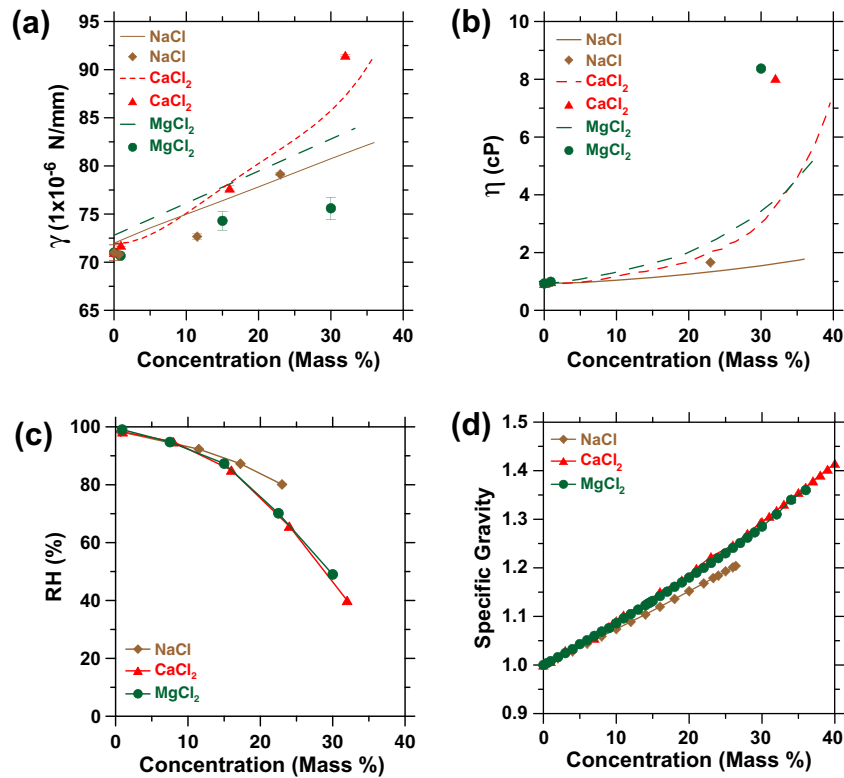


Fig. 1. Properties of deicing salts at 23–25 °C: (a) surface tension, (b) viscosity, (c) relative humidity, and (d) specific gravity (Rosenburgh, 2010, unpublished data).

are higher than that of the saturated solution of these salts which are 75.4% RH for NaCl [26], 33.0% RH for MgCl₂ [26] and 22% for CaCl₂ [22].

4.4. Specific gravity of deicing salt solutions

Fig. 1d shows the specific gravity of different deicing solutions as a function of concentration. The specific gravity of the solution increases with concentration. The CaCl₂ and MgCl₂ increase at very similar rates with an increase in concentration, while the NaCl increases slightly less than the CaCl₂ and MgCl₂ (i.e., 25% less increase with concentration). This may be attributed to the colligative properties of solutions.

5. Wetting and drying for concrete with deicing solutions

5.1. Experimental program of wetting and drying of concrete with deicing solutions

The concrete mixture that was used for these tests was a typical concrete mixture. The mixture proportions of this concrete are shown in Table 1. The fresh air content was 5.7%, which was measured according to ASTM C231-09 [27]. The hardened air content of

the concrete was 4.4% as assessed using an automated optical scanning approach [28] based on the method proposed by Peterson et al. [29].

The concrete was produced in a central mix plant and discharged from a ready mix concrete truck. A series of 100 mm × 200 mm cylinders were cast. After 1 day of curing, the cylinders were demolded and sealed in double plastic bags at 23 ± 0.5 °C until the samples reached an age of 28 days. After 28 days of curing the cylinders were removed from bags and three 50 mm ± 2 mm thick samples were cut from the central portion of each cylinder using a wet saw.

Two different sets of samples were used in this study. The first set of samples was used to evaluate the effect of sample conditioning on water absorption. In each condition, three samples were used. A total of five conditions were considered: ASTM C1585-04, oven-dry, 50% RH, 65% RH and 80% RH. To ensure that these samples that were conditioned at 50%, 65%, and 80% RH, reached equilibrium, a 12-month conditioning period was considered. The second set of samples that were used for aqueous salt solution absorption, drying, and de-ionized water re-absorption were conditioned at 50 ± 2% RH, 23 ± 0.5 °C for 36 months and two samples were tested for each salt solution.

To prepare the specimens for fluid absorption testing, the sides of the samples were sealed with epoxy. After the epoxy had hardened, the top surface was covered with plastic to avoid evaporation from the sample during testing.

The absorption test involves recording incremental mass change measurements during the first 6 h after the sample comes in contact with the fluid and subsequently taking one measurement every day for the next 8 days. The amount of absorbed fluid is normalized by the cross-section area of the specimen exposed to the fluid using:

$$i = \frac{m_t}{(a \cdot \rho)} \quad (3)$$

Table 1

Mixture proportions of concrete assuming saturated surface dry (SSD) conditions.

Material	Proportion
Cement (kg/m ³)	316
Class C fly ash (kg/m ³)	60
Water (kg/m ³)	150
Fine aggregate (kg/m ³)	736
Coarse aggregate (kg/m ³)	1049
Air entraining admix. (ml/100 kg of cem. materials)	20
High range water reducer admix. (ml/100 kg of cem. materials)	456
Retarder admixture (ml/100 kg of cem. materials)	98

where i (mm^3/mm^2) is the normalized absorbed fluid, m_t (g) is the change in specimen mass at time t , a (mm^2) is the area of the specimen exposed to the fluid (i.e., that of the bottom face), and ρ (g/mm^3) is the density of the absorbed fluid (this is provided in greater detail in Section 4.4). These absorption measurements are then plotted as a function of the square root of time, as provided by Eq. (1). The sorptivity is the slope of this graph.

The second series of samples were tested using seven different fluids. Their composition was primarily based on one of three different industrially available deicing products, either NaCl, MgCl_2 or CaCl_2 . A low concentration was used for each salt solution as well as a higher concentration that was selected to be near the eutectic composition for each salt. De-ionized water was also used as a reference fluid.

5.2. Experimental results from wetting with water for different conditioning methods

Fig. 2 shows the results from water absorption tests performed on the first series of samples that were conditioned with different environmental conditions as mentioned earlier (ASTM C1585-04 accelerated conditioning, 80% RH, 65% RH, 50% RH and oven drying). It should be remembered that these samples were conditioned for 12 months while the remainder of the samples discussed in this paper were conditioned at 50% RH for a much longer time. Sample preparation has an enormous impact on the water absorption results as more severe drying enables a greater volume of water to be absorbed during the test.

5.3. Experimental results from wetting and drying with deicing solutions

Fig. 3 illustrates the results of the fluid absorption test as a function of time (for concrete at 50% RH for a longer conditioning time than the samples in Fig. 2). It can be seen that even though the concrete that is used for all the tests in Fig. 3 has the same conditioning and exposure conditions, the volume of solution absorbed by each material is dependent on the deicing salt solution and the concentration of the deicing salt solution that was absorbed. The sample with the low concentration of NaCl showed a slight increase in the rate of absorption (as compared with water) as well as the amount of fluid absorbed. This is consistent with the data reported by MacInnis and Nathawad [15]. The absorption of all the other fluids was reduced when compared with water. As a result, it can be concluded that in general as the salt concentration increased the rate of absorption reduced and the total absorption was reduced. Further work is needed to examine lower concentra-

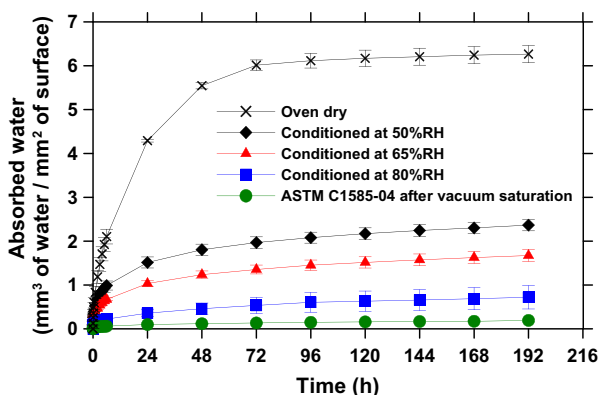


Fig. 2. Water absorption on samples subjected to different conditioning procedures.

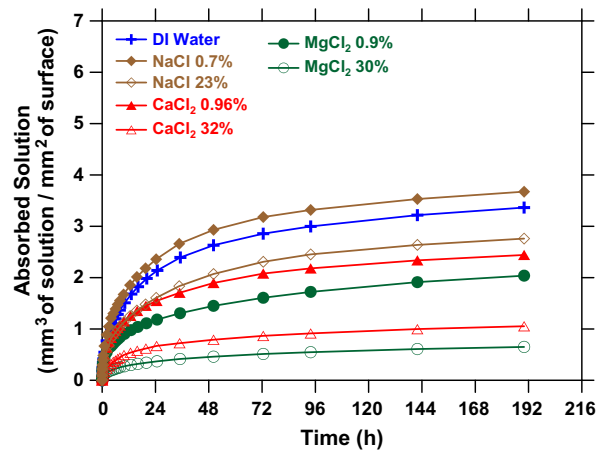


Fig. 3. Volume of deicing solutions absorbed by concrete as a function of time (typical standard deviation less than $0.1 \text{ mm}^3/\text{mm}^2$).

tions for NaCl to ascertain why a slight increase is typically reported.

After the fluid absorption test was performed for 8 days the samples were dried at $50 \pm 2\%$ RH, $23 \pm 0.5^\circ\text{C}$ for 7 days. The samples were kept in the same one-faced exposed condition for the drying test; however, the exposed surface that was facing down in the absorption testing was placed facing up to simulate drying from the top. During the drying test the mass of the samples was recorded at regular intervals.

Fig. 4 shows the volume of water loss during the drying period. It is important to note that the drying test will result in only the water portion of the solution being evaporated from the system leaving the salt to become more concentrated in the solution before it eventually precipitates out. It can be noticed that as the concentration of deicing solution was increased the mass loss during drying decreased. This was particularly evident in the high concentration solutions, which showed nearly no mass loss or even a slight gain during drying.

5.4. Experimental results from wetting previously exposed to deicing solutions

The same set of samples that were previously tested for absorption and drying were oven dried, to be tested for second absorption test using de-ionized water. This was done to investigate the

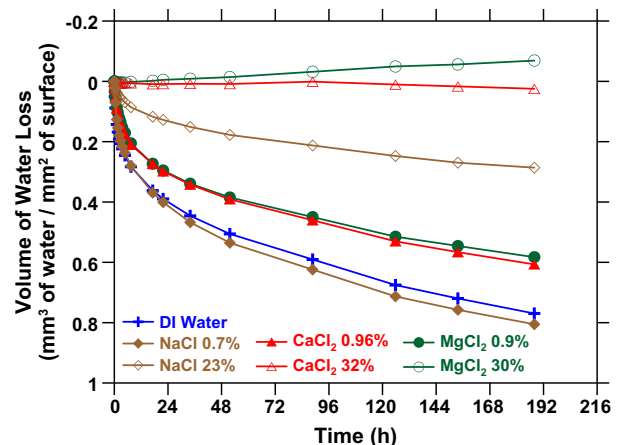


Fig. 4. Drying of concrete prewetted with different salt solutions as a function of time (typical standard deviation less than $0.03 \text{ mm}^3 \text{ water}/\text{mm}^2$).

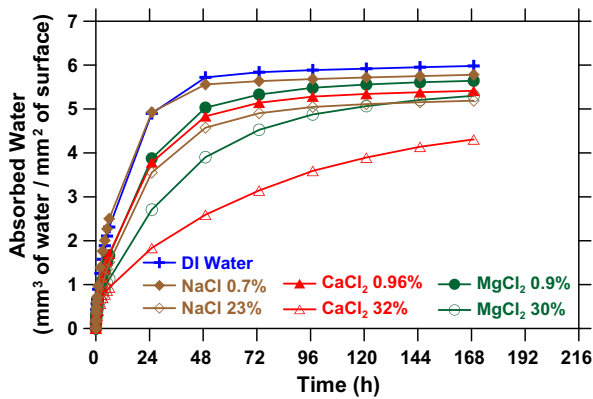


Fig. 5. Volume of de-ionized water absorbed by concrete as a function of time in the second fluid absorption test (fluid from the original test is shown in the caption).

effects of previously absorbed salts on re-absorption of de-ionized water. However, in the case of the oven-dried samples, higher water absorption is expected. This can be due to the empty capillary pores and possibly to microcracking generated during oven drying [30–34].

To prepare the samples for the de-ionized water re-absorption test, the samples were placed in an oven at $105 \pm 2^\circ\text{C}$ until they reached equilibrium. The equilibrium condition was defined as a difference less than 0.5% between two successive mass measurements (24 h apart). It is important to note that this drying process will evaporate only the water portion of the solution pre-absorbed, leaving salt in the pores. Since these samples were oven dried, their absorption rates and absorbed water are not comparable with any previous tests.

Fig. 5 shows the results for this second absorption test. It can be seen by comparing the results to the results in Fig. 3 that the behavior of the samples was dependent on the deicing solutions and the concentrations of deicing solutions used in the first wetting test. These results are a clear indication that the history of the samples affects the results of fluid absorption. This suggests when sorption testing is performed on field concretes, some understanding of the admixtures or salts that remain in the pore system is needed to fully interpret the results.

5.5. Drying of mortars saturated with different deicing salts

Moisture desorption is an established technique for evaluating the effect of moisture loss at a given humidity for a material. A

moisture sorption analyzer was used to carefully control temperature and humidity. Mortar samples were prepared ($w/c = 0.42$ and 55% aggregate by volume) and cast in a cylindrical mold with a 34 mm diameter and 50 mm height. At an age of 28 days the specimens were demolded and 34 mm diameter 0.8 ± 0.05 mm thick slices were taken from the middle of the samples. The samples were dried under controlled conditions (at $23 \pm 0.1^\circ\text{C}$ and $50 \pm 1\%$ RH) in a CO_2 free chamber until they reach mass equilibrium. Then, samples were submerged for a minimum of 5 days in aqueous solutions with 23% NaCl, 32% CaCl_2 , and 30% MgCl_2 by mass.

For the samples submerged in NaCl, CaCl_2 , and MgCl_2 solutions a 50 mg to 70 mg piece of sample was placed in a tared quartz pan after a minimum of 5 days of submersion. The pan containing the sample was then suspended from the balance (± 0.001 mg accuracy) and placed in the relative humidity chamber to equilibrate at $23.0 \pm 0.1^\circ\text{C}$ and $97.5 \pm 0.1\%$ RH for up to 96 h or until the sample had achieved a stable mass (less than an 0.001% mass change/15 min). Then, the relative humidity was reduced to reach 95%. After the sample mass equilibrated, the relative humidity in the chamber was changed in 10% RH steps to 55% RH, allowing the sample to attempt to equilibrate (12 h or 0.01% change in mass over 15 min) at each new humidity. After equilibrating at 55% RH the samples were dried to 0% RH. For the sample submerged in de-ionized water the procedure was similar, but the relative humidity was reduced in 5% steps from 97.5% to 2.5%, and then reduced to 0% RH.

Fig. 6 shows the plot of mass change as a function of time for the mortar saturated in de-ionized water. The sample soaked in water can be seen to lose mass with the decrease of RH. For this system, when the environment is below 100% RH, water will move from the pores to outside of the sample and classical drying behavior is observed. The maximum mass of the sample is 8.5% higher than the mass of the oven dry sample.

Fig. 7 shows a plot of mass change for the mortar samples submerged in aqueous solutions of 23% NaCl, 32% CaCl_2 , and 30% MgCl_2 . It can be observed that initially upon placement in the testing chamber at 97.5% relative humidity the mass of the sample increases for the first 96 h until the relative humidity of the chamber is changed. The samples absorb water during this time of preconditioning, with values much higher than the 8.5% increase in mass of the sample with de-ionized water as compared with the oven dry sample.

The sample loses weight as the relative humidity is decreased however it should be noted that the sample mass does not decrease to below the initial mass obtained from soaking the sample

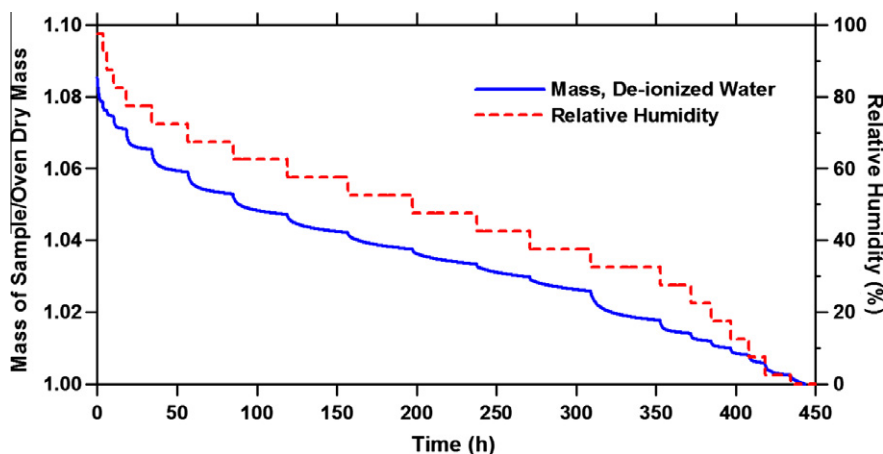


Fig. 6. Mass change at decreasing RH for samples containing de-ionized water.

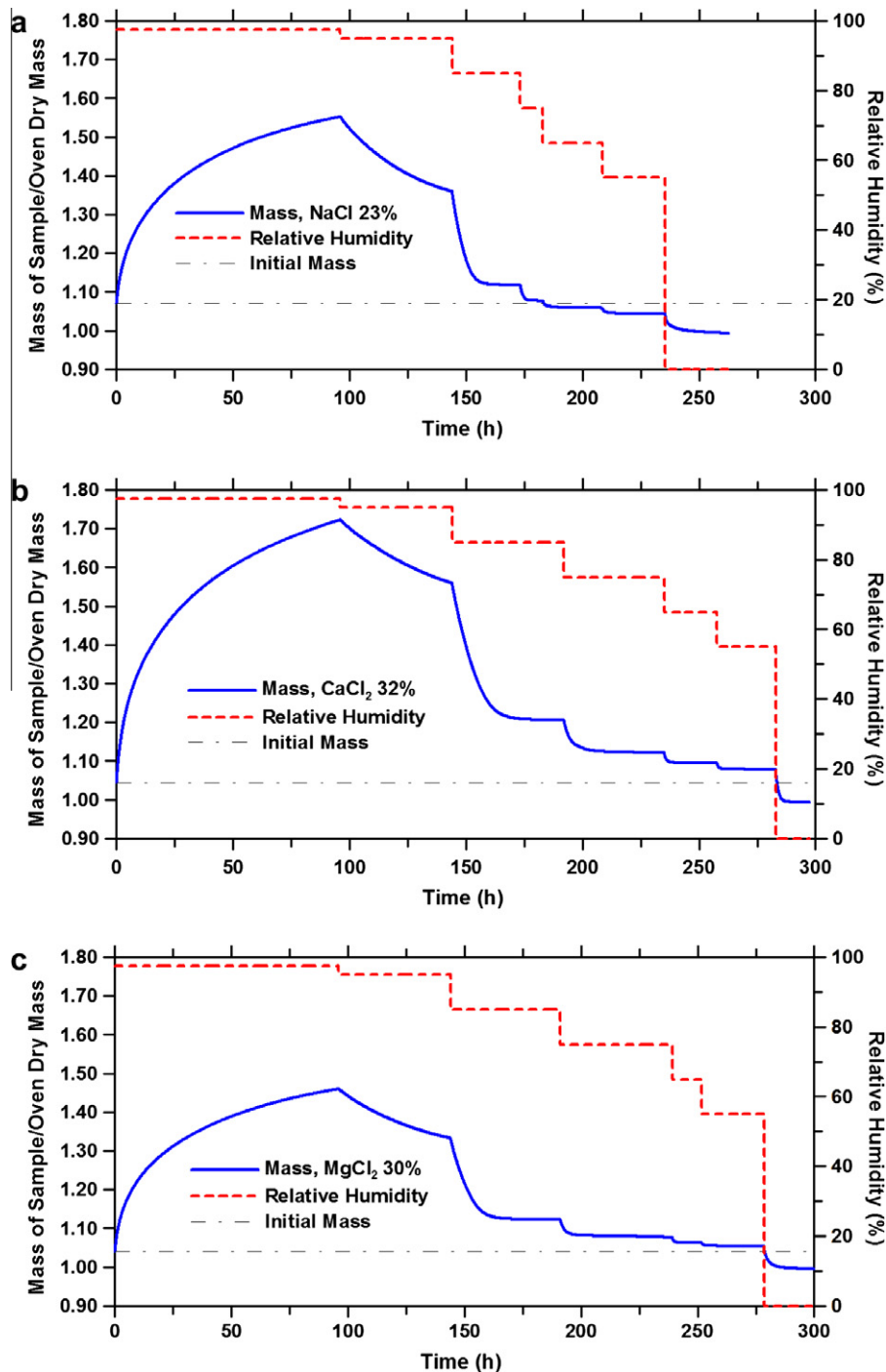


Fig. 7. Mass change for samples submerged in aqueous solutions containing deicing salts: (a) NaCl 23%, (b) CaCl₂ 32%, and (c) MgCl₂ 30%.

in the deicing solution until relative humidity was decreased below 85%, 55% and 55% for NaCl, CaCl₂ and MgCl₂ respectively. This will be compared with the equilibrium relative humidity of the salt solution later in the paper.

6. Discussion of results

6.1. Aqueous solution absorption behavior as a function of surface tension and viscosity

Eq. (3) showed that the rate of absorption was related to the square root of surface tension and viscosity. Fig. 8 plots the square

root of the ratio of surface tension and viscosity versus mass concentration of salt. Pure salt solutions are shown as lines while industrial deicing solutions are presented as solid points, and the open points represent the measured sorption response of concrete (i.e., salt sorptivity/water sorptivity) from Fig. 3. Fig. 8 confirms that as the solution concentration increases, the rate of fluid absorption (i.e., sorptivity) decreases. Further, while the properties of pure solutions may not exactly represent the response of industrially available deicing solutions they do provide a comparable trend. Reasonable agreement is seen between the measured sorption and square root of the ratio of surface tension and viscosity the measured properties. Additional work is currently being

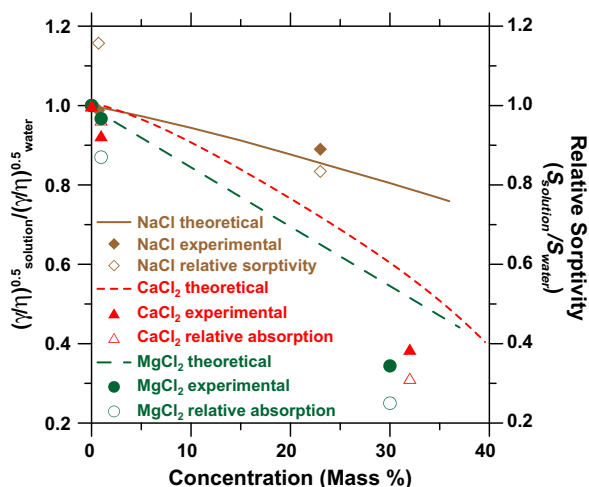


Fig. 8. Relative sorptivity for deicing solutions.

performed to extend these results to a wide range of temperatures.

6.2. Drying time versus wetting time

Comparing Figs. 3 and 4 indicates that wetting happens much faster than drying. When de-ionized water was used as the absorbed fluid, the amount of fluid that was evaporated from the sample after 8 days was $0.8 \text{ mm}^3/\text{mm}^2$. In contrast, it took just 2 h for samples to absorb the same amount of fluid. These differences are even larger when salt solutions were used as the absorbed fluid. When MgCl_2 solution was used as the absorbed fluid, the amount of fluid that was evaporated from the sample after 8 days was $0.07 \text{ mm}^3/\text{mm}^2$, but it took just 10 min for the samples to absorb the same amount of fluid.

This can be attributed to the nonlinear moisture diffusivity of concrete [35–37]. With drying of concrete at the surface, the moisture diffusivity decreases orders of magnitude. This, in effect, forms a “barrier” against moisture diffusion [38].

This is important as it suggests that field concrete may be more susceptible to increasing its level of saturation over time rather than drying out. Further, it shows that laboratory tests that use equal times for drying and wetting increase the saturation level of the concrete over time. Researchers [35] observed an increase in sample mass during wetting and drying cycling with deicers which was attributed to microcracking; however an increase in mass would be consistent with the wetting and behavior observed in this paper.

6.3. Reduced drying with salt solutions – the role of solution equilibrium humidity

The relative humidity of different salt solutions presented in Fig. 1b help to understand the results from the drying tests. The equilibrium relative humidity for the 23% NaCl, 32% CaCl_2 and 30% MgCl_2 solutions are 80%, 40% and 50% respectively. When the samples are placed in an environment with a relative humidity that is greater than or equivalent to the approximate equilibrium relative humidity over the aqueous solution in the pores water will not be lost to then environment. (Fig. 4) and the sample can actually gain mass (Figs. 4 and 7), most likely due to the water absorption on the surface of the sample. This can be seen by the thinner (dashed lines in Fig. 7), which show the initial mass of the sample after it has been submerged in an aqueous solution for over 5 days.

At relative humidity higher than the equilibrium of the aqueous salt solution the samples will increase in mass. At relative humidities where the environment is less than the equilibrium humidity over the salt solution, the samples will be expected to decrease in mass. The drying behavior of systems containing concentrated aqueous solutions of deicing salts is complex and requires additional research.

6.4. Effect of solution on rewetting

When samples of concrete that were previously exposed to deicing solutions were rewetted with water they had an absorption and rate of absorption that depended on the history of the specimens (Fig. 5). The absorption of water can be 30–50% less in specimens that were exposed to deicing solutions at some point in their lives. This is an important, yet subtle, factor to understand. This is important since absorption tests of field concrete may be mistakenly interpreted by relating the reduction in sorption to pore filling or delayed sorption. Both of these observations (lower sorptivity and delayed sorption) are consistent with data here for samples that did not have reduced porosity or differences in sample damage.

7. Summary and conclusions

This paper has reported experimental results of fluid absorption measurements and drying of concrete in the presence of deicing solutions. The following observations can be made. First, the absorption of fluid in concrete depends on the drying environment used to condition the samples. As expected samples stored at a lower RH absorbed a greater volume of fluid. Second, it was observed that the deicing solutions are absorbed at a reduced rate due to the change in viscosity and surface tension. This reduction can be related to the square root of the ratio of surface tension and viscosity [10]. Third, the time scale between drying and wetting is different and concrete is more likely to become preferentially increasingly wet over time. This implies specimens with salt solutions will always have a higher degree of saturation since they dry in a lower relative humidity range and gain water in a wider relative humidity range. Fourth, the drying of concrete containing aqueous solutions with deicers differs from that of water. The equilibrium relative humidity of the aqueous solution plays an important role on limiting drying. In the cases when the equilibrium humidity of the deicing solution is lower than that of the drying environment, the concrete will absorb water. Finally, the presence of deicing salts in field samples impacts the absorption when field samples are tested in the lab using water. Concretes that had previously been exposed to deicing solutions can also exhibit reduced rate of absorption, even if water is the fluid being absorbed. This suggests that care must be taken in analyzing field concrete exposed to deicing salt solutions.

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