The Influence of Deicing Salt Exposure on the Gas Transport in Cementitious Materials

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ABSTRACT

The gas transport properties of concrete can provide information that can be used in the prediction of the service life. However, to interpret the transport properties properly, some corrections may be needed to account for the moisture content and the distribution of moisture in the concrete. This paper investigates the influence of deicing salts on gas transport. Specifically, oxygen permeability and oxygen diffusion were examined for mortar samples initially saturated with different deicing salt solutions and then exposed to drying. The presence of deicing was found to increase the degree of saturation of mortar samples and as such it decreases permeability and diffusivity. These findings were further confirmed through desorption analysis. This study suggests that care must be taken when performing investigations using concrete that may have been previously exposed to deicing salts either in situ or when this concrete is extracted and used for laboratory testing.

Keywords. Deicing salts, gas transport, degree of saturation, desorption

INTRODUCTION

A considerable portion of building materials research focuses on the study of the durability. In concrete research, a large portion of research has focused on developing transport tests that can be used for service life prediction. Specifically, gas permeability and gas diffusivity measurements have attracted the interest of the concrete community due to their quasi-non-destructive nature, the short testing time that is required to perform the test (Kropp & Hilsdor, 1995), and the high reproducibility of the results (Abbas, et al., 1999). Moreover, several methods have been developed to measure in-situ the gas permeability of concrete

(RILEM TC 189, 2007) (Figg, 1973) (Hong, 1989) (Basheer, et al., 2001) (Torrent, 1992).

The description of gas transport is complex and three distinct mechanisms are considered to occur simultaneously or independently. These mechanisms include 1) free-molecule or Knudsen diffusion, 2) molecular or ordinary diffusion and 3) surface diffusion (Sercombe, et al., 2007). Each mechanism is not completely understood but there is some evidence that it is greatly affected by total pressure, temperature, type of gas considered and properties of pore structure available to gas, in terms of pores size and pores connectivity (Sercombe, et al., 2007), (Houst & Wittmann, 1994).

In the literature many studies have focused on the analysis of the parameters that influence gas transport and gas transport testing. It has been shown that porosity and tortuosity can greatly influence the gas permeability and diffusivity measurements (Wong, et al., 2006). From the idealized gas theory, gas permeability varies with pore diameter squared (Dullien, 1992) while gas diffusivity in large pores is independent of the pore size (Mason & Malinauskas, 1983) (Garboczi, 1990). It is important to note however that this depends on both the total porosity and the vapor filled porosity. This points to the fact that the gas transport properties are strongly dependent on the amount of moisture and on its distribution inside the concrete element (Abbas, et al., 1999) (Houst & Wittmann, 1994). For specimens with a higher moisture content, the thickness of the layer of moisture that covers the pores walls increases (Hagymassy, et al., 1969) causing the decrease of the mean free path of gas molecules (Xi, et al., 1994) and the reduction of gas transport (Abbas, et al., 1999). It has been shown that a reduction in gas transport is considerable when the degree of saturation is greater than 60% since the pores relevant for transport process are increasingly filled as the degree of saturation increases (Parrott, 1994).

Many concrete structures are exposed to salts due to either a marine environment or the application of deicing salts to pavements (Spragg, et al., 2011), bridge decks (Wallbank, 1989), or parking lots. The presence of deicing salt is known to influence the durability performance of concrete structures. Evidence of salt deterioration has been reported in masonry structures (Lubelli, et al., 2004), building stones (Birginie, et al., 2000), coastal structures (Berke & Hicks, 1991) and concrete elements (Sutter, et al., 2006). Several mechanisms may be associated with deicing salt damage including pressure that develops due to osmosis, crystallization, intermediate compounds, or the increase of the risk of frost damage due to the increase in the degree of saturation (Litvan, 1976), (Li, et al., 2012) (Scherer, 1999) (Scherer 2011, Personal Communication). Salts are also responsible of chemical interaction within the concrete, resulting in leaching and decomposition of the hydrated cement products, accelerated concrete carbonation, or alkali-silica reaction (Wang, et al., 2006) (Rangaraju, et al., 2005). This paper will focus on the potential influence of deicing salts on the gas transport properties.

The effect of salts on gas transport properties has been studied for rocks (Peysson, et al., 2011) (Birginie, et al., 2000). However, very few studies can be found that investigate the influence of salt on gas transport for concrete. One study was found where salt saturated concretes were preferentially used to help the concrete for use in transuranic waste repository (Pfeifle & Hansen, 1996).

The presence of deicing salts alters gas transport due to two primary reasons. First, the salts can change the moisture content (degree of saturation) and moisture distribution in the pore

structure of the cementitious system. It is known that deicing salts change the density, viscosity, surface tension, and water activity of the solution (Spragg, et al., 2011). Salts change the drying process (Villani, et al., 2012) and alter the equilibrium relative humidity of the pore solutions (Benavente, et al., 2003) (Villani, et al., 2012). Consequently, samples containing deicing salt are likely to have a higher degree of saturation when compared with a sample containing water at the same relative humidity (Villani, et al., 2012) (Castro, et al., 2011) (Litvan, 1976). Since the degree of saturation is higher in systems containing salts, a lower gas permeability/diffusivity can be expected (Abbas, et al., 1999). Second, the salt can precipitate in the pores (Scherer, 1999). In cementitious systems that initially contain salt solutions, it is known that when the water evaporates the solution becomes more concentrated. The concentration gradient generated causes the salt to begin to diffuse in the convective flow is much higher compared to the diffusive flow (Peysson, et al., 2011) the system will results in non-homogeneous salts distribution. Non-linear slat distributions can also develop if the sample has a moisture gradient before the deicing salt is introduced.

MATERIALS AND SAMPLES PREPARATION

Gas permeability, gas diffusivity and desorption tests were performed using mortar. The mortar had a water-to-cement ratio of 0.42 with 55% aggregate by volume. The mixing procedure was in accordance with ASTM C 192.

For gas permeability and gas diffusivity, cylindrical samples (diameter of 102 mm (4 in.) and length of 204 mm (8 in.)) were prepared. The samples were demolded at an age of 24-hours and then sealed in double plastic bags for approximately 12 months. The cylinders were then cored using a 68 ± 2 mm diameter bit. The samples were then cut using a wet saw to obtain samples with a height of 25 ± 2 mm (1 in.) for oxygen permeability measurements. For oxygen diffusivity measurements larger samples were used. The oxygen diffusion samples were obtained from 102 by 204 mm cylinders (4x8 in.) using a wet diamond saw to obtain samples with 102 mm (4 in.) of diameter and 50 mm (2 in.) length.

A total of 16 samples were prepared for oxygen permeability and 8 samples were prepared for oxygen diffusivity. The number of samples used is based on the variability of each test. Specimens were initially oven dried at $50^{\circ} \pm 0.5^{\circ}$ C and $30\% \pm 1\%$ RH until constant mass and subsequently submerged in four different aqueous solutions: deionized water, 16% calcium chloride (CaCl₂), 15% of magnesium chloride (MgCl₂) and 17.5% sodium chloride (NaCl). The concentrations of the solutions are reported as percentage by mass. In each solution, four oxygen permeability samples and two oxygen diffusivity samples were placed and kept submerged until their mass reached the equilibrium ($\Delta m < 0.2\%$ within 7 days). The specimens were then conditioned in a temperature and humidity controlled chamber at 65% $\pm 1\%$ RH and at $23^{\circ} \pm 0.5^{\circ}$ C until equilibrium ($\Delta m < 0.1\%$).

For desorption analysis, smaller cylinders (34 mm of diameter and 50 mm tall) were prepared. The samples were sealed after casting and remained sealed until demolding (at 28 days). Thin slices of mortar (0.80 \pm 0.05 mm) were cut from the cylinder using a water cooled diamond tipped wafer cut saw. The slices of material were stored at 23° C \pm 0.1° C and 50% \pm 2% RH, in a CO₂ free chamber. Prior to testing, samples were submerged for 24 hours in their respective solutions.

TESTING METHODS AND ANALYSIS

Oxygen Permeability. The oxygen permeability has been measured using a falling head permeameter (Ballim, 1991) (Alexander, et al., 1999). This test involves the measurement of a unidirectional flow of gas through the specimen due to the presence of a pressure gradient between the upper and the lower side of the sample. The bottom surface of the sample is in contact with the oxygen contained in the vessel at a known pressure, while the top surface is at the atmospheric pressure. The sides of the sample are sealed using a compressed rubber collar to insure unidirectional flow. The gas flow is quantified by monitoring the pressure drop with time, with the pressure of the initial time of 100 kPa. The pressure is monitored for a 6 hours period (or until the pressure has decreased to a pressure of 50 kPa (Alexander, et al., 1999)).

The oxygen permeability (k) can be evaluated by combining D'Arcy's law for the case of flow of compressible gas with the ideal gas law as shown in Equation (1) (Ballim, 1991):

$$k = \frac{\omega \cdot V \cdot g \cdot d}{R \cdot A \cdot T} \cdot \frac{1}{t} \cdot \ln\left(\frac{P_0}{P}\right) \tag{1}$$

where ω is the molecular mass of oxygen (0.032 kg/mol), V is the volume of oxygen under pressure, which is precisely known from the instrument manufacturer (m³), g is the acceleration due to gravity (9.81 m²/s), R is the universal gas constant (8.314 N·m/K·mol), A is the cross sectional area of the sample (m²), d is the sample thickness (m) and T is the absolute temperature, 296.15° K (23±1° C).

Oxygen Diffusivity Tests. The oxygen diffusivity was measured using an instrument originally developed by Lawrence et al. (1984). The samples used for oxygen diffusivity testing are fitted in a silicon rubber ring that is compressed to provide a condition that insures unidirectional flow. The test is performed by exposing one surface to a stream of pure oxygen and the opposite surface to a stream of pure nitrogen at the same pressure. The oxygen and nitrogen gases counter diffuse through the sample and the oxygen concentration in the outlet cell is measured with a zirconia analyzer (Servomex 4100) at 0, 10, 20, 60 and 90 minute.

To compute the oxygen diffusivity from the test it is assumed that oxygen is transported in concrete by ordinary and Knudsen diffusion (Schwiete, et al., 1969). Equation (2) has been developed based on Fick's law which enables an average diffusion coefficient (D_N) as shown (Lawrence, 1984):

$$D_N = \frac{RT}{P} \frac{Q_{mol/s} \cdot L}{A \cdot \Delta C} = \frac{Q_{m^3/s} \cdot L}{A \cdot \Delta C}$$
(2)

where $Q_{mol/s}$ or $Q_{m3/s}$ is the rate of oxygen diffusion at atmospheric pressure, either (mol/s) or (m³/s) respectively, *L* the thickness of the sample (m), *A* the cross section area of the sample (m²) and ΔC is change in concentration through the specimen (%). The change in concentration ΔC is evaluated considering the flow rate of oxygen and the flow rate of nitrogen, the pressure of the gas stream, and the initial and final concentration of oxygen.

Desorption. Desorption isotherms have been measured using an automated sorption/desorption analyzer (TA Q5000) where the samples have been subjected to drying at a constant temperature over a range of relative humidity. The relative humidity between 0% and 98.5% are generated by mixing dry and wet nitrogen streams. The 50 to 70 mg samples were submerged in an aqueous solution before being placed in a tared quartz pan. The pan was then suspended in a relative humidity chamber to equilibrate at 23.0 ± 0.1 °C and at the equilibrium relative humidity for up to 96 hours or until the sample had achieved a stable mass (less than an 0.001% mass change/15 min). The equilibrium RH is the water activity of the deicing salt solution in which the samples were stored for 24 hours (85.0% for 16.0% calcium chloride solution (CaCl₂), 87% for 15.0% magnesium chloride (MgCl₂) solution and 87% for 17.5% sodium chloride solution (NaCl) (Spragg, et al., 2011). The relative humidity was then reduced in 10% RH steps until 5% RH, allowing the sample to equilibrate (12 hours or 0.001% change in mass over 15 min) at each humidity. After that the sample was dried to 0% RH.

RESULTS AND DISCUSSION

Oxygen Permeability and Oxygen Diffusivity Results. Oxygen permeability is presented as function of the degree of saturation in Figure 1 and in Figure 2. The variability (standard deviation) between four samples for oxygen permeability and between two samples for oxygen diffusivity is also reported. Oxygen diffusivity measurements were performed applying a pressure of 1.0 bar and an oxygen flow rate of 55 ml/min.

The degree of saturation (DOS) has been calculated by dividing the difference between the mass of the sample at the time of testing and the mass of the sample after oven drying by the difference between the sample mass at saturation and the oven dried sample mass. As a result, when a sample that contains a salt solution is completely dried (i.e., 0% RH) the degree of saturation is not zero and this could be expected since salt crystals will remain in the system.



Figure 1: Oxygen permeability versus degree of saturation (DOS)

Figure 2: Oxygen diffusivity versus degree of saturation (DOS)

It is apparent from Figure 1, 2 that the presence of deicing salts increases the degree of saturation. As the degree of saturation increases a reduction is observed for the oxygen permeability and diffusivity. A sudden decrease in the case of oxygen diffusivity (Figure 2) can be seen when the DOS is higher than 60% since the main pores involved in gas transport are becoming filled and disconnected. This is in accordance to previous research results that related the degree of saturation with transport properties (Abbas, et al., 1999).

The difference in the magnitude of the decrease in the oxygen permeability compared to oxygen diffusivity is likely related to the different sensitivity that occurs between the two instruments.

Influence of Deicing Salts on the Desorption of Samples. The higher degree of saturation seen in Figure 1, 2 can be explained by the drying of mortar containing deicing salts. The drying behavior was examined using desorption tests. The results presented in Figure 3 show the mass of solution (i.e., the mass of the salt and water) in the sample as function of relative humidity



Figure 3: Desorption curves for mortar samples submerged in salt water solutions

Figure 4: Mass change during each RH step for samples submerged in 16% calcium chloride and deionized water.

At a given relative humidity (RH) the amount of solution in the samples that contain salt is higher than it is for samples containing only water. During drying, water evaporates from the solution while salts remain in the samples. Consequently, an increase in salt solution concentration occurs during drying.

A slower drying process occurred when salts are present due primarily to the ionic interaction between solute (salts) and solvent (water) (Benavente, et al., 2003). The ionic interaction reduces the water loss in a given time period, when compared to drying of samples containing only water. This behavior was noticed during the desorption analysis as samples containing salts showed an average equilibrium time (for each RH step) 2 to 5 times longer

than the sample with only water (Figure 4). This change has been shown in the diffusion coefficient for a wider range of deicing salts and salt solutions (Villani, et al., 2012).

The slower rate of drying that occurs when salts are present is also related to the change in the equilibrium relative humidity of the solutions (Benavente, et al., 2003) (Villani, et al., 2012). The reduction of the equilibrium relative humidity is a consequence of the change in the liquid properties in terms of surface tension, molecular volume and activity for solution containing salts compared to pure water. For a system initially saturated, the decrease in equilibrium relative humidity causes a delay in the initiation of drying (as RH is decreased) since the drying will start only when the external relative humidity is lower than the equilibrium relative humidity of the solution in which the sample was saturated. This is true assuming the same pore structure for the samples tested. If the relative humidity is higher than the equilibrium relative humidity the sample may gain mass due to hygroscopic nature of salts (Koelhler, et al., 2006) (Spragg, et al., 2011) (Villani, et al., 2012).

Salt distribution analysis. As mentioned in the introduction, the decrease of gas permeability and diffusivity in presence of deicing salts might be due also to the salts precipitation in the pores during drying. To investigate this, chloride profiles were determined from samples previously used for oxygen diffusivity measurements. The samples were ground with a milling machine using diamond tipped drill bit and the powders was collected every 2 mm depth. The chloride content was then measured using an automated titration procedure, which is a modification of AASHTO T260 (Di Bella, et al., 2012).

Although the samples were totally submerged and all surfaces were exposed to solution, the analysis of chloride distribution has been done only in the direction of gas flow (along the height of the cylinder). The influence of lateral absorption is assumed constant for all depths.

A similar trend in the chloride profile can be observed for the three salts. A higher salt concentration occurs in the first 15 mm and the inner core (20 mm) shows a uniform salt distribution. Since the samples have reached equilibrium while submerged in solution, we expect the solution have saturated the entire sample and consequently the non-uniform distribution has been generated during drying (wicking action).

The reduction of porosity due to possible salts precipitation has been calculated. The following assumptions have been made. First, during drying, only water is lost. We assume that the original concentration of salt precipitated. Second, salt precipitation occurred primarily in the external layer (15 mm thick) where the chloride profiles show a gradient.

The amount of salt precipitated m_{salt} has been calculated using the following equation:

$$m_{salt} = \left(\frac{C}{1-C}\right) \cdot m_{water} \tag{3}$$

where C is the initial concentration of the salts solutions where the samples have been submerged (-) and m_{water} is the amount of water evaporated during drying (g).

The results are presented in Table 1. The total porosity has been evaluated according to ASTM C642, substituting the boiling procedure suggested with vacuum saturation. The

reduction in porosity $\Delta \Phi$ has been evaluated dividing the volume occupied by salts $(m_{salt}/2 \cdot / \rho_s)$ over the total porosity on the 15 mm thick cylinder ($\Phi_0 \cdot V_{cyl-15}$).

		5	1	
	m _{salt}	Density of	Total Porosity	Reduction in
		solid salt ρ_s	Φ_0	Porosity $\Delta \Phi$
	(g)	(g/mm^3)	(%)	(%)
15.0% MgCl ₂	5.98	2.32E-03	20.3	4.2
16.0% CaCl ₂	6.00	2.15E-03	20.3	5.8
17.5% NaCl	3.36	2.16E-03	20.3	2.5

Table 1: Porosity Reduction due to Salt Precipitation

The reduction in porosity (5%), even having assumed the worst conditions, is much smaller compared to the actual reduction in oxygen diffusivity seen (12% to 70% in presence of salts, with respect to water submerged samples). Some obstruction may occur at pore necks which could have a greater impact.

CONCLUSIONS

The influence of deicing salts on gas transport properties has been examined experimentally using mortar samples. Two factors influence the gas transport properties in samples containing deicing salts. The first factor is that deicing salts in solution may precipitate in the pores due to drying and therefore reduce the gas transport (i.e., occlude). The second factor is that deicing salts in solution can increase the degree of saturation of the sample.

The first factor has been demonstrated to be relatively small. The second factor (increased degree of saturation) is a primary cause for the reduction in gas permeability and diffusivity in the mortar samples. The desorption isotherm of concrete containing deicing salt solution showed a considerable increase in the degree of saturation (DOS) when compared to system that contains pure water. The results from the mortar samples indicated that deicing salts influence the gas permeability and diffusivity of concrete. The practical implications are that testing of field samples needs to carefully consider the conditioning history of the samples.

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