Effect of confining pressure and microcracks on mass transport properties of concrete

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Abstract
This paper investigates the effect of low confining pressure on transport properties of cement-based materials and establishes if it can be used to study the influence of microcracks on transport. Oxygen diffusivity and permeability of paste and concrete (w/c ratios: 0.35 & 0.50; curing ages: 3 & 28 days) were measured at increasing confining pressures up to 1.9MPa (4-8% of 3 day compressive strength). Prior to transport testing, samples were subjected to gentle stepwise drying at 21°C or severe oven drying at 105°C to induce microcracking. Microcracks were quantified using fluorescence microscopy and image analysis. Permeability decreased significantly with increasing confining pressure and this was more significant for samples with a greater degree of microcracking. Image analysis shows that microcracks undergo partial closure when confined, but the total accessible porosity was not significantly affected. Implications of these results with respect to the influence of microcracks on transport properties are discussed.

Keywords: Microcrack, microstructure, permeability, diffusivity, transport properties, confining pressure, durability, cement-based materials

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1. Introduction

The transport properties of cementitious materials have been studied for many decades. This is because movement of aggressive species such as chlorides, carbon dioxide, oxygen, sulphates and alkalis are responsible for most deterioration processes affecting concrete structures including reinforcement corrosion, sulphate attack and alkali-aggregate reaction.

Many transport tests, such as gas diffusion, gas permeation and water permeation, require the sample to be confined and sealed to prevent leakage through the sides of the sample during testing. The sample can be sealed using a variety of methods. These include sealing with epoxy and silicone [Kermani, 1991; CRD-C 48-92, 1992; Wang et al., 1997], or by mechanically loading a rubber ring that expands laterally to seal the sample [RILEM TC 116-PCD (CEMBUREAU method); Hearn & Mills, 1991], or by air/oil pressure through a rubber sleeve or membrane (Hassler cell) [Whiting, 1988; CRD-C 163-92, 1992; Chen et al., 2013].

However, it is not common to specify, measure, or report the confining pressure applied on the sample in research publications. Where this information is available, we observed that a large variation in confining pressure ranging from 0.7 MPa up to 5.4 MPa has been used in previous studies [RILEM TC 116-PCD; Chen et al., 2009; Perlot et al., 2013]. This is surprising because concerns may be raised regarding possible fluid leakage through the sides of test samples when a low confining pressure is used. Similarly, damage to the microstructure may occur if samples are subjected to a high confining pressure [Hooton, 1988; Banthia & Bhargava, 2007]. Furthermore, concrete inevitably suffers from microcracking due to tensile stresses from drying shrinkage and thermal effects. These microcracks may close up when the sample is confined, which may subsequently influence the measured transport properties. However, the effect of confining pressure and microcracks on the transport properties of concrete is not well understood. Consequently the correct procedure for measuring the transport properties of microcracked concrete is uncertain.

Numerous studies have been carried to understand the effect of mechanical load induced macro-cracks on transport properties of cementitious materials. In many studies, the samples were subjected to stresses to induce cracking, unloaded and then tested for transport properties [Kermani, 1991; Samaha & Hover, 1992; Wang et al., 1997; Aldea et al., 1999a,b; Hearn, 1999; Picandet et al., 2001; Akhavan et al., 2012; Djerbi Tegguer et al., 2013]. In some studies, transport measurements were carried out while the sample was simultaneously subjected to a load [Hearn & Lok, 1998; Banthia & Bhargava, 2007; Desmettre & Charron, 2012; Chen et al., 2013; Rastiello et al., 2014]. However, very few studies have been carried out on the relationship between cracks and transport properties of concrete, where the crack characteristics and transport properties were simultaneously measured under load [Desmettre & Charron, 2012; Rastiello et al., 2014].

Most of the literature reviewed above concerns the transport properties of concretes containing mechanically-induced damage produced by loading the sample at 30% up to 100% of ultimate strength or by controlling the crack opening displacement from 25 μm up to 0.55 mm that produces relatively large cracks. There is generally a lack of studies on the influence of drying-induced microcracks and this is surprising considering that most concrete structures are subjected to drying shrinkage. This is partly due to the fact that drying-induced microcracks are small (<10 μm) and heterogeneous, and partly due to difficulties in studying microcracks in controlled experiments. There are also difficulties in isolating the influence of microcracks from other factors such as moisture content and accessible porosity that inevitably change when concrete is dried and have major influences on transport.

This paper presents an investigation of the effect of confining pressure on the oxygen diffusivity and permeability of pastes and concretes that have been subjected to different drying regimes to induce varying levels of microcracking prior to transport testing. The drying-induced microcracks
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were characterised using fluorescence microscopy and image analysis. The aim of the research is two-fold: 1) to determine the influence of relatively low confining pressure on measured transport properties and 2) to establish whether measuring the transport property of a sample at increasing confining pressure can be used as a means to isolate and study the influence of microcracks on mass transport properties of concrete.

2. Experimental

2.1 Materials and mix proportions

Paste and concrete samples were cast with ordinary Portland cement CEM I 32.5R at free water/cement ratio of 0.35 and 0.50. The oxide composition of the cement is shown in Table 1. The estimated Bogue composition of the cement is 52.7% C₃S, 19.3% C₂S, 10.6% C₃A, 7.4% C₄AF by mass. The loss on ignition, fineness and specific gravity of the cement are 2.1%, 2905 cm²/g and 3.06 g/cm³, respectively (Table 1). Thames Valley sand (< 5 mm) and limestone (< 10 mm) were utilised as fine and coarse aggregates and their particle size distributions are shown in Fig. 1. The limestone aggregate complies with BS EN 12620:2002+A1 overall grading, and the sand complies with the BS 882:1992 medium grading. The specific gravity, 24-hour absorption, and moisture content of the aggregates are given in Table 2. The total aggregate volume fraction in the concrete samples was either 60% or 68%. Batch water was adjusted to account for aggregate absorption so that the target free w/c ratio was achieved. Mix proportions for all samples are shown in Table 3.

2.2 Sample preparation and conditioning regime

Three replicate cylindrical samples of 50 mm thickness and 100 mm diameter were cast for each mix for transport measurements and microcrack characterisation. Three replicate cube samples (100×100×100 mm³) were cast for compressive strength and density tests. Cement and aggregates were dry mixed in a 30-litre capacity pan mixer for around 30 seconds. Water was then added and mixed for a further 3 minutes. A vibrating table with adjustable intensity was used for compaction. Samples were cast in steel moulds, compacted in three layers and each layer was vibrated until no significant amount of air bubbles escaped. The compacted samples were covered with plastic sheet and wet hessian at room temperature for the first 24 hours. Afterwards, samples were demoulded and sealed cured at room temperature (21°C) to 3 or 28 days by wrapping with a least 6 layers of cling film and sealing in plastic bags.

After curing, samples were sealed on the curved surface by two layers of waterproof adhesive tape to ensure one-dimensional moisture flow during conditioning. This is deemed to be a more realistic representation of the way in which most structures dry in service. Samples were subjected to two conditioning regimes to dry the samples until mass equilibrium prior to transport testing and to induce microcracking. The conditioning regimes are: a) oven drying at 105°C, and b) stepwise drying at room temperature (21°C) from 93% RH to 86%, 76%, 66% and 55% RH in a CO₂ free environment. The stepwise drying regime induces a much smaller moisture gradient within the sample and slower drying rate, so this should reduce the amount of microcracking. The actual RH in the conditioning chamber is ±3% RH of the targeted value. Samples were dried at each RH until mass equilibrium (<0.01% mass loss per day) before moving to the next RH step. The entire conditioning regime took about 14 days for the 105°C oven drying and 400 days for the stepwise drying regime to reach mass equilibrium. Oven-dried samples were then cooled to 21°C in a vacuum desiccator prior to transport testing. Samples were kept in their respective conditioning chamber or desiccator to avoid any moisture change over the period of testing.

It should be noted that the 105°C drying condition imposed in this study is more severe than the natural drying experienced by most structures. However, this drying regime is similar to those
employed in other related studies (e.g., Lion et al. [2005], Chen et al. [2013]) and it is a conditioning regime that has been recommended by codes of practice (e.g., RILEM TC 116-PCD).

2.3 Transport tests

Two transport properties (oxygen diffusivity and oxygen permeability) were measured since the influence of confining pressure and microcracks were expected to vary for different transport mechanisms. In both tests, the sample is placed in a silicone rubber ring which is then fitted into an outer steel ring of the test cell and covered with a steel plate (Fig. 2). Compressive load is then applied on the silicone rubber ring, which expands laterally and produces a near-uniform compressive stress to seal the assembly (Fig. 2b). This provides an air tight grip on the sample and prevents side leakage so that flow occurs through the sample.

Oxygen diffusivity was determined by exposing the opposite flat faces of the sample to streams of O\(_2\) and N\(_2\) at equal pressure. The gases counter-diffuse and the concentration of O\(_2\) in the outflow stream was measured with a zirconia analyser at steady-state conditions to determine diffusivity. Steady-state flow was achieved within 30 minutes to 1 hour. Oxygen permeability was determined by applying a gas pressure at 0.05, 0.15 and 0.25 MPa above atmospheric pressure and measuring the steady-state flow rates. Steady-state flow was achieved within 30 to 50 minutes for each applied pressure. The apparent permeability coefficient (\(k_g\)) was calculated following Darcy’s equation at each pressure, from which the intrinsic permeability (\(k_{int}\)) was determined by applying Klinkenberg’s correction. This was done by fitting \(k_g\) to the equation \(k_g = k_{int} (1 + \beta/P_m)\), where \(P_m\) is the mean pressure and \(\beta\) is the Klinkenberg constant, which is dependent on the characteristics of the gas and sample. The intrinsic permeability (\(k_{int}\)) was obtained from the y-intercept of the best-fit line of the \(k_g\) versus 1/\(P_m\) plot. The coefficient of regression of the least-squares fit in the Klinkenberg correction was always greater than 0.99. Three replicate samples were tested and the results averaged. Further details of the test methods are described in Wong et al. [2007].

2.4 Confining pressure

Oxygen diffusivity and permeability were measured whilst the sample was subjected to several levels of confining pressure. This was achieved by increasing the compressive load applied to the silicone rubber ring. A pressure sensitive film (Fujifilm) was placed between the sample and the silicone rubber ring (Fig. 2). The film develops a purplish-red colour when pressure is applied and its colour intensity increases with increase in contact pressure. Because the colour intensity is proportional to the applied pressure, the confining pressure acting on the sample can be determined from a pressure-colour reference chart. The error in the measured pressure is less than ± 10% according to the manufacturer (Fujifilm). The silicon rubber ring was loaded at 10, 15, 25 and 35 kN. To ensure that the lowest applied load is sufficient to seal the sample during the transport test without gas leakage, blank tests on a steel disc with similar dimensions to the test sample were performed at loads of 7, 8, 9 and 10 kN. Oxygen gas at 0.25 MPa pressure was applied to the steel disc and the amount of gas leakage was measured at the outlet to verify the effectiveness of the seal.

2.5 Compressive strength and density

Concerns may arise that the applied confining pressure causes damage to the sample. Hence, it is necessary to compare the maximum confining pressure applied to the test specimen with its compressive strength. The compressive strength [BS EN 12390-3:2009] and relative density tests were performed on 100 mm cubes for all the 3-day cured pastes and concretes and the replicate test results were averaged. The relative density was measured by weighing the cubes first in air and then fully immersed in water.
2.6 Microcrack characterisation using image analysis

Fluorescence microscopy and image analysis were carried out to study the effect of conditioning regime and confining pressure on drying-induced microcracks. Samples were impregnated with fluorescence-dyed epoxy while being subjected to a confining stress of 0.6MPa or 1.9MPa. This was achieved by pouring a low-viscosity epoxy onto the top flat surface of the sample and then applying compressed air at 0.7MPa above atmospheric pressure for 6 hours to force the epoxy into the sample. The impregnated sample was then taken out of the test cell and kept for another 2 days until the epoxy sufficiently hardened. Subsequently, the sample was sectioned with a diamond saw to produce four 8-mm thick slices to examine the epoxy intruded area and degree of internal microcracking. The slices were ground using silicon carbide paper of grit sizes 80 and 120 to obtain flat surfaces for imaging.

Each slice was illuminated with a 15W UV lamp to induce fluorescence and then photographed in a dark room with a 24MP digital SLR camera. The camera was operated at a small aperture to increase depth of field and slow shutter speed to achieve adequate exposure. This is sufficient to obtain a macro view of the cross-section and to measure the epoxy intruded area and depth. Subsequently, the epoxy impregnated portion of the slice was imaged at a higher resolution for microcrack analysis using a petrographic microscope (Olympus BX51) operated in fluorescence mode. A large number of overlapping images was captured at 50x magnification (2048×1536 pixels, pixel size 0.89 µm). The images were then aligned and stitched together to produce a large montage of the sample and microcracks.

Image analysis was carried out to measure the epoxy intruded area fraction and average epoxy intruded depth. The fraction of epoxy intrusion area was obtained by dividing the impregnated area by the sample cross sectional area. The average epoxy intruded depth was obtained by dividing the impregnated area by the width of the sample. All detectable microcracks were carefully traced to obtain a crack binary image for analysis. Tracing was done manually on the enlarged montage and its accuracy was checked frequently by cross-referencing with the actual live image. The number of microcracks, their average orientation with respect to the exposed surface, crack density, width and length distribution was measured using image analysis. Crack density was defined as the total length of microcracks divided by the impregnated area. To obtain the distribution of crack orientation, width and length, a series of 50 µm spaced horizontal gridlines was placed on the montage and measurements were made where cracks intersected the gridlines.

3. Results

3.1 Confining pressure

Fig. 3 shows the colour intensities obtained on the pressure-sensitive film when the sample was confined at different applied compressive loads. The figure shows that the colour density increased with the increase in applied load. The corresponding confining pressures are 0.33, 0.57, 1.15 and 1.93MPa for applied compressive load of 10, 15, 25 and 35kN respectively. As expected, the measured confining pressure varies linearly with applied load. The best-fit line across the measured data has a regression coefficient of greater than 0.99.

Table 4 shows the results from the blank tests on a steel disc confined at loads of 7, 8, 9 and 10kN. The results show that a compressive load of 9kN and above is sufficient to prevent gas leakage through the curved face of the test sample. A compressive load of 9kN corresponds to a confining pressure of 0.3MPa, which is 20% higher than the highest applied gas pressure in the permeability test. Note that in the actual testing of cement paste and concrete samples, compressive loads of greater than 10kN are used. Therefore, we are confident that the transport test results on the cement paste and concrete samples are not affected by leakage through the curved face.
3.2 Compressive strength and relative density

The 3-day compressive strength and relative density of the test samples are shown in Table 5. As expected, samples with lower w/c ratio had higher compressive strength and relative density. The compressive strength ranged from 23.5 to 49.0 MPa. Therefore, the maximum applied confining pressure of 1.9 MPa is only 4-8% of the sample compressive strength. This is small relative to the sample strength and is not expected to cause additional damage to the sample, which would otherwise influence the measured transport properties.

3.3 Transport properties

Table 6 shows the measured transport coefficients and precision expressed as standard error (σ/√n). Unfortunately, it was not possible to obtain reliable results for C 0.5-60-28d (105°C dried) at 0.3 MPa confining pressure. This particular set of data displayed unusually high variability between replicates and so meaningful comparison with other data in the series cannot be made and the applied Klinkenberg correction for permeability measurement produced a poor R² correlation coefficient. These issues are indicative of the seal failing during testing causing gas leakage through the side, therefore this set of data was not considered in the analysis. It should also be noted that sample C 0.5-68-3d that was oven dried at 105°C was subsequently reconditioned at 21°C at increasing RH of 33%, 55% and 66% until mass equilibrium. The sample was then tested for transport properties at different confining pressures and the results are indicated with an asterisk in Table 6.

The measured diffusivity ranged from 3.8E-8 to 63.6E-8 m²/s, while permeability ranged from 2.2E-17 to 71.9E-17 m². Concrete samples tend to produce lower transport coefficients relative to paste samples. This is almost certainly due to the reduction in volume and increase in tortuosity of the pores as a result of the aggregate particles. For example, the diffusivity of concretes after 105°C drying is about 70% lower than the corresponding cement paste. This magnitude of decrease is within the range predicted using a three-dimensional model of diffusivity of concretes containing ellipsoidal aggregate, which accounts for the effect of aggregate dilution, tortuosity and presence of porous aggregate-paste ITZ [Dehghanpoor et al., 2013]. As expected, the transport coefficients of pastes or concretes increase with increase in w/c ratio, and with increase in severity of the drying regime. This trend is observed regardless of the applied confining pressure during transport testing. For example, the measurements carried out at 0.3 MPa are plotted in Fig. 4 to show the effect of w/c ratio and drying regime on transport. One exception however, is P 0.5-3d (stepwise) which showed a slightly lower permeability compared to P 0.35-3d (stepwise). This is probably an experimental error.

Fig. 5 shows the Klinkenberg constants (β) plotted against oxygen permeability. The Klinkenberg constants ranged from 0.05 to 0.22 MPa, and show a general decreasing trend with increase in permeability. These observations are consistent with previous studies [e.g. Bamforth, 1987]. In addition, there is a slight increase in β with increase in the applied confining pressure.

The results clearly show that increasing the severity of drying (stepwise → 105°C) produces a far greater impact on the measured transport properties compared to that obtained by increasing w/c ratio (0.35 → 0.50). Comparing data obtained at the same w/c ratio, curing age, and confining pressure, the measured diffusivity after 105°C drying is a factor 2 to 7.8 (2 to 25.7 for permeability) higher than that obtained after stepwise drying. In contrast, increasing the w/c ratio from 0.35 to 0.5 increases measured diffusivity by only a factor of 1.4 to 1.5 (1.3 to 4.2 for permeability). It would be tempting to attribute these changes in transport properties to drying-induced microcracks, thereby enabling one to quantify the influence of microcracks on transport. However, this is not as
straightforward as it seems because the stepwise dried samples also contain microcracks (Section 3.5). Furthermore, changes in moisture content during drying would also make a significant contribution to increase in transport. Removing more moisture increases the accessible porosity and this effect is compounded by microcracks that develop with drying. The pores and/or microcracks may close as a result of the applied confining pressure and this will be investigated in Section 3.5.

3.4 Influence of confining pressure on transport properties

Fig. 6 shows the normalised oxygen diffusivity and oxygen permeability plotted against confining pressure. The normalised coefficients are obtained by dividing the measured values by the value obtained at the lowest confining pressure for each series. Results for diffusivity and permeability are plotted in separate graphs, but using the same scale on the y-axis to aid comparison.

The results in Fig. 6 (a & c) clearly show that regardless of the w/c ratio, oxygen diffusivity of pastes and concretes subjected to stepwise drying stayed relatively constant when the test was carried out at increasing confining pressure. For samples subjected to 105°C drying, a minor decrease in diffusivity (up to 4%) was observed when confining pressure was increased to 1.9MPa. However, it is interesting to observe that oxygen permeability decreased significantly with increase in confining pressure for all samples (Fig. 6 b & d). Regardless of w/c ratio, this effect was more significant for pastes compared to concretes, and for samples conditioned at 105°C compared to samples conditioned by the ‘gentle’ stepwise drying. For example, the magnitude of decrease in permeability ranged from 14% to 46% for 105°C dried samples compared to 6% to 15% for stepwise dried samples when confining pressure was increased to 1.9MPa.

3.5 Microcrack characteristics

Fluorescent microscopy and image analysis show that microcracks were present in all test samples, including those that were conditioned by the gentle stepwise drying. The number of microcracks that was observed and measured per sample ranged between 196 and 702. The smallest and largest microcrack widths were 1 and 60 µm, respectively. The severity of microcracking induced by different drying regimes is shown in Table 7, and in Fig. 7. The results collectively show that the samples subjected to 105°C drying had wider, longer, and more microcracks compared to those subjected to stepwise drying. The average width increased by a factor of 1.5-2.1, while the crack density increased by a factor of 2.2-4.2 when dried at 105°C compared to those subjected to stepwise drying. Most of the microcracks develop at an angle approximately perpendicular to the exposed surface. This is indicative of the microcracks being induced by drying shrinkage.

The image analysis results showing the influence of increasing confining pressure on epoxy intruded area and microcracks are presented in Table 8 and Fig. 8. It is interesting to note that the average epoxy intruded area and epoxy intrusion depth were relatively similar for the two applied confining pressures (Table 8). This suggests that increasing the confining pressure did not cause a significant decrease in the total accessible porosity or cause closure of the accessible pore structure. However, it produced a significant decrease in the detectable microcracks. The total length, density and number of detectable microcracks decreased by about 40%, 45% and 23% respectively when confining pressure was increased from 0.6MPa to 1.9MPa.

Fig. 8 (a & b) shows typical examples of the epoxy intrusion through the cross-section of the same sample, but subjected to different confining pressures. Fig. 8 (c) shows an image of a microcrack that can be seen in the boxed area in Fig. 8(a) that was captured at a much higher resolution (pixel size = 0.89 µm) using fluorescence microscopy. The results show that confining pressure has a significant influence on the pattern of the epoxy intrusion. Since the drying-induced microcracks are nearly perpendicular to the exposed surface (Fig. 8c & Table 7), the applied confinement would cause closure of the microcracks. It appears that at a relatively low confining pressure of 0.6MPa,
the microcracks remain open and provide continuous flow channels for the epoxy. Hence, the epoxy predominantly intruded through microcracks (Fig. 8a). When the confining pressure was increased to 1.9MPa, partial closure of the microcracks occurred as evident in the image analysis results shown in Table 8. Subsequently, a larger fraction of the epoxy intruded the sample via the pore structure.

4. Discussion

The results show that transport properties of paste or concrete samples decreased with increase in confining pressure, and the decrease was more significant in samples that had a greater degree of microcracking. It is important to note that the observed trend is not due to leakage since blank tests have been carried out at the lowest confining pressure and found that the seal was indeed effective (Table 4). The results from microscopy and image analysis show that the decrease in transport properties was due to closure of the microcracks when the sample was compressed. Since most of the microcracks are nearly perpendicular to the exposed surface and propagate into the sample, their closure is expected to have a substantial impact on transport properties.

However, the effect of confining pressure was more significant for permeability than diffusivity. This strongly indicates that permeability is more sensitive to microcracks compared to diffusivity. Theoretically, flow through a cracked media driven by pressure gradient scales to the cube of the crack width, whereas diffusion is proportional to the total accessible porosity [Gérard & Marchand, 2000]. This finding is consistent with those from other studies [e.g. Wong et al., 2007 & 2009] that examined the relative influence of microcracks on diffusivity and permeability. The observation that diffusivity was not significantly affected is also important because it shows that the applied confinement and the closure of microcracks did not produce a significant decrease in the total accessible porosity. This is supported by the finding that the total epoxy intrusion remained approximately the same at different confining pressures (Table 8). Thus, the observed decrease in permeability is mainly caused by the closure of microcracks rather than a reduction in accessible porosity.

The data in Figure 6 and Table 8 suggest that crack closure and decrease in permeability will continue if confinement was increased beyond 2MPa, in particular for samples containing high levels of microcracking. The data in Table 6 show that when compressed, the permeability of highly micro-cracked samples (105°C dried) approaches that of samples with low levels of microcracking (stepwise dried). However, it would be unreasonable to expect the values to become equivalent at higher confining pressures because the measured mass transport properties are also hugely influenced by the sample moisture content. Clearly, the stepwise dried samples would have a higher degree of moisture content compared to those dried at 105°C, which in turn determines the amount accessible porosity. In addition, microcracks may not be able to close completely if there are blockages (caused by loose aggregate or debris, for example), or if there are relative movements causing misalignment between the two sides of the crack wall.

Very few studies have systematically investigated the effect of confining pressure on microcracks and transport properties of concrete. Most of the available studies have been carried out at confining pressures that are much higher than the values used in the present study. However, their findings seem consistent with our study. For example, Mills [1987] showed that both water and gas permeability of concrete (w/c ratios: 0.42, 0.56, 0.64 & 0.77, 35 days sealed curing) decreased by 0 to 77% for water permeability and 19 to 50% for gas permeability as the lateral confining pressure increased from 5MPa to 25MPa. However, no explanation for this effect was provided. Lion et al. [2005] found that apparent gas permeability of mortars (0.5 w/c ratio, 58% vol. sand, 2-year water cured) that were dried at 60°C, 105°C and 205°C decreased by 27%, 46% and 41% respectively when confining pressure was increased from 4MPa to 28MPa. The authors attributed this to closure of microcracks, but no direct evidence of this was presented.
Chen et al. [2013] tested the gas permeability of heat-treated mortars (0.5 w/c ratio, 58% vol. sand, 6 months water cured) at several confining stresses ranging from 5MPa to 25MPa. Interestingly, they observed that the permeability of mortars dried at 60°C and 105°C was relatively insensitive to confinement pressure, which seems to be at odds with the findings of Lion et al. [2005] who tested a similar mortar using a similar test set up. However, Chen et al. [2013] showed that for mortars heat-treated at 200°C to 400°C, gas permeability decreased by up to 23% when the confining stress increased from 5 to 25MPa. They deduced that this was caused by closure of the heat induced microcracks rather than the closure of porosity. In another study, Banthia & Bhargava [2007] showed that the water permeability of plain concrete and fibre reinforced concrete with 0.1%, 0.3%, 0.5% fibre volume fraction (0.6 w/c ratio, 7 days lime-saturated water cured) decreased by up to 38% (plain concrete), 43% (0.1% fibre), 47% (0.3% fibre) and 67% (0.5% fibre) when confining stress was increased to 30% of the ultimate compressive strength. They hypothesised that the decrease in water permeability was due to pore compression, but did not carry out measurements to verify this.

The closure of microcracks at low confinement stress levels has been investigated experimentally and numerically in rock materials [e.g. Batzle et al., 1980; Li & Nordlund, 1993; Liu et al., 2001]. For example, Batzle et al. [1980] made direct observations of microcrack closure in Westerly granite and Frederick diabase samples under uniaxial compressive stress via a scanning electron microscope. The microcracks had widths less than 10 μm and were either inherent in the sample or thermally induced (at 500°C or 700°C). The authors observed that some microcracks may undergo complete closure at low confining stress of 1MPa, while others remain open even at stresses above 10MPa. They explained that the mechanics of microcrack closure is complicated and depends on factors such as shape and surface roughness of the cracks, how well the crack walls match, and crack orientation with respect to applied stress. Some cracks may be propped open until the material lodged inside was crushed. Furthermore, local stress concentrations may produce new fracturing and shearing motion of the crack walls, which is another source of complication.

Implications

Confining pressure is rarely specified or measured during transport testing of cement-based materials. Where such information is available in the literature, we found that confining pressures ranging from 0.7 to 5.4MPa have been used [RILEM TC 116-PCD; Chen et al., 2009; Perlot et al., 2013]. However, our results show that the confining pressure used in transport testing is an important parameter that could influence measured results. This is particularly significant for gas permeability testing and the effect is evident even at relatively low confining pressures of 0.3 to 1.9MPa. Hence, this must be taken into consideration in interpreting results.

Because microcracks can close when samples are confined, testing the transport properties of concrete that inherently contain cracks is not a straightforward procedure. Testing at very low confinement pressure increases the risk of leakage and may not be representative of the behaviour of concrete in real structures subjected to relatively high levels of compressive stress. However, transport testing in a highly stressed state is not easy to achieve experimentally and could also cause additional damage.

It should be noted that most concrete structures in service are subjected to compressive stresses that are much higher than the largest confining pressure tested in this work. Our results show that drying-induced microcracks would not play a major role in transport of aggressive species in structural elements that are subjected to compressive stresses. These include typical columns and walls which are under the action of axial compression. In concrete elements subjected to tensile stresses however, the drying-induced microcracks are likely to widen and propagate, and potentially accelerate the transport of aggressive species. These include typical beams and slabs subjected to
bending action that result in compressive and tensile stresses acting on different parts of the structure. For example, drying-induced microcracks are likely to widen and propagate in the region below the neutral axis of a simply-supported beam or slab that is under tension. Further work will be necessary to better understand the influence of microcracks on the transport properties of concrete structures under load.

5. Conclusions
The aim of the research was to determine if a relatively low confining pressure would influence results, and to establish if measuring transport at increasing confining pressure can be used as a means to isolate and study the influence of microcracks on transport properties. Results show that permeability decreased significantly (by up to 46%) with increase in confining pressure (up to 1.9MPa), and this effect is more pronounced for samples with a higher degree of microcracking (105°C dried). The largest applied confining pressure was only 4-8% of the sample 3-day compressive strength. However, diffusivity was relatively insensitive to a change in confining pressure. Fluorescence microscopy and image analysis show that drying-induced microcracks have widths of 1 to 60 μm. It is also observed that the applied confining pressure causes the microcracks to undergo partial closure, but this did not produce a significant change in total accessible porosity. The study concludes that the confining pressure used in transport testing is an important parameter that could influence results. A low confining pressure increases the risk of leakage, while higher confining pressure may cause changes to the microstructure and subsequent damage at extreme values. However, testing transport properties of concrete in an unstressed state does not reflect the actual behaviour of structures in service. Therefore, the confining pressure used must be taken into consideration in interpreting transport results. Measuring transport property repeatedly on the same sample at increasing confining pressure is a promising approach to isolate the influence of microcracks from other factors such as porosity and moisture content. The results show that drying-induced microcracks have a significant effect on gas permeability, but much less impact on diffusivity.

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REFERENCES


Table captions:

Table 1 Oxide composition, ignition loss, fineness and specific gravity of CEM I
Table 2 Aggregate properties
Table 3 Mix proportions and curing age of samples
Table 4 Measured gas leakage through a steel disc to determine the smallest load required to seal the sample during transport testing
Table 5 Measured compressive strength and relative density of paste and concrete samples. Standard errors are shown in brackets
Table 6 Mass transport results. Standard errors are shown in brackets
Table 7 Effect of drying regime on the characteristics of microcracks in C0.5-68-3d & C0.5-60-28d concrete. Values in brackets are standard errors.
Table 8 Results from image analysis showing the effect of confining pressure on the epoxy intrusion area, epoxy intrusion depth and the number, total length and density of detectable microcracks. Sample P 0.5-3d.
Figure captions:

Figure 1 Particle size distribution of fine and coarse aggregate.

Figure 2 Test setup for measuring the influence of confining pressure on permeation and diffusion of gas through concrete.

Figure 3 Change in colour intensity of the pressure-sensitive film corresponding to applied compressive load and confining pressure.

Figure 4 Effect of w/c ratio and drying regime on a) oxygen diffusivity, and b) oxygen permeability of 3-day cured pastes and concretes measured at confining pressure of 0.3MPa

Figure 5 Klinkenberg constant ($\beta$) plotted against oxygen permeability ($k_{int}$)

Figure 6 Effect of confining pressure on the diffusivity and permeability of pastes and concretes

Figure 7 Influence of drying regime on the frequency distribution of microcrack width (a) and length (b) for C-0.5-60-28d

Figure 8 Typical epoxy intrusion patterns observed on the sample cross-section at confining pressures of (a) 0.6MPa and (b) 1.9MPa. Fig. 8(c) is a montage of a microcrack seen in the boxed area in (a), obtained by stitching images captured using fluorescence microscopy. Sample is P 0.5-3d dried at 105°C.
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(a) Diffusivity of pastes

(b) Permeability of pastes
Figure 6 Effect of confining pressure on the diffusivity and permeability of pastes and concretes

(c) Diffusivity of concrete  
(d) Permeability of concrete

Figure 7 Influence of drying regime on the frequency distribution of microcrack width (a) and length (b) for C-0.5-60-28d

(a)  
(b)
Figure 8 Typical epoxy intrusion patterns observed on the sample cross-section at confining pressures of (a) 0.6MPa and (b) 1.9MPa. Fig. 8(c) is a montage of a microcrack seen in the boxed area in (a), obtained by stitching images captured using fluorescence microscopy. Sample is P 0.5-3d dried at 105°C.
### Table 1 Oxide composition, ignition loss, fineness and specific gravity of CEM I

<table>
<thead>
<tr>
<th>Oxide composition (%)</th>
<th>Ignition loss (%)</th>
<th>Fineness (cm²/g)</th>
<th>Specific gravity</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO 63.4</td>
<td>SiO₂ 20.6</td>
<td>Al₂O₃ 5.6</td>
<td>Fe₂O₃ 2.4</td>
</tr>
</tbody>
</table>

### Table 2 Aggregate properties

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Max size (mm)</th>
<th>Specific gravity</th>
<th>24-hour absorption (%)</th>
<th>Moisture content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sand</td>
<td>5</td>
<td>2.54</td>
<td>0.52</td>
<td>0.21</td>
</tr>
<tr>
<td>Limestone</td>
<td>10</td>
<td>2.71</td>
<td>0.88</td>
<td>0.46</td>
</tr>
</tbody>
</table>

### Table 3 Mix proportions and curing age of samples

<table>
<thead>
<tr>
<th>Mix ID</th>
<th>Cement: kg/m³</th>
<th>Total water: kg/m³</th>
<th>Free w/c</th>
<th>MSA: mm</th>
<th>Sand: kg/m³</th>
<th>Limestone: kg/m³</th>
<th>Aggregate vol. (%):</th>
<th>Curing age: days</th>
</tr>
</thead>
<tbody>
<tr>
<td>P 0.35-3d</td>
<td>1462.8</td>
<td>512.0</td>
<td>0.35</td>
<td>----</td>
<td>----</td>
<td>----</td>
<td>----</td>
<td>3</td>
</tr>
<tr>
<td>P 0.5-3d</td>
<td>1197.4</td>
<td>598.7</td>
<td>0.50</td>
<td>----</td>
<td>----</td>
<td>----</td>
<td>----</td>
<td>3</td>
</tr>
<tr>
<td>C 0.35-68-3d</td>
<td>458.0</td>
<td>160.3</td>
<td>0.35</td>
<td>10</td>
<td>718.2</td>
<td>1077.3</td>
<td>68</td>
<td>3</td>
</tr>
<tr>
<td>C 0.5-68-3d</td>
<td>374.9</td>
<td>194.2</td>
<td>0.50</td>
<td>10</td>
<td>718.2</td>
<td>1077.3</td>
<td>68</td>
<td>3</td>
</tr>
<tr>
<td>C 0.5-60-28d</td>
<td>471.7</td>
<td>235.8</td>
<td>0.50</td>
<td>10</td>
<td>633.7</td>
<td>951</td>
<td>60</td>
<td>28</td>
</tr>
</tbody>
</table>

### Table 4 Measured gas leakage through a steel disc to determine the smallest load required to seal the sample during transport testing

<table>
<thead>
<tr>
<th>Gas pressure (MPa)</th>
<th>Compressive load (kN)</th>
<th>Average gas leakage (cm³/s)</th>
<th>Standard error</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.25</td>
<td>7</td>
<td>7.37</td>
<td>0.0346</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>0.01</td>
<td>0.0001</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

### Table 5 Measured compressive strength and relative density of paste and concrete samples. Standard errors are shown in brackets

<table>
<thead>
<tr>
<th>Sample</th>
<th>Free w/c</th>
<th>Relative density</th>
<th>Compressive strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P 0.35-3d</td>
<td>0.35</td>
<td>2.06 (0.003)</td>
<td>49.0 (0.58)</td>
</tr>
<tr>
<td>P 0.5-3d</td>
<td>0.50</td>
<td>1.95 (0.002)</td>
<td>23.5 (0.26)</td>
</tr>
<tr>
<td>C 0.35-68-3d</td>
<td>0.35</td>
<td>2.45 (0.002)</td>
<td>46.2 (0.88)</td>
</tr>
<tr>
<td>C 0.50-68-3d</td>
<td>0.50</td>
<td>2.43 (0.003)</td>
<td>29.3 (0.28)</td>
</tr>
</tbody>
</table>
Table 6 Mass transport results. Standard errors are shown in brackets

\textit{a) Oxygen diffusivity (× 10⁻⁸ m²/s)}

<table>
<thead>
<tr>
<th>Sample</th>
<th>Conditioning regime</th>
<th>Confining pressure (MPa)</th>
<th>0.3</th>
<th>0.6</th>
<th>1.2</th>
<th>1.9</th>
</tr>
</thead>
<tbody>
<tr>
<td>P 0.35-3d</td>
<td>21°C (stepwise)</td>
<td></td>
<td>5.8 (0.35)</td>
<td>5.8 (0.35)</td>
<td>5.7 (0.33)</td>
<td>5.7 (0.31)</td>
</tr>
<tr>
<td></td>
<td>105°C</td>
<td></td>
<td>42.3 (0.20)</td>
<td>41.8 (0.32)</td>
<td>40.8 (0.45)</td>
<td>40.6 (0.50)</td>
</tr>
<tr>
<td>P 0.5-3d</td>
<td>21°C (stepwise)</td>
<td></td>
<td>8.2 (1.72)</td>
<td>8.1 (1.73)</td>
<td>8.1 (1.71)</td>
<td>8.3 (1.72)</td>
</tr>
<tr>
<td></td>
<td>105°C</td>
<td></td>
<td>63.6 (0.74)</td>
<td>63.5 (0.71)</td>
<td>62.8 (0.90)</td>
<td>62.0 (0.73)</td>
</tr>
<tr>
<td>C 0.35-68-3d</td>
<td>21°C (stepwise)</td>
<td></td>
<td>3.8 (0.44)</td>
<td>3.8 (0.43)</td>
<td>3.7 (0.41)</td>
<td>3.7 (0.41)</td>
</tr>
<tr>
<td></td>
<td>105°C</td>
<td></td>
<td>12.5 (0.16)</td>
<td>12.4 (0.12)</td>
<td>12.2 (0.13)</td>
<td>12.2 (0.16)</td>
</tr>
<tr>
<td>C 0.5-68-3d</td>
<td>21°C (stepwise)</td>
<td></td>
<td>5.5 (0.11)</td>
<td>5.5 (0.08)</td>
<td>5.6 (0.09)</td>
<td>5.6 (0.04)</td>
</tr>
<tr>
<td></td>
<td>105°C</td>
<td></td>
<td>19.0 (0.04)</td>
<td>18.8 (0.12)</td>
<td>18.7 (0.13)</td>
<td>18.6 (0.10)</td>
</tr>
<tr>
<td></td>
<td>105°C*</td>
<td></td>
<td>16.9 (0.14)</td>
<td>16.7 (0.39)</td>
<td>16.3 (0.07)</td>
<td>16.2 (0.02)</td>
</tr>
<tr>
<td>C 0.5-60-28d</td>
<td>21°C (stepwise)</td>
<td></td>
<td>8.5 (0.47)</td>
<td>8.5 (0.48)</td>
<td>8.3 (0.44)</td>
<td>8.3 (0.47)</td>
</tr>
<tr>
<td></td>
<td>105°C</td>
<td></td>
<td>16.7 (2.23)</td>
<td>16.8 (2.28)</td>
<td>16.8 (2.08)</td>
<td></td>
</tr>
</tbody>
</table>

\textit{b) Oxygen permeability (× 10⁻¹⁷ m²)}

<table>
<thead>
<tr>
<th>Sample</th>
<th>Conditioning regime</th>
<th>Confining pressure (MPa)</th>
<th>0.3</th>
<th>0.6</th>
<th>1.2</th>
<th>1.9</th>
</tr>
</thead>
<tbody>
<tr>
<td>P 0.35-3d</td>
<td>21°C (stepwise)</td>
<td></td>
<td>3.4 (0.34)</td>
<td>3.3 (0.35)</td>
<td>3.2 (0.30)</td>
<td>2.9 (0.15)</td>
</tr>
<tr>
<td></td>
<td>105°C</td>
<td></td>
<td>53.4 (3.41)</td>
<td>41.9 (2.65)</td>
<td>32.4 (0.76)</td>
<td>29.1 (0.63)</td>
</tr>
<tr>
<td>P 0.5-3d</td>
<td>21°C (stepwise)</td>
<td></td>
<td>2.8 (0.66)</td>
<td>2.5 (0.53)</td>
<td>2.4 (0.51)</td>
<td>2.2 (0.55)</td>
</tr>
<tr>
<td></td>
<td>105°C</td>
<td></td>
<td>71.9 (2.08)</td>
<td>55.9 (3.60)</td>
<td>48.3 (3.88)</td>
<td>44.4 (2.79)</td>
</tr>
<tr>
<td>C 0.35-68-3d</td>
<td>21°C (stepwise)</td>
<td></td>
<td>2.5 (0.11)</td>
<td>2.5 (0.10)</td>
<td>2.4 (0.10)</td>
<td>2.3 (0.07)</td>
</tr>
<tr>
<td></td>
<td>105°C</td>
<td></td>
<td>7.5 (0.38)</td>
<td>6.8 (0.08)</td>
<td>6.5 (0.11)</td>
<td>6.3 (0.08)</td>
</tr>
<tr>
<td>C 0.5-68-3d</td>
<td>21°C (stepwise)</td>
<td></td>
<td>10.2 (0.09)</td>
<td>10.0 (0.26)</td>
<td>9.9 (0.53)</td>
<td>9.6 (0.36)</td>
</tr>
<tr>
<td></td>
<td>105°C</td>
<td></td>
<td>30.6 (0.77)</td>
<td>28.2 (0.27)</td>
<td>27.5 (0.65)</td>
<td>26.4 (0.34)</td>
</tr>
<tr>
<td></td>
<td>105°C*</td>
<td></td>
<td>29.7 (0.00)</td>
<td>24.9 (0.16)</td>
<td>24.1 (0.08)</td>
<td>23.3 (0.23)</td>
</tr>
<tr>
<td>C 0.5-60-28d</td>
<td>21°C (stepwise)</td>
<td></td>
<td>21.7 (8.15)</td>
<td>20.3 (7.77)</td>
<td>19.0 (6.82)</td>
<td>18.7 (6.14)</td>
</tr>
<tr>
<td></td>
<td>105°C</td>
<td></td>
<td>50.5 (8.05)</td>
<td>40.9 (3.64)</td>
<td>37.3 (1.25)</td>
<td></td>
</tr>
</tbody>
</table>

Table 7 Effect of drying regime on the characteristics of microcracks in C0.5-68-3d & C0.5-60-28d concrete. Values in brackets are standard errors.

<table>
<thead>
<tr>
<th>Sample / Drying regime</th>
<th>Average crack width: µm</th>
<th>Average crack density: mm⁻¹</th>
<th>Orientation of crack with respect to drying surface °</th>
</tr>
</thead>
<tbody>
<tr>
<td>C0.5-68-3d</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Stepwise</td>
<td>4.2 (0.16)</td>
<td>0.05 (0.002)</td>
<td>86.1 (2.7)</td>
</tr>
<tr>
<td>105°C</td>
<td>8.8 (0.10)</td>
<td>0.21 (0.032)</td>
<td>88.5 (2.2)</td>
</tr>
<tr>
<td>C0.5-60-28d</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Stepwise</td>
<td>5.7 (0.5)</td>
<td>0.05 (0.011)</td>
<td>89.4 (4.7)</td>
</tr>
<tr>
<td>105°C</td>
<td>8.6 (1.2)</td>
<td>0.11 (0.006)</td>
<td>90.9 (2.8)</td>
</tr>
</tbody>
</table>
Table 8 Results from image analysis showing the effect of confining pressure on the epoxy intrusion area, epoxy intrusion depth and the number, total length and density of detectable microcracks. Sample P 0.5-3d.

<table>
<thead>
<tr>
<th>Confining pressure (MPa)</th>
<th>Average intrusion area (%)</th>
<th>Average intrusion depth (mm)</th>
<th>Number of microcracks</th>
<th>Total length of microcracks (mm)</th>
<th>Density of microcracks (mm⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.6</td>
<td>2.9 (0.1)</td>
<td>1.4 (0.03)</td>
<td>52</td>
<td>179.7</td>
<td>0.49</td>
</tr>
<tr>
<td>1.9</td>
<td>3.2 (0.3)</td>
<td>1.5 (0.16)</td>
<td>40</td>
<td>108.8</td>
<td>0.27</td>
</tr>
</tbody>
</table>