

# Macro/micro-pore structure characteristics and the chloride penetration of self-compacting concrete incorporating different types of filler and mineral admixture



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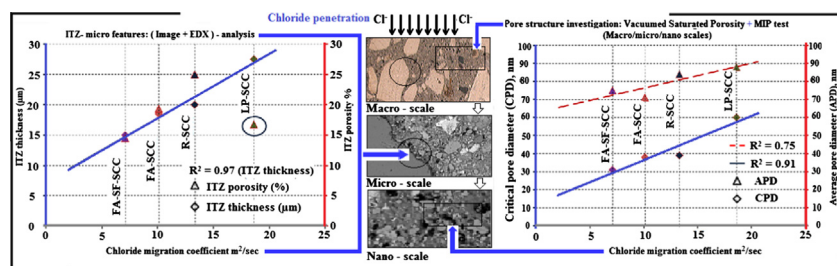
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## HIGHLIGHTS

- Study of chloride ingress and microstructure features of sustainable SCC.
- Modification of rapid chloride migration test is achieved.
- A chloride ingress–pore structure relationship is proposed for the first time.
- Pore percolation is primarily responsible for chloride ingress in sustainable SCC.
- Nano features of the pores are dominant in controlling chloride ingress in SCC.

## GRAPHICAL ABSTRACT



## ARTICLE INFO

### Article history:

Received 13 May 2014

Received in revised form 18 August 2014

Accepted 24 August 2014

### Keywords:

Chloride penetration  
Rapid migration test  
Self-compacting concrete  
Microstructure  
Interfacial transition zone  
Cement replacement  
Pores percolation

## ABSTRACT

The relationship between the internal pore structure features at different scales and the local micro-characteristics of the interfacial transition zone (ITZ) to the non-steady state chloride migration coefficient ( $D_{nssm}$ ) is investigated for one normal and three types of sustainable high performance self-compacting concrete mixes. The pore structure classification at different scales and the percolation degrees of the ITZ's pores were determined using both vacuum-saturated and Mercury Intrusion Porosimetry (MIP) techniques. Further, the local micro-permeation features of the ITZ, such as thickness, porosity and the chemistry of its hydration products is examined using the SEM coupled with the EDX analysis on polished, carbon-coated, flat specimens. Chloride movement was achieved using a modified rapid migration test.

It was deduced that the degree of percolation of the pores of the ITZ had a significant role in controlling the chloride penetration process. Further, it is proposed that the ITZ thickness might be, primarily, responsible in determining the chloride ions' migration velocity especially when coarse and unreactive filler are used. At nano scale, it is also suggested that the critical pore diameter in the cement matrix is more significant than is the average pore diameter in controlling the chloride resistance in SCC.

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## 1. Introduction

As reported by many investigations, the chloride ion penetration from an external source (sea, underground and de-icing water) is considered as one of the main causes of the initiation of steel reinforcement corrosion which then leads to reduction in the

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serviceability life of the affected concrete structure. This topic has become an increasingly important area in the study of concrete durability since the middle of the last century [1]. With the widespread use of SCC, which is relatively a new type of concrete, in different concrete structures exposed to severe chloride environments such as bridges, culverts, tunnels, tanks, dams, and precast concrete products, much information is needed to assess the chloride ingress and its relationship to the concrete's internal microstructure at macro, micro and even at nano-scales.

Since concrete/mortar is considered as a porous composite material with three different phases (cement matrix, aggregate and ITZ between them), the chloride ions can penetrate the concrete through the continuous pores of each phase. The aggregate phase (fine/coarse) has less effect on the chloride penetration due to the lower diffusion coefficient in comparison with cement matrix [2]. However, from a mix design point of view, the existence of high amounts of aggregate (high volume fraction) could have two, conflicting, effects on the transport properties of the concrete including the chloride movement. On the one hand, it leads to more numerous ITZs, and more volume of this region, which may assist the chloride ion movement due to the high porosity in comparison to the background matrix porosity. On the other hand, it increases the tortuosity of penetration paths through the matrix [3].

It is known that the SCC has a dense microstructure and a dense cement matrix, containing reactive and non-reactive fillers, when compared to normal vibrated concrete (NVC) [4,5]. Sometimes SCC has the same or higher water to cementitious material ratio as NVC in which case, the fillers and added workability due to the high dosage of SP may be responsible for the greater density. Sometimes SCC has lower water to cementitious material ratio, which directly contribute to the greater density and is facilitated by the SP-induced workability. Thus, the SCC could have a less easily penetrated microstructure. However, as reported by Assié et al. [6], referencing Zhu et al. [7], the modification of the microstructure might not be enough to assure sufficient resistance to the chloride penetration as this property might be governed by the tortuosity, the percolation and the connectivity of the internal pore network.

Although research work has been done to estimate the chloride penetration resistivity as part of the durability assessment of SCC, the available experimental data about the SCC showed that no definite conclusion could be drawn about whether SCC has similar, larger or smaller resistance to chloride penetration than NVC at the same strength level. Assié et al. [6] claimed that a SCC having a similar or, even better, compressive strength than a NVC, although at a higher w/c ratio, had equivalent chloride diffusion and water absorption. In their study, the SCC investigated employed limestone filler for all mixes. On the other hand, the results of Audenaert et al. [8] revealed that no definite conclusion was possible about whether SCC has a larger or smaller chloride migration coefficient in comparison with NVC. Recently, Dinakar et al. [9] noticed that, in spite of higher permeable voids and higher water absorption, high fly ash SCC showed lower chloride penetrability in comparison with NVC at any strength grade. The authors suggested that it could be as a result of a higher chloride binding capacity of the cement matrix caused by the presence of high available  $C_3A$  in the cementitious materials system with increased fly ash content.

Understanding the relationship between the microstructure of the concrete as it relates to degradation, especially chloride penetration and carbonation, still presents a great challenge for the concrete technologist. This is due to both the complexity of the chemistry and the microstructure of SCC, in particular the different macro/micro/nano scale characteristics and the complexities of these two physico-chemical phenomenon as well. For SCC, questions have been raised as to which has the dominant role: the micro permeation characteristics of the ITZ or the pore characteristics of the bulk cement matrix?

To attempt an answer to this question, Leemann et al. [10] studied the effect of using different types of cement on the porosity of the ITZ and its relationship to chloride resistance, using the rapid chloride migration test according to the Swiss standard SIA 262/1. In this study, the authors concluded that the change of the internal pore structure of the cement matrix, as caused by the use of different types of cement in SCC mixes, had a stronger effect on the non-steady state chloride migration coefficients obtained than did the pore volume in the ITZ. However, the long curing time (62 days) used in this investigation might reduce any controlling effect of the local microstructure characteristics of the ITZs. Moreover, Leemann et al. made no attempt to address how the change of the internal pore structure, including the tortuosity effect of the aggregate and the percolation of pores in the ITZ, nor the nature of the pore system in the matrices, could affect the chloride resistance of SCC as only one technique was used (Image analysis) for characterizing the ITZ pores microstructure. This cannot give enough information about the effect of the internal capillary pores and their percolation to fully defend their conclusion. While Leemann et al. results were based on the use of different cement types to examine the chloride resistivity of SCC, the current research work is designed to examine the effects of the internal microstructure and its role in determining the chloride ingress process in normal and sustainable high performance SCC.

In previous research work [11,12] the authors have attempted to develop a further understanding of the relationship between carbonation and the microstructural properties of sustainable SCC via accelerated tests. In the present paper it is aimed to quantitatively analyze the correlation between these characteristics, in terms of the internal pore structure and the local micro-permeation of the ITZ properties as consequence of a change of filler and mineral admixture type at high cement replacement percentages, to the chloride penetration velocity.

To achieve this, a non-steady state accelerated test was modified from the recommendations of NT Build 492 – Nordtest method [13] and used in the present study. The modification, as described in Section 3.3, was mainly performed in order to reduce the time of the test to, as much as possible, avoid the change of the ionic composition of the pore water solution (pH value) due to the migration of the  $OH^-$  ions and hence, limiting the local chloride binding ability due to the difference in reactivity levels of the fillers and the mineral admixtures used through the relatively long time of the standard test.

The main issues addressed in this paper are:

- (i) Effect of using different types of filler and mineral admixtures, at relatively high rates of cement replacement, on the internal pore structure and the local micro-permeation characteristics of ITZ of different types of SCC.
- (ii) Determination of the non-steady state chloride migration coefficient ( $D_{nssm}$ ) for SCCs with different binder types using a modified rapid migration test.
- (iii) Macro/micro and nano internal pore structure property relationships with the  $D_{nssm}$ .

These issues have been studied in order to provide further understanding of the microstructure of the sustainable SCC and the part it plays in determining the resistance to chloride penetration.

## 2. Experimental program

### 2.1. Materials

Ordinary Portland cement CEM I, 52.5 R conforming to EN 197-1 was used to produce all the SCC and mortars. Natural limestone filler (LP) from Longcliffe quarry (Derbyshire, UK), fly ash (FA) class F conforming to BS EN 450-1 produced by the

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