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Microstructure, thermal analysis and chloride penetration of self-compacting concrete under different conditions

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This paper concerns the characterisation of the raw materials used in four formulations of self-compacting concrete (SCC) and the study of their fresh state, compressive strength, thermal analysis, transport properties and chloride penetration under immersion and tidal conditions. The control concrete corresponds to a concrete with class 40 MPa based on Portland cement and limestone filler. A second formulation involves the complete substitution of limestone filler by sand filler. In the two other formulations, 10% and 15% of metakaolin are substituted for cement. The results show that SCC with metakaolin exhibits higher resistance to chloride penetration at different states. A higher strength development is obtained when using sand filler. This is true up to 90 d of maturation; beyond this, the compressive strength increases for the control SCC. However, mixtures with metakaolin present compressive strength values close to or higher than the ones of the control mix at both early- and long-term ages. This research shows that the substitution of Portland cement by 15% metakaolin in SCC is affordable in all aspects.

Notation

C_{Ag}	concentration of silver nitrate
D_{nSSm}	migration coefficient ($\times$10–12 m^2/s)
$\mathrm{D_{ns}}$ (dif)	apparent diffusion coefficient of chloride ions in the saturated condition (m^2/s)
L	thickness of the sample (mm)
M_{pe}	sampled weight of powder product
S	sorptivity
T	anolyte solution temperature (°C)
t	duration of the test (h)
U	applied voltage (V)
V_{e}	volume of silver nitrate
V_{fiol}	volume of the filtrate solution
V_{p}	volume of the solution
W	water content
W_{e}	evaporable water content
W_{ne}	non-evaporable water content
X_{d}	depth of penetration of chloride ions (mm)

Introduction

Globally, concrete is one of the most commonly used materials worldwide after water. In structures made of concrete, in order to obtain the necessary strength and reduce the porosity as well as the air inside the concrete and then ensure reliability, the concrete is vibrated in different ways. Most of the time, the vibration effect is not accurate and the mechanical properties of the concrete are not satisfactory. With the development of concrete structures and the prefabricated industry in general, coinciding with the lack of skilled workers, the notion of producing self-compacting concrete (SCC) was presented for the first time by Okamura and Ouchi (2003) in Japan. SCC can move under its own weight. In addition, its consolidation does not require vibration during concreting. SCC, with its specific characteristics, can increase durability and stability in a reinforced concrete structure. A durable concrete must retain its original form, quality and facility of realisation under environmental working conditions. Since the movement of aggressive media fluids from the environment in concrete is the main cause for most deterioration in concrete, the lifetime of reinforced concrete elements and permeability are the two most influential factors in sustainability. To better understand the lifetime of the mechanical and environmental stresses of materials, it is necessary to have sufficient knowledge of the properties of materials, in particular their transport properties. In this respect, the various transport processes of harmful substances through concrete have been distinguished as diffusion, absorption and penetration as a function of the driving force of the process and the nature of the material being transported. The contribution of mineral additions to the activity of the cement binder essentially results from these effects: physico-chemical, microstructural and chemical (Bessa-Badreddine, 2004). According to Bessa-Badreddine (2004), the chemical effect allows an improvement in the quality of the bonds in the cementitious matrix with decreasing hydrate volume. In general, the compressive strength increases by the use of pozzolanic additions (Ahmadi *et al.*, 2014; Najimi *et al.*, 2012; Sadrmomtazi *et al.*, 2017), because pozzolan reacts with portlandite from the cement hydration reaction and creates a new hydrated product that could be very useful in increasing the compressive strength. Thereby, the presence of a large number of submicron particles in the cement matrix around the cement grains multiplies the possibilities of germination of the hydrated products and develops the microstructural complexity and the efficiency of the bonds (Bessa-Badreddine, 2004). The binding contribution of the mineral additions thus has a structural role in the connections of the cementitious matrix from the mechanical point of view, which suggests a quantitative role in the reduction of the porosity. In terms of durability, this structural contribution translates to better resistance in the face of severe chemical aggression. The use of fillers in a concrete formulation generates an acceleration of its mechanical resistance at young ages (De Larrard, 1999; Pera *et al.*, 1999). The fine particles of the filler, when well deflocculated by a superplasticiser, promote the hydration of the cement, mainly by a physical effect, and lead to a denser cementitious matrix. These effects have a significant influence on mechanical strength up to 28 d but subsequently become less significant (Assié, 2004). In addition, the fillers may be inert, active or even pozzolanic.

Pozzolans enhance concrete performance by reacting with carbon (C) and hydrogen (H) to form the secondary calcium–silicate–hydrate (C–S–H) gel. Alongside other cementitious materials, such as silica fume, ground-granulated blast-furnace slag and pulverised fly ash, which are frequently used for concrete production, metakaolin is a material that is being used more frequently in the concrete industry. Pure metakaolin has been used successfully as a cementitious additive in concrete since 1990 (Hassan *et al.*, 2012). When the kaolin is heated to 700–900°C, it is calcined and loses up to 14% of hydroxyl water, changing to metakaolin (Caldarone *et al.*, 1994). Metakaolin reacts with the calcium hydroxide formed during the hydration of Portland cement and creates additional cementitious products, modifying the concrete structure via improved mechanical performance as well as overall durability (Hubertova and Hela, 2007).

Indeed, metakaolin improves the behaviour of concrete via two effects:

- A filler effect: the fine particles of metakaolin will be inserted into the empty interstitial spaces between the grains of cement. This modifies the properties in the fresh state and makes it possible to obtain a more compact composition.
- Acceleration of hydration: for the same duration of hydration, a greater quantity of hydrates is formed. Then the pores are gradually filled.

It is well known that metakaolin improves concrete properties, such as compressive strength and resistance to chloride penetration. The application of metakaolin is known to considerably refine the pore structures of concrete and decrease the calcium hydroxide (Dubey and Banthia, 1998). Kim *et al.* (2007) show that the resistance to chloride ion penetration reduces significantly as the proportion of silica fume and metakaolin binders is increased. The results of Kannan and Ganesan (2014) show replacement with 30% metakaolin leads to a 26% improvement in compressive strength, a 98·10% decrement in total charge passed in an RCPT test and a 38·76% decrement in water permeability.

Currently, very little information is available on the properties of SCC containing metakaolin and sand filler, and there is no extensive sustainability data regarding the use of metakaolin and sand filler in SCC. Therefore, the use of SCC containing metakaolin and sand filler requires further study. The studied transport properties are mainly obtained using chloride migration, water penetration, capillary absorption and electrical resistivity tests. However, one can observe a lack in the literature regarding the influence of sand filler on transport properties in SCC and also on the characterisation of the microstructure and the thermal analysis of metakaolin, as well as on their performance in non-saturated conditions such as in the tidal zone in marine environments. This paper investigates and compares the effect of a 10% and 15% metakaolin substitution of Portland cement, and of the total replacement of limestone filler by sand filler, on the rheological behaviour, compressive strength and transport properties of SCC at early ages and up to 180 d. The impact on the resistance to chloride penetration both in immersion and tidal conditions is also investigated. For this purpose, first the chemical and mineralogical composition as well as the microstructure of the cementitious material were studied. Then different tests were performed to study the fresh phase of SCC, and mechanical testing was subsequently done on hardened states to evaluate the compressive strength of the different prepared mixtures. The tests concerning the transport phenomena were also performed at different ages in order to evaluate the durability properties over time. Finally, the resistance to chloride penetration of the different mixtures was evaluated in immersion and tidal conditions.

Experimental programme

Materials and mix proportion

A constant Portland cement type II content (350 kg/m^3), a constant W/C_m ratio of 0·4 and constant gravel to sand ratio of $G/S=1$ were used for the preparation of four concrete mixtures. One of the concrete mixtures contained sand filler (SCCS); one contained limestone filler (SCCL); the remaining two mixtures were also formulated with limestone filler, but metakaolin was used as an additive by substituting two different percentages of Portland cement with metakaolin at 10% (SCCM10) and 15% (SCCM15). Portland cement with a specific gravity of 3·15 g/cm^3 and a Blaine fineness of

Table 1. Chemical analysis of Portland cement type II, limestone filler, sand filler and metakaolin

Chemical analysis: % by mass	Cement (type II)	Limestone filler	Sand filler	Metakaolin
Loss on ignition	1·60	44·50	7·11	0·78
SiO_2	18·9	1·93	55·85	65·93
Al_2O_3	4·60	0·67	14·42	13·71
Fe_2O_3	3·19	0·32	7·01	0·23
CaO	62·71	49·91	9·50	5·46
MgO	2·80	1·72	2·83	3·84
SO_3	2·37	—	0·08	—
CO_2	—	—	5·70	—
$CaSO_4$	—	—	0·14	—
Cl	—	—	—	—
Insoluble residue	0·62	—	—	—
Alkalis ($Na_2O\%+0{\cdot}658K_2O$ %)	1	—	—	—
Na_2O+K_2O	—	4·33	—	8·93
Free CaO	1·42	—	—	—
Humidity	—	0·10	—	0·10
C_3S	56·20	—	—	—
C_2S	16·23	—	—	—
C_3A	6·30	—	—	—
C_4AF	12·40	—	—	—

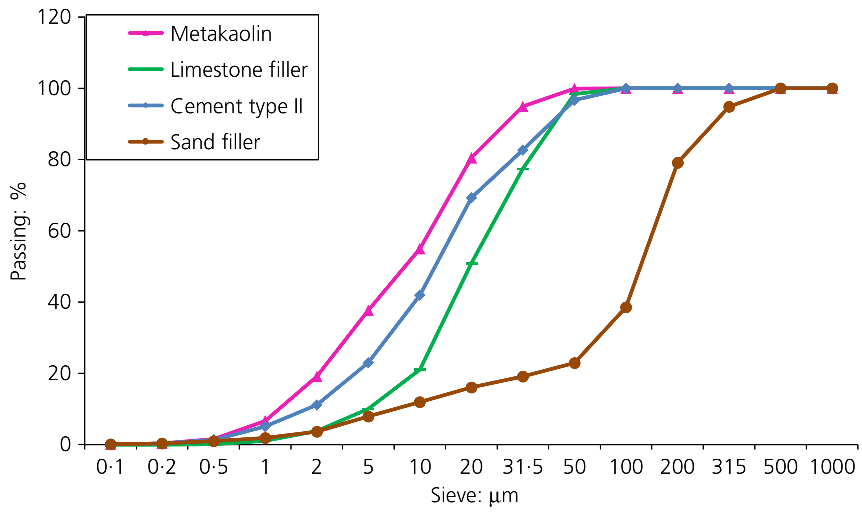


Figure 1. Particle size distribution of fine materials

2900 cm^2/g, in compliance with ASTM C 150 2009 (ASTM, 2009), was used. The metakaolin used in this study has a specific surface area of 3920 cm^2/g and a specific gravity of 2·49 g/cm^3. The chemical analysis and particle size distribution of cementitious materials are shown in Table 1 and Figure 1, respectively.

For all mix designs, crushed angular material of 6–12 mm nominal size was used as a coarse aggregate (gravel), and natural sand with a maximum size of 4 mm was used as a fine aggregate. A high-range water-reducing admixture, with a specific gravity of 1·11 g/cm^3 based on chains of modified polycarboxylate ether (PCE 200), was used in all mixtures to produce SCC. Potable water was also used to prepare concrete mixes. The dosage of superplasticiser was experimentally determined from tests on fresh concrete to obtain a slump flow diameter of 710 ± 20 mm for all SCCs. Table 2 shows the mix proportions of the mixtures. To enhance the stability of SCC mixes, 160 kg/m^3 filler was used in the four mixtures.

Mix design

All concrete mixtures were prepared in a 150-l mixer. The batching sequence consisted of a decant total of the fine and coarse aggregate being placed into the mixer and mixed for 3 min. During this period, two-thirds of the water required was added. Next, cementitious materials were added and mixing was continued for one more minute. After this, the superplasticiser and the remaining water were introduced and the blend

Table 2. Mix design of SCC mixtures (kg/m^3)

Mix name	Water	Cement	W/C_m	Gravel	Sand	Limestone powder	Superplasticiser	Metakaolin	Superplasticiser: % C_m
SCCL	140	350	0·4	800	800	160	5·10	—	1·46
SCCS	140	350	0·4	800	800	—	5·62	—	1·61
SCCM10	140	315	0·4	800	800	160	4·90	35·0	1·40
SCCM15	140	297·5	0·4	800	800	160	6·87	52·5	1·96

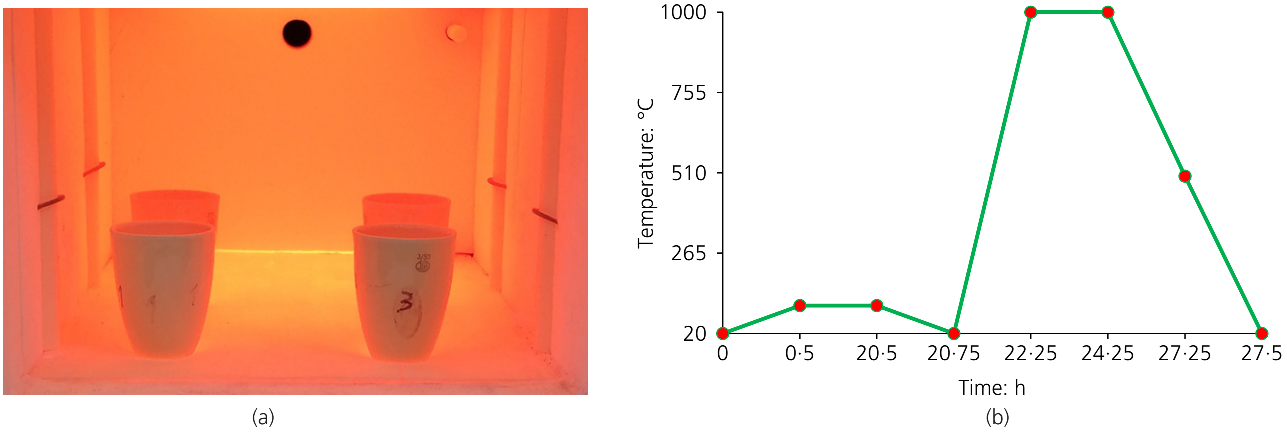


Figure 2. (a) Placement of the crucibles in the furnace; (b) Loading and unloading of the temperature for the measurement of mass charges

was mixed for 2 min. The mixer was covered with a plastic cover to minimise the evaporation of the mixing water.

Casting and curing

Cubic and cylindrical samples were cast in accordance with ASTM C 31 (ASTM, 2012) and ASTM C 511 (ASTM, 2013). After casting, samples were covered with two layers of plastic sheets and placed in a temperature-controlled room at 22 ± 2°C for 24 h. All samples were demoulded after 24 h and cured up to the age of testing in saturated lime solution to prevent possible leaching of calcium hydroxide ($Ca(OH)_2$) from these specimens. The hardened concretes were tested for compressive strength and durability as well as water absorption and permeable voids, capillary absorption, water penetration, chloride diffusion in non-permanent and permanent state, electrical resistivity and determination of the chloride penetration in marine environments.

Test method

Compressive strength

Cubic specimens (15 × 15 × 15 cm^3) were used to determine the mechanical resistance during compression of the various concretes studied. After demoulding, the concrete samples were stored in the lime-saturated water until the date of the test. The compression test was carried out for several ages as young age and long-term conservation (1, 3, 7, 14, 28, 90 and 180 d). This test was performed in accordance with BS 8110-1:1997 (BSI, 1997).

Analysis by scanning electron microscopy (SEM) and X-ray diffraction (XRD)

SEM observations were performed on pozzolans using a JSM 7100F SEM. Before analysis, the samples were metallised with gold–palladium in order to make their surface conductive. XRD analysis was performed on metakaolin using a Bruker D8 X-ray diffractometer with a monochromatic copper (Cu) source and a fast linear detector, the Lynx Eye.

Evaporable and non-evaporable water content measurement

The thermal measurement of the evaporable and non-evaporable water content was carried out by measurements of mass loss as a result of different thermal loadings. The evaporable water content corresponds to the mass loss of water between 105°C and 20°C (Equation 1). Also, the non-evaporable water content corresponds to the mass loss of water between 1000°C and 105°C (Equation 2). The measurements were carried out on binder pastes at 1, 3, 7, 14, 28, 90 and 180 d of ageing. The binder pastes have the same composition as the binder used in concretes but without limestone, and use a lesser amount of superplasticiser. The measurements were carried out on hardened and crushed binder pastes (about 3 g). The powders obtained were put in crucibles and placed in a furnace (see Figure 2(a)). The samples were carried in the ambient temperature to 1000°C with a rate of 10°C/min and bearing 20 h at 105°C and 2 h at 1000°C (see Figure 2(b)). The evaporable water content (W_e) and non-evaporable water content at 105°C

(W_{ne}) are calculated by the following equations

$$1. \quad W_e = \frac{W_{20°C} - W_{105°C}}{W_{20°C}}$$

$$2. \quad W_{ne} \text{ at } 105°C = \frac{W_{105°C} - W_{1000°C}}{W_{105°C}}$$

Water absorption and permeable voids

Absorption and permeable pore volume measurement was performed according to ASTM C 642-97 (ASTM, 1997). This is determined by weighing: (*a*) mass of a specimen concrete, (*b*) mass in the air (while still soaked in liquid), (*c*) mass in water and (*d*) dry mass. The test was carried out after 7, 14, 28, 90 and 180 d of curing in lime water on slices from cast cylinders with dimensions of 100 × 50 mm. Three samples by concrete formulation were tested.

Capillary absorption (sorptivity)

This test was carried out according to AFPC-AFREM recommendations (Durabilité des Bétons, 1997) on slices from cast cylinders with dimensions of 100 × 50 mm (three samples per formulation). The coefficient of sorptivity (S) after 24 h is measured for each formulation at different curing times (7, 14, 28, 90 and 180 d).

Water penetration

This test was measured on three 150 × 150 × 150 mm^3 cube specimens after 7, 14, 28, 90 and 180 d of cure in lime water by applying water under a pressure of 0·5 MPa for 72 h in accordance with BS EN 12390-8 (BSI, 2009). Afterwards, the samples were split and the depth of penetration of the water in concrete measured.

Resistance to chloride penetration

Figure 3 provides a summary of measurement methods and diffusion coefficients obtained. In this study, diffusion in non-steady-state conditions according to the ASTM C 1543-02 standard (ASTM, 2002) and migration in non-steady-state conditions under electric fields by the NT BUILD 492 method (Nordtest, 1999) is investigated.

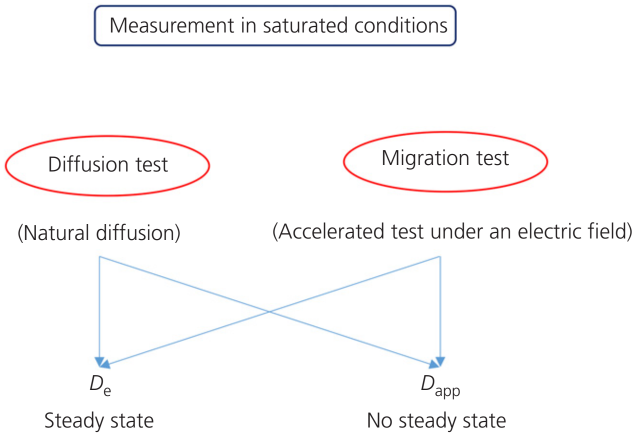


Figure 3. Chart method of chloride diffusion (Deby, 2010)

Non-steady-state diffusion test

This test makes it possible to characterise the material with respect to its resistance to the simple diffusion of chloride ions in the non-steady state. To carry out this test, ASTM C 1543-02 (ASTM, 2002) was used on three concrete specimens (Ø100 × 50 mm) per mix design with ageing of 28 d. They are sealed by an epoxy resin on all the surfaces except on one of the two bases, through which the diffusion of the chlorides will be effected. This sealing is necessary to ensure a one-dimensional penetration of the chlorides (Deby, 2010). The accessible surface is exposed to a saline solution for a specified period of time. The concrete specimens are maintained for 90 and 180 d in the saline solution containing 165 g/l of sodium chloride (NaCl).

At the end of the exposure period, the depth of penetration of chloride ions was measured using silver nitrate ($AgNO_3$) solution. The diffusion coefficient is then deduced using the formula proposed by Baroghel-Bouny *et al.* (2007)

$$3. \quad D_{ns}(\text{dif}) = \frac{x^2}{4t}$$

where D_{ns} (dif) is the apparent diffusion coefficient of chloride ions in the saturated condition (m^2/s), x is the depth of penetration of chloride ions (m) and t is the immersion duration of the specimens in the saline solution (s).

In order to measure the free-chloride concentration profile in the tested specimen, 3-mm slices of concrete (with a precision of 0·2 mm) were cut from the unsealed surface using a fully automatic device manufactured for this study (Figure 4). It should be noted that this apparatus was used in previous work carried out by the first author.

The dissolution and measurement of the free chlorides was obtained by filtration of a solution made by mixing the powder product (almost 5 g) and distilled water. Figure 5 illustrates the apparatus for filtering the solution in order to measure the concentration of free chlorides by silver nitrate solution. The chloride ion concentration is determined by adding the solution of silver nitrate (with concentration of 0·01 M) until the solution turned red. This test was performed according to the standards laid out by APFC-AFREM (Durabilité des Bétons, 1997). The content of free chlorides expressed in grams per 100 g of sample is given by the following relation

$$4. \quad C_l = \frac{(35{\cdot}5 \times C_{Ag} \times V_e \times V_p)}{(10 \times M_{pe} \times V_{fiol})}$$

Figure 4. The device used to obtain concrete powder samples at different depths from the exposure surface (Samimi *et al.*, 2017)

Figure 5. The filtration apparatus

where C_{Ag} is the concentration of silver nitrate (≈0·01 M), V_e is the volume of silver nitrate, M_{pe} is the sampled 5 g of powder product, V_{fiol} is the volume of the filtrate solution up to 250 ml in a volumetric flask, and V_p is the volume of the solution taken (≈ 50 ml) to mix with 2 ml concentrated nitric acid in order to acidify the medium and stabilise the chlorides in solution.

Migration test in non-steady-state conditions under electric fields

For the qualification of the compositions, the natural diffusion in concrete is generally too slow. Many procedures are proposed to accelerate it, in particular by imposing an electric field where migration is concerned. Depending on the mechanisms involved, which must be determined from the magnitude obtained, and according to the usage, it is important to choose the appropriate procedure (Rozière, 2007). Obtaining easily measurable quantities by avoiding tedious procedures in fairly short time frames is a solution frequently researched. In this study, migration in non-steady-state conditions under electric fields by the NT BUILD 492 method (Nordtest, 1999) is investigated. The electric field migration test involves applying a potential difference on either side of the concrete sample through electrodes. Under the influence of the electric field created, the movement of the chlorides, as well as that of the other ionic species, is accelerated towards the electrode with the opposite sign. This test was undertaken for different ages for 3, 7, 14, 28, 90 and 180 d of curing in lime-saturated water on slices of 100 × 50 mm from cast cylinders. According to method NT BUILD 492 (Nordtest, 1999), the chloride migration coefficient can be calculated from Equation 5.

5.
$$D_{\mathrm{nSSm}} = \frac{0{\cdot}0239(273+T)L}{(U-2)t} \times \left(X_{\mathrm{d}} - 0{\cdot}0238\sqrt{\frac{(273+T)LX_{\mathrm{d}}}{U-2}}\right)$$

where D_{nSSm} is the migration coefficient (×10–12 m^2/s), U is the applied voltage (V), T is the anolyte solution temperature (°C), L is the thickness of the sample (mm), X_d is the penetration depth of chlorides (mm) and t is the duration of the test (hours).

Test for the determination of chloride penetration in marine environments

To evaluate the resistance to chloride penetration of the studied SCCs in a marine environment, two different exposure zones are considered and experimentally simulated: the tidal zone and immersion zone. Immersion conditions were simulated by a total immersion of SCC samples in saline solution. Tidal conditions were simulated by imposing wetting and drying cycles. In one chamber, saline water was pumped and drained from another chamber every 6 h to simulate immersion and tidal conditions by using a water pump and electric valve. The test was carried out on slices of cylinders at 90 d of ageing with dimensions of 100 × 50 mm (three samples per formulation). All surfaces of samples are sealed except one, the upper surface, by which chloride diffusion will ingress in order to ensure a one-dimensional penetration of chlorides (Deby, 2010). The samples sealed by resin are saturated in lime water for almost a week (up to arrival at a constant mass). The concentration of the saline solutions prepared for the tidal and immersion media is identical and equal to 40 g/l. The duration of this test for both immersion and tidal conditions is fixed at 90 d in this study. At the end of the test, the depth of penetration of chloride ions was determined by spraying 0·1 M silver nitrate solution on split surfaces.

Electrical resistivity

In this study, the measurement of the electrical resistivity of SCC was carried out according to the FM 5-578 method (FDOT, 2004) using a surface resistivity measuring device with a four-probe Wenner linear array, with a range of 0–100 K Ω-cm, with a resolution of 0·1 KΩ-cm and an accuracy of ± 2% of reading. This test was carried out on cylindrical specimens with diameter 100 mm and 200 mm in height (three specimens per formulation) at various ages of concrete: 3, 7, 14, 28, 90 and 180 d.

Result and discussion

Microstructure investigation

SEM and XRD analysis

Observations by SEM were performed on the studied cementitious material using a JSM 7100F SEM. Figures 6 and 7 present SEM images of limestone filler and sand filler. As indicated, limestone filler is composed of small particles in comparison to sand filler. SEM images of metakaolin powder are illustrated in Figure 8. Metakaolin appears to be composed of dense particles that resemble crystal material. XRD results are shown in Figure 9. XRD analysis of metakaolin showed that it is composed of anorthite and quartz minerals, which are siliceous natural metakaolin.

Fresh state

In order to evaluate the effects of pozzolan on the fresh properties of SCC, slump flow, V-funnel and L-box tests were performed according to the procedure recommended by the EFNARC Committee (EFNARC, 2002); sieve, J-ring and

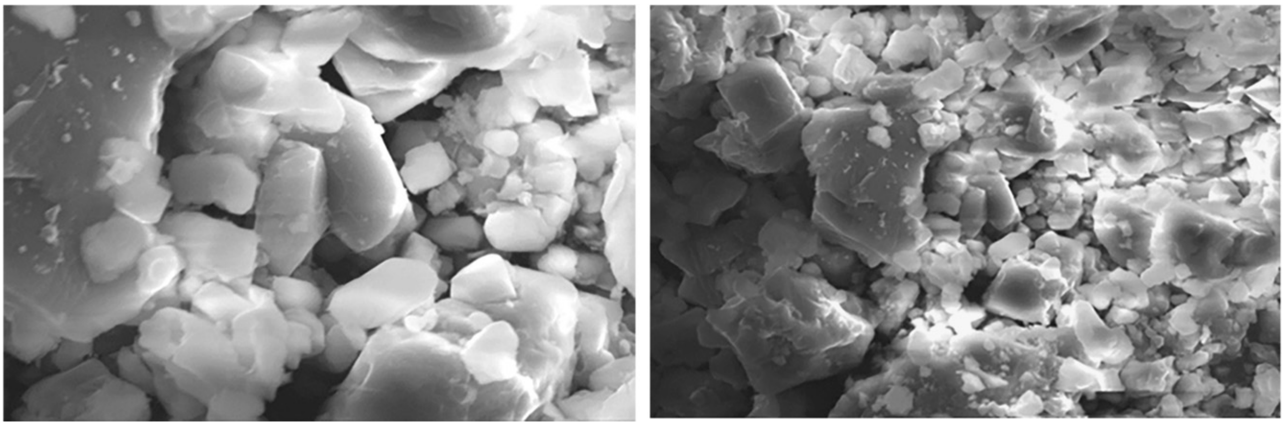


Figure 6. SEM images and chemical analysis of limestone filler

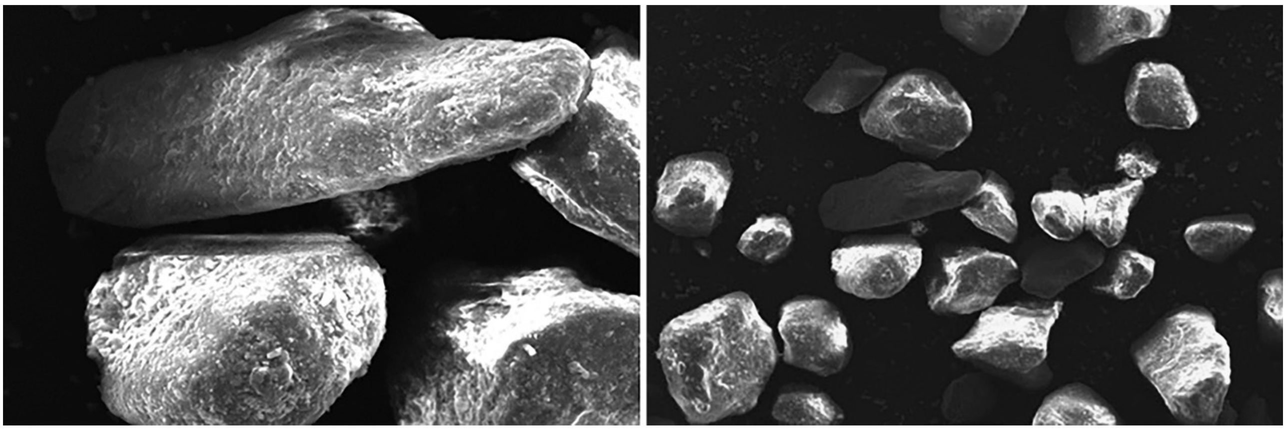


Figure 7. SEM images and chemical analysis of sand filler

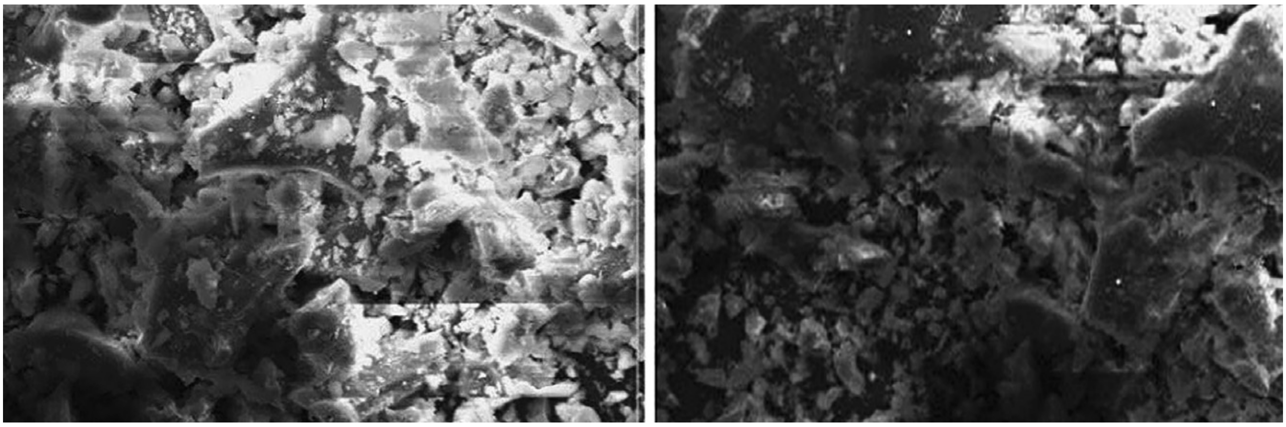


Figure 8. SEM images and chemical analysis of metakaolin powder

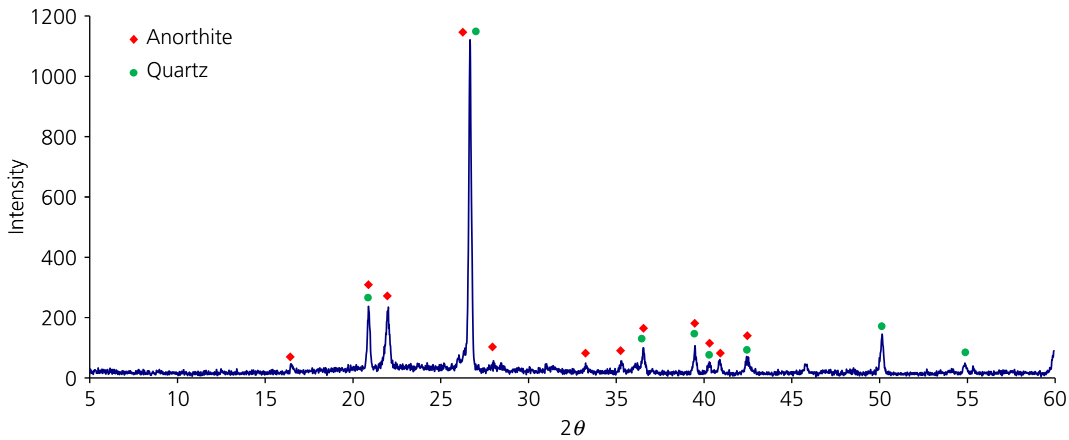


Figure 9. XRD analysis of metakaolin pozzolan. A full-colour version of this figure can be found on the ICE Virtual Library website (www.icevirtuallibrary.com)

Table 3. Test results of fresh SCCs

Mix name	Slump flow		L-box			V-funnel *t*: s		Sieve test	J-ring		U-tube
	Dia: mm	T_{50}: s	h_2/h_1	T_{20}: s	T_{40}: s	1 min	5 min	Segregation: %	ΔH: mm	Dia: mm	$h_2–h_1$: mm
SCCL	730	2	0·9	1·3	2	9	8	5	13	704·6	9
SCCS	719	2·7	0·73	2·2	5·7	6·6	8·4	5·5	5	700	26
SCCM10	690	4·8	0·71	2	5·7	10·63	12·4	4·24	15	650	6
SCCM15	725	2·5	0·8	1·9	5·2	12·37	17·15	7	15	630	14

U-tube tests were also performed according to the procedures recommended by AFGC 2000 (AFGC, 2000), ASTM C 1621 (ASTM, 2017) and BS EN 206-1 (BSI, 2001), respectively. The slump flow of all SCC mixes was near to 700 ± 30 mm and no segregation was observed in the mixes (Table 3). The spreading rate of the concrete is also an indication often taken into account (T_{50} – e.g. time to reach a 500-mm spreading diameter; if it takes more than 5 s a large plastic viscosity is concluded, and if the measured time is less than 1 s, lower viscosity is indicated – in these cases, the risk of segregation and bleeding will increase and an aureole may be created). The ability of concrete to flow through a restricted area without blockage and segregation was evaluated using a V-funnel test. The time of flow from the opening of the outlet to the seizure of flow was recorded and the results presented in Table 3. The V-funnel flow time also depends on the type of application, but it is grouped into two classes by EFNARC (2002): the VF1 class has a flow time of less than 10 s and the VF2 class has a flow time of between 7 and 27 s. Although there are other test methods for assessing passing ability, for example the L-box test, the most common test method is the J-ring test. The experimental flows of a J-ring for concrete mixes based on ASTM C 1621 (ASTM, 2017) are indicated in Table 5. An L-box ratio test was carried out on concrete mixes to measure the cohesiveness and ability of SCC to pass through reinforcements without segregation. The L-box ratio is reported to be between 0·7 and 0·9 for normal SCC; however, a range of 0·8 to 1·0 is also proposed by EFNARC guidelines (EFNARC, 2002). Results of h_2/h_1 ratios relative to this study are presented in Table 5. It can be seen that the values range from 0·71 to 0·9, which are within the specified limits for SCC. The U-box test allows the mobility of confined concrete to be characterised and verifies that the installation of the concrete will not be opposed by phenomena such as unacceptable blockages; the value limit is equal to $h_2 - h_1 = 10$ mm. This test was performed for all mixes and the results are presented in Table 3. It can be seen that the range of values is within the specified limits, approximately. The implementation of SCC, under only the effect of gravity, requires a very high fluidity of the material but it is also essential that the concrete maintains a satisfactory stable and perfect homogeneity. Various tests can be used to characterise the resistance to static segregation of SCC to remain homogeneous after its placement until it begins to set. One such test is the so-called 'stability sieve' developed by GTM (AFGC, 2002) to assess the weight percentage milt (the P_{milt} noted later). The acceptable limitations are as follow: (*a*) $0\% < P_{\text{milt}} < 15\%$: satisfactory stability, (*b*) $15\% < P_{\text{milt}} < 30\%$: critical stability (segregation test necessary on site) and (*c*) $P_{\text{milt}} > 30\%$: very poor stability (systematic segregation, unusable concrete).

Compressive strength

The results of the compressive strength of the mixes at the age of 1, 3, 7, 14, 28, 90 and 180 d are plotted as a function of time in Figure 10. From the results shown in Figure 10, the compressive strength of all formulations at all maturities is between 6·3 and 67·5 MPa. According to these results, a higher resistance development is noted when using sand filler. This is true up to 90 d of maturation; beyond, the resistance stabilises for the concrete with sand filler but continues to increase for the SCC with limestone filler. This may be explained by the adhesion between the matrix and the calcareous grains, which becomes better with time. The greatest resistance of 1 to 3 d and 7 to 180 d was obtained for SCCS and SCCM15, respectively. Concrete of 15% metakaolin had a very positive effect on the compressive strength from 7 d of the cure. This result is quite consistent with the results given by Siddique and Klaus (2009). On the other hand, the increase in the compressive strength of the concrete containing 10% metakaolin compared to the control concrete occurred from the age of 90 d. This increase for SCCM10 is more visible at the age of 180 d. The compressive strength in concrete containing 10% metakaolin is 1·02 and 1·1 times higher than that of the control concrete at 90 and 180 d of curing, respectively. In this study, the increase of compressive strength in SCC containing metakaolin is in a good agreement with the experimental results of previous studies performed on SCC, normal concrete and geopolymer concrete whose cement has been replaced by metakaolin at various percentages (Alanazi *et al.*, 2017; Badogiannis and Tsivilis, 2009; Dinakar *et al.*, 2013; Güneyisi *et al.*, 2012; Hassan *et al.*, 2012; Khatib and Clay, 2004; Siddique and Klaus, 2009).

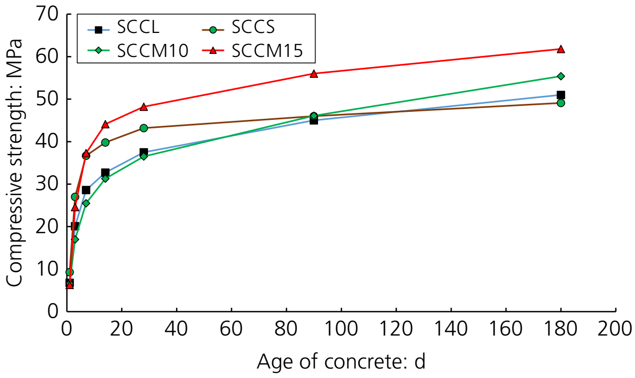


Figure 10. Compressive strength of the different formulations studied

The major difference between the metakaolin found in Iran and that found in Europe is the silicon dioxide (SiO_2) and aluminium oxide (Al_2O_3) content. As a matter of fact, silicon dioxide is greater in metakaolin found in Iran. Also, the compressive strength of concrete with metakaolin from Europe, during early age, is less than that of concrete mixtures without additives. On the other hand, the Iranian metakaolin increases the compressive strength from an earlier age. This behavioural difference is well documented in research work conducted by the present authors and Gilan *et al.* (2012) in Iran, as well as in research conducted by San Nicolas *et al.* (2014) in France.

Evolution of evaporable water and non-evaporable water content

The main purpose of conducting thermal analysis is to measure the content of evaporable and non-evaporable water at 105°C of binder pastes. The amount of evaporable water can be linked to the porosity. The non-evaporable water is considered to be an important and reliable indicator of the hydration progress in the modification of the microstructure. In this way, thermal measurement of evaporable and non-evaporable water content can be useful to justify the mechanical behaviour and durability properties of the studied SCCs. The results relating to the evaporable and non-evaporable water at 1, 3, 7, 14, 28, 90 and 180 d of ageing are shown in Figures 11 and 12, respectively. An increase over time of maturation of non-evaporable water content was found, accompanied by a decrease in evaporable water for all

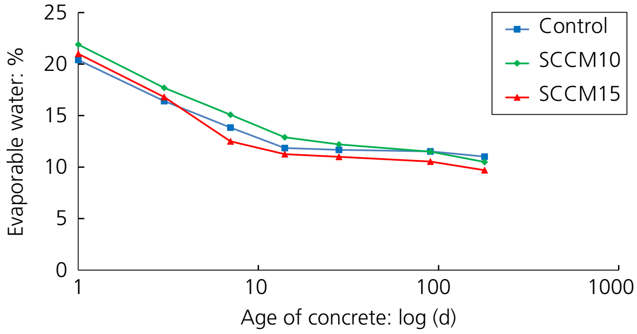


Figure 11. Evaporable water content in the studied mixtures at different ages

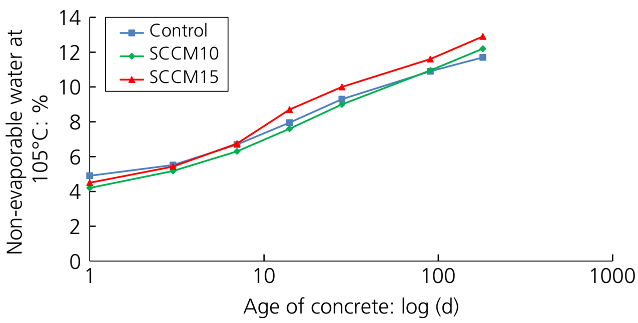


Figure 12. Non-evaporable water content at 105°C in the studied mixtures at different ages

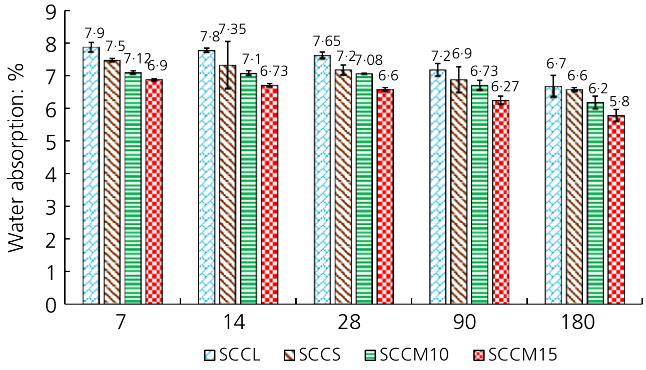


Figure 13. Water absorption (%) of studied SCCs

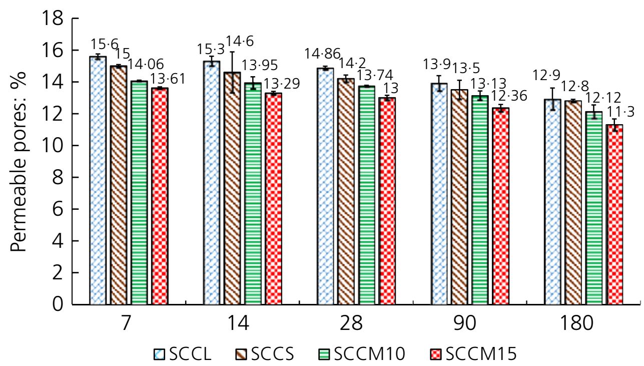


Figure 14. Volume of permeable pores (voids) (%) of studied SCCs

formulations. This first observation is predictable due to the continued hydration of the binders. Moreover, a paste with metakaolin, even if it contains less cement and therefore less clinker, has been able to develop a non-evaporable water content close to that of the control, which is seen to be greater in the case of binders with 15%, and this from 7 d of maturation. These results indicate the presence of additional hydration, which may be related to the pozzolanic activity and the formation of hydrates.

Water absorption and permeable voids

The test results for water absorption and void permeability are presented respectively in Figures 13 and 14.

The results indicated that using metakaolin in place of cement and sand filler instead of limestone filler in SCC can reduce water absorption and the number of permeable pores in comparison to control concrete at all conservation durations. According to the results illustrated in Figure 13, the water absorption rate in SCCM15 is lower than in all other formulations. The water absorption in SCCM15 is 1·14, 1·15, 1·16, 1·15 and 1·16 times lower than that of the control concrete at 7, 14, 28, 90 and 180 d of ageing, respectively. The number of permeable pores of the studied SCCs is presented in Figure 14. According to the results illustrated in this figure, the SCC containing metakaolin shows good behaviour in the reduction of the number of permeable pores compared to the control concrete. This fact is highlighted by the increase in the additive level of metakaolin from 10 to 15% cement replacement. The reason can be found in the conversion of continuous pores to more discontinuous pores due to the pozzolanic reaction of metakaolin. Its pozzolanic reaction can contribute to the formation of additional hydration, which can lead to lower connected porosity. Indeed, the decreased void content increases the load-carrying capacity of concrete, and therefore the compressive strength of SCCM10 and SCCM15 is increased. Moreover, the amount of non-evaporable water in paste with metakaolin and the compressive strength of SCCM10 and SCCM15 are completely consistent with the results with regard to water absorption and permeable pores. This fact is more visible with long curing times. The different properties studied here can be related to compressive strength, as shown in Figures 15 and 16. A linear equation seems to describe correctly the evolution of water absorption and permeable pores with compressive strength. Compressive strength increases with the decrease of these properties.

Water penetration

The rate of water penetration into the concrete is a parameter that allows the performance of the concretes to be compared

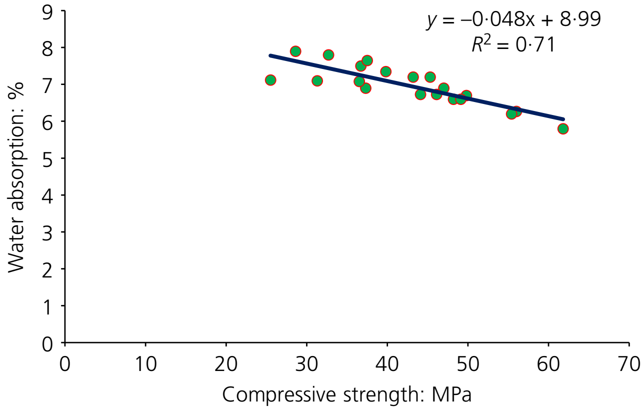


Figure 15. Relationship between water absorption and compressive strength

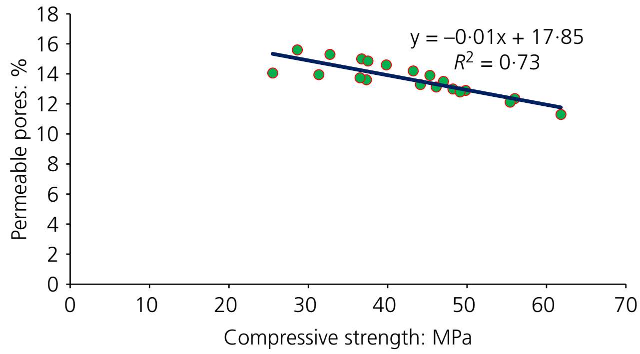


Figure 16. Relationship between the volume of permeable pore and compressive strength

with respect to permeability. The penetration of water in concrete can cause physical and chemical damage. Water as a solvent is able to dissolve many cementitious components. Many ions cause damage in concrete by the penetration of water in concrete. For this reason, the penetration of water in concrete can be used as an indicator in the study of concrete durability. The results of the measurement of water penetration are shown in Figure 17. This figure shows that the substitution of 10 and 15% Portland cement with metakaolin leads to a reduction in the penetration depth of the water and thus to the reduction of the water permeability. The maximum and minimum water penetration depth in the SCCs studied after all ages of hardening was obtained for SCCS and SCCM15, respectively. The depth of water penetration of the control SCC was 1·39, 1·45, 1·47, 1·67 and 2·5 times higher compared to that of the SCCM15 at 7, 14, 28, 90 and 180 d, respectively. By contrast, the water penetration depth of the control concrete is 1·56, 1·66, 1·8, 2·05 and 2·47 times lower than that of the SCCs at 7, 14, 28, 90 and 180 d, respectively. According to the results given in Figure 17, the SCC containing 15% metakaolin, SCCM15, exhibits the best performance. This can be explained by its higher pozzolanic reactions, which have a strong impact on the decreased permeability in SCC.

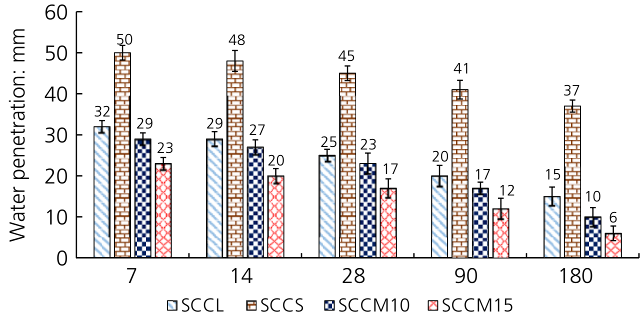


Figure 17. Water penetration in the studied SCCs after 7, 14, 28, 90 and 180 d of curing

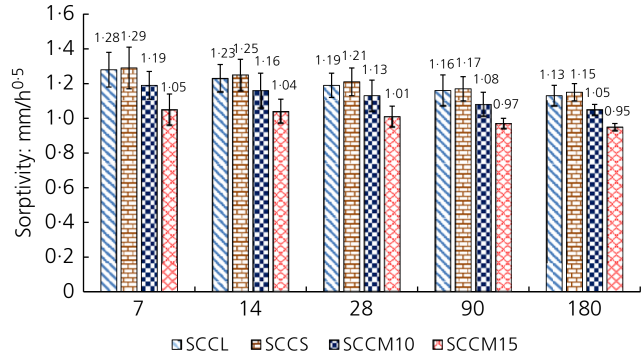


Figure 18. Sorptivity coefficients of the studied SCCs at different curing ages (mm/h$^{0.5}$)

Capillary absorption (sorptivity)

The higher the capillary absorption, the more quickly the material is able to be invaded by the liquid in contact. The sorptivity coefficients (*S*) after 24 h of testing are presented for the various SCCs at different ages (7, 14, 28, 90 and 180 d) in Figure 18. According to this figure, the absorption coefficient of concrete containing sand filler (SCCS) is slightly higher than that of the control SCC at all ages of curing. From Figure 18, the capillary absorption coefficient of SCCL is 1·08 and 1·19 times higher than that of SCCM10 and SCCM15 aged 180 d, respectively. The results show that the incorporation of metakaolin has a very beneficial effect on the reduction of capillary absorption of SCCs. On the other hand, this reduction is greater when the additional content is increased.

Resistance to chloride penetration

The chloride ions penetrate into concrete and cause, from a certain concentration in the pore solution, at the reinforcements, the local destruction of the passivation film and the initiation of localised corrosion. In general, the chloride in the normal case does not directly effect all the damage of concrete (Dhir *et al.*, 1990; McCarter *et al.*, 1992). The exposure of concrete to chloride has two main sources: sea water and icing salts. The penetration of chlorides in concrete is established by sophisticated physical and chemical processes from three sources (De Schutter and Audenaert, 2007):

- The location of the structure. In marine environments, the amount of chloride in contact with the concrete depends on the location of the structure, provided that they are completely submerged or placed in the tidal zone or only in contact with the salt fog. However, in situations such as roads, frequent use of icing salts in very cold weather causes difficulties in calculating the amount of pulverised chloride on the structures.
- The properties of chloride penetration of the different materials used in the mixture and the age of the concrete.
- The mechanisms of chloride penetration are not isolated (simple diffusion), but are combined with convection (absorption), chemical and physical connections, and interaction of other coexisting ions. The change in temperature, rain and sun introduces variations which should also be taken into account.

The exposure conditions of the structure in its environment are a key factor for the modelling of structures subjected to chloride corrosion. Depending on the type of exposure, different transport mechanisms are considered for ion chlorides (see Figure 19).

NON-STEADY-STATE DIFFUSION

The results for the penetration depth and chlorides diffusion coefficient in non-steady-state conditions for the SCCs studied at 28 d of treatment after 90 and 180 d of immersion in the saline solution are illustrated in Figures 20 and 21, respectively.

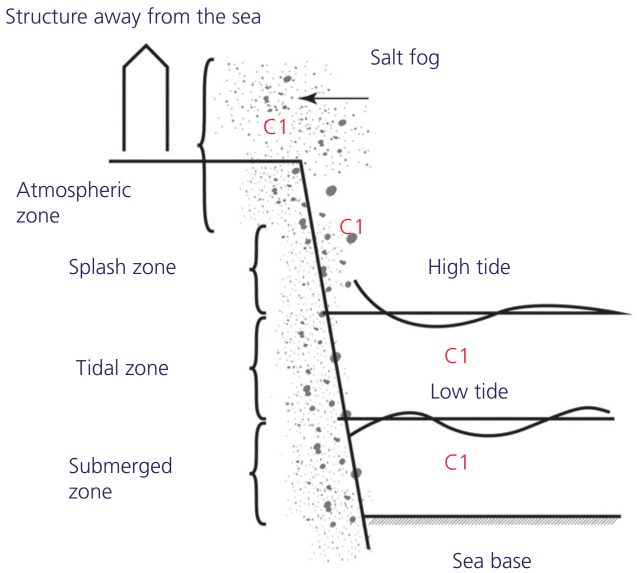


Figure 19. Marine environment (De Schutter and Audenaert, 2007)

The results illustrated in Figures 20 and 21 show that the penetration depth of the chlorides and consequently the apparent diffusion coefficient of the chlorides in the SCC based on sand filler and the control SCC are almost identical. On the other hand, for SCCs with metakaolin, the penetration depth of the chlorides and therefore the apparent diffusion coefficient are much lower compared to those of the control concrete. The positive effect of incorporating metakaolin is undeniable. Furthermore, SCCs containing 15% metakaolin are relatively more resistant to chloride penetration than SCC based on 10% metakaolin.

FREE-CHLORIDE CONCENTRATION PROFILE

Chlorides exist in concrete in two forms: free and bound. The bound chloride is found in the form of a complex salt such as calcium monochloro-aluminate hydrate; it can also be attached to the C–S–H. It is assumed that bound chloride cannot initiate corrosion. However, free chloride ions, which are highly mobile, have a key role in the corrosion of reinforcements. The content of free chlorides is generally determined by measuring the content of water-soluble chlorides. In this study, the content of water-soluble chlorides was measured on concrete powders obtained from the exposed surface using the device explained in the 'Test method' section. The profiles of the free chlorides measured in the concrete are plotted in Figure 22.

The results presented in Figure 22 show that the SCCs containing metakaolin have lower penetration depths of the chlorides

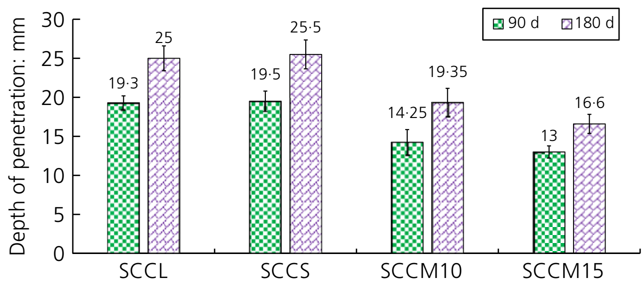


Figure 20. Depth of chloride ions penetration in the studied SCCs at 28 d of ageing after 90 and 180 d of immersion in saline solution

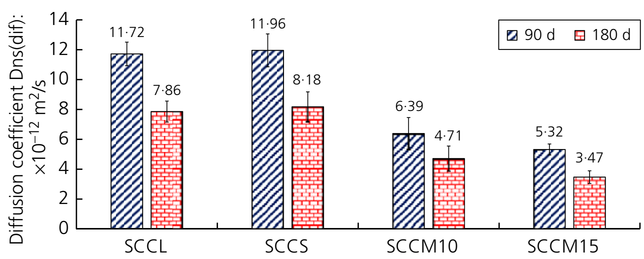


Figure 21. Chloride diffusion coefficient in SCCs at 28 d of ageing after 90 and 180 d of immersion in saline solution

compared to the control concrete, which is in agreement with the colorimetric measurements. The chloride penetration profiles deduced from the water-soluble distance from the surface are higher than those measured using a colorimetric method based on silver nitrate solution for diffusion in non-steady state. This can be explained by the lesser sensitivity of the colorimetric method to the low values of chloride content and probably also to the possible ingress of chloride into the sound zone of the sample during the conservation time between the end of the diffusion test and the measurement of water-soluble chloride. These results are in agreement with the results of the capillary absorption measurements, which were found to be higher for the control concrete compared to the mixtures with pozzolan at all ages of curing. The higher capillary absorption indicates a greater connected porosity and consequently a higher free-chloride content in the concrete.

Non-steady-state migration

The aim of this part of the experimental study is to evaluate the effect of sand filler and metakaolin used on the evolution of the chloride migration coefficient in the four treated SCCs. The cure period effect is also taken into account by considering different conservation ages: 3, 7, 14, 28, 90 and 180 d. The evolution of the migration coefficient of the chlorides during the maturation time for the different SCCs is shown in Figure 23.

According to the results illustrated in Figure 23, the SCC with sand filler behaves in a very similar way to the control concrete. On the other hand, the evolution of this coefficient over time is different in the case of SCCs with metakaolin. On very short terms (3 d), SCCs with metakaolin have higher values compared to control concrete. This is due to their greater porosity. However, the migration coefficient of these SCCs will quickly drop and then be lower than that of the control concrete. At the age of 180 d, the migration coefficient of the SCCM10 and SCCM15 is 1·5 and 1·9 times lower, respectively, than that of the control SCC. Moreover, Figure 23 shows the curing effect on the decrease in the chlorides migration coefficient. In this study, the positive influence of metakaolin in reducing the chloride penetration is in agreement with the results obtained by Kim *et al.* (2007) and Ramezanianpour and Bahrami (2012). A very rapid decrease is observed in the mixtures with metakaolin, whereas this decrease is slower and less important in the case of the control SCC and the SCC containing sand filler. This result can be explained at least partially by the low porosity, as can be expected from the capillary absorption results. Another explanation for the positive effect of metakaolin is related to the reactivity of this pozzolan and consequently the consumption of calcium hydroxide, which most likely results in increasing tortuosity and reduction of hydroxide ions in the interstitial solution. Therefore, the ionic concentration in the interstitial solution decreases. Consequently, the conductivity and penetration of chloride ions will decrease.

The values obtained at 180 d make it possible to classify the control concrete and the SCC containing metakaolin with good resistance against the penetration of chlorides according to the classification given by Tange Jepsen *et al.* (2001) and indicated as follows

- $D < 2 \times 10^{-12}$ m^2/s: very good resistance against the penetration of chlorides
- $D < 8 \times 10^{-12}$ m^2/s: good resistance against the penetration of chlorides

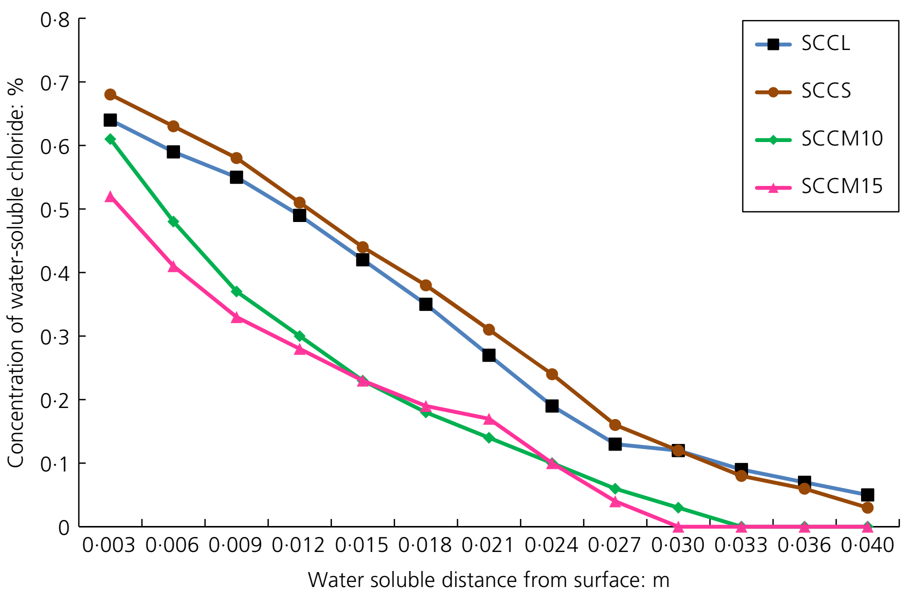
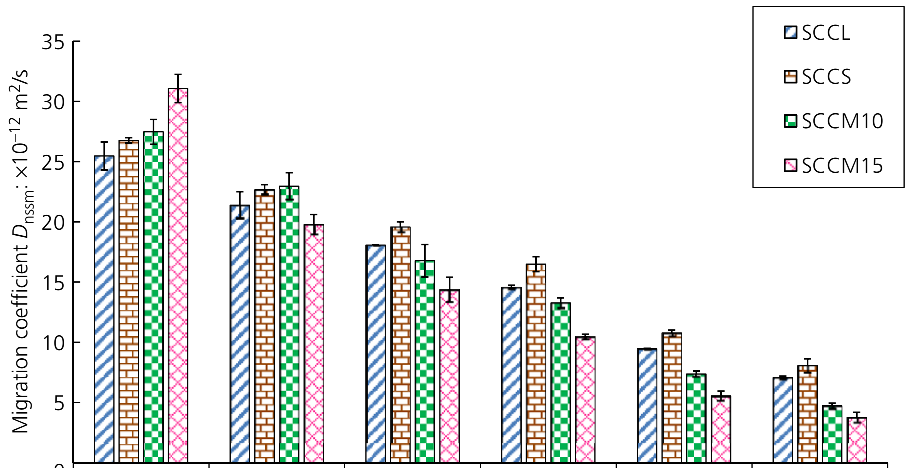
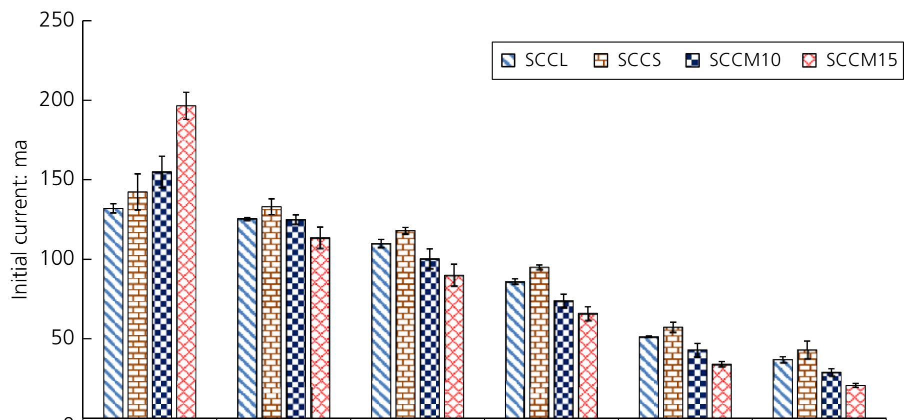


Figure 22. Water-soluble chloride penetration profile in the different studied SCCs



	3	7	14	28	90	180
SCCL	25·47	21·39	18·07	14·57	9·47	7·07
SCCS	26·77	22·67	19·57	16·49	10·77	8·07
SCCM10	27·47	22·97	16·77	13·27	7·37	4·73
SCCM15	31·07	19·77	14·37	10·47	5·56	3·77

Figure 23. Migration coefficient of different mixes at different ages



	3	7	14	28	90	180
SCCL	132	125·5	110	86	51·33	37
SCCS	142·33	133	118	95	57·33	43
SCCM10	155	125	100·33	74	43	29
SCCM15	196·5	113·5	90	66	34	20·67

Figure 24. Initial current of different mixes at different ages due to RCMT apparatus

- $D < 16 \times 10^{-12}$ m^2/s: moderate resistance against chloride penetration
- $D > 16 \times 10^{-12}$ m^2/s: not suitable for an aggressive environment.

The SCC with sand filler is also considered to have almost good resistance to chloride penetration at the same maturity of 180 d.

The quality of the concrete can also be assessed by measuring the current that passes when applying 30 V across the sample. This means that the greater the current, the lower the resistance of the concrete to the penetration of chloride ions. The amount of initial current of different mixes at different ages due to the RCMT apparatus is presented in Figure 24. According to these results, the amount of initial current in the SCC containing sand filler is greater than in the other formulations. From 7 d of cure, the initial current passed in SCCM15 is lower than in the other mixtures. This can be justified because the 15% substitution of metakaolin in SCC using pozzolanic reaction can create a denser microstructure by converting continuous pores to discontinuous pores. Figure 25

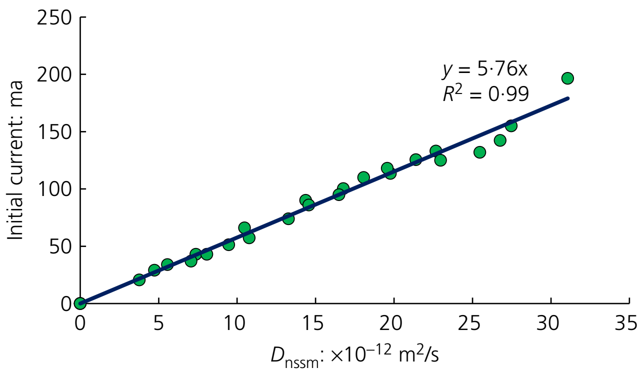


Figure 25. Evolution of initial current according to migration coefficient of the studied SCCs

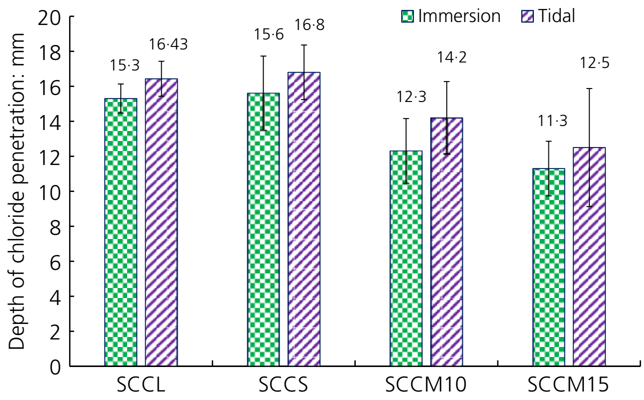


Figure 26. Depth of chloride ions penetration in SCCs at age of 90 d after 90 d of immersion, and tidal in saline solution

shows that there is a direct relationship between the migration coefficient and the initial current. The initial current measurement appears to be a good indicator of sustainability, which can be directly related to the migration coefficient.

RESISTANCE TO CHLORIDE PENETRATION IN IMMERSION AND TIDAL CONDITIONS

Numerous other investigations have been carried out on the penetration of chloride ions in the vicinity of seashores and ports (Bermudez and Alaejos, 2010; Conciatori *et al.*, 2008). It is well known that Kuzel's salt ($3CaO.Al_2O_3{\cdot}0{\cdot}5CaSO_4{\cdot}0{\cdot}5CaCl_2{\cdot}10(11)H_2O$) and Friedel's salt ($3CaO.Al_2O_3CaCl_2{\cdot}10H_2O$) can be formed when the chloride ions are present during cement hydration. When the chloride ions react with C3A, Friedel's salt is formed. Also, the reaction of the chloride ion with C4AF leads to the formation of Kuzel's salt. In order to evaluate the studied SCCs' resistance to chloride penetration in the marine environment, two different conditions of exposure were considered and experimentally simulated: tidal and immersion.

The immersion condition was simulated by the total immersion of the SCC samples in saline solution. The tidal condition was simulated by imposing wetting and drying cycles as explained previously. At the end of the tests, after 90 d of ageing, the penetration depth of the chloride ions in all formulations was determined for both immersion and tidal conditions. The results are shown in Figure 26 and illustrated in Figure 27. The penetration depth of the chlorides in all mixtures is higher under tidal conditions than immersion conditions due to the drying of the surface of the concrete. Its value for the SCCL, SCCS, SCCM10 and SCCM15 is respectively 1·07, 1·08, 1·15 and 1·11 times higher under the wetting–drying conditions compared to the immersion conditions. This increase in chloride penetration should be higher under more severe drying conditions, as in the case of the presence of wind and sun. The mixtures with metakaolin show better resistance to chloride penetration, and this under both tidal and immersion conditions, which shows the positive effect of the use of metakaolin. In this study, the results showing the increase in resistance of SCCs containing metakaolin in marine environments are in good agreement with the experimental results of research studies performed on concrete blended with metakaolin in a seawater environment, as presented, for example, by Duan *et al.* (2015).

In fact, chloride penetration should be greater in tidal conditions than in immersion conditions, based on the following two points:

- The increase of concrete's diffusion speed rate: in this situation, the saline solution penetrates in concrete through capillary suction by means of the drying and wetting of the concrete. As such, the humidity and chloride ions increase inside the concrete, causing the chloride ion diffusion speed to increase.
- The increase of concrete's demolition rate: in this situation, due to the lack of humidity in tidal zones, saline crystals appear on the surface of concrete samples resulting in increased internal stress and micro-crack. This chloride ion penetration increase can be much greater when the hard drying conditions are magnified by wind and/or sun (Samimi *et al.*, 2017).

Electrical resistivity

Electrical resistivity is one of the main indicators of the durability of cementitious materials. In general, high electrical resistivity is correlated with a high resistance to chloride penetration and consequently a high resistance to corrosion. The results of electrical resistivity for the different mixtures are presented in Figure 28. This figure clearly shows the very significant influence of metakaolin on the increase in electrical resistivity at different ages: 3, 7, 14, 28, 90 and 180 d. This increase is more remarkable when the percentage of metakaolin addition is increased. This may be related to the pozzolanic reaction, which leads to a dense structure and a lower

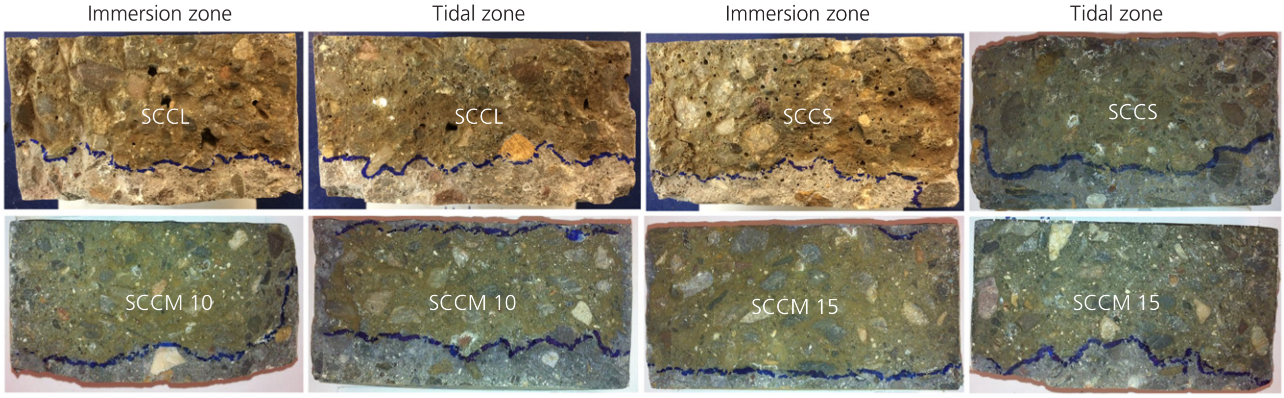


Figure 27. Chloride penetration depth of SCCs samples after 90 d of testing, highlighted using silver nitrate solution: left: in immersion conditions; right: in tidal conditions

concentration of OH- in the interstitial solution. This fact ultimately increases the electrical resistivity and improves the durability of the concrete against the penetration of chlorides. For SCC containing sand filler, the electrical resistivity is always lower than that of the control concrete. According to Figure 28, the age of the concrete has a positive influence on the increase of the electrical resistivity, which is predictable due to the further hydration of the binder. However, this increase is greater when metakaolin is used.

The electrical resistivity in concrete increases when the chloride migration coefficient decreases. This can be clearly observed in Figure 29. According to the relation between electrical resistivity and the chloride migration coefficient, it can be concluded that there is a close relationship between the two last properties. These results are in good agreement with the experimental results of previous studies performed on concrete composites based on various supplementary materials (Ramezanianpour and Bahrami, 2012).

Conclusion

Based on the obtained results from this experimental study performed on a reference SCC with limestone filler (SCCL), two SCC composites with limestone filler and 10% and 15% metakaolin substitution (SCCM10 and SCCM15, respectively), and one other SCC composite based on sand filler (SCCS) instead of limestone, the following conclusions can be drawn:

(1) The results of the mineralogical analysis showed that the studied metakaolin is composed of a mixture containing glass phases as well as mineral phases such as quartz and anorthite.
(2) The results of the fresh characterisation showed that the substitution of 10 and 15% Portland cement with metakaolin as well as the total substitution of the limestone filler by sand filler makes it possible to obtain stable SCCs with good capacities of filling without blocking.

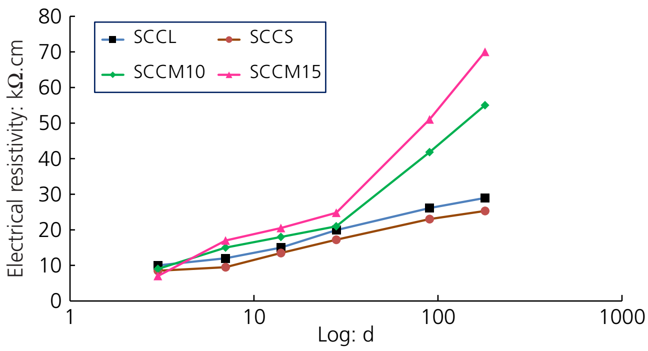


Figure 28. Evolution of the electrical resistivity with curing time

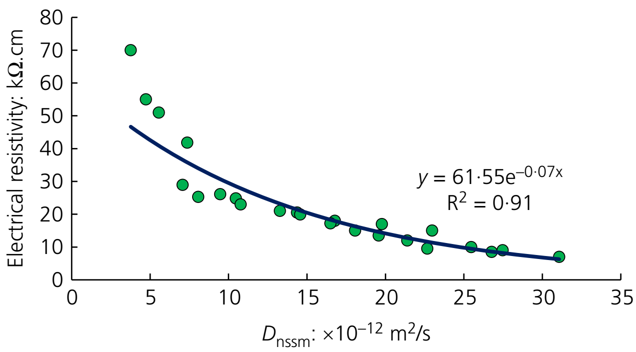


Figure 29. Evolution of the electrical resistivity according to migration coefficient

(3) The results of the mechanical characterisation of the four SCCs showed that the total substitution of the limestone filler by sand filler makes it possible to obtain an SCC with better mechanical resistances compared to the control SCC. This is true up to 90 d of maturation; beyond this, the resistance stabilises for the concrete with

sand filler but continues to increase for the control SCC based on limestone filler.

(4) The results also showed that the substitution of 10% Portland cement by metakaolin reduces the resistance in the very short term (from 1 to 28 d), but from 90 d of maturation the compressive strength is superior to that of the control concrete. On the other hand, the SCC containing 15% of the metakaolin shows a higher compressive strength from 7 d of cure in comparison with the other formulations. This was confirmed by the measurements of evaporable water and non-evaporable water content.

(5) The results relating to the compressive strength and porosity are coherent – namely, that the more compact the matrix is, which would be due to the granular skeleton (sand filler) or the pozzolanic reaction (metakaolin), the greater the compressive strength and the greater the absorption after immersion or after immersion/boiling, and permeable pore number decreases.

(6) The results of capillary absorption show that the incorporation of metakaolin has a very beneficial effect on the reduction of capillary absorption. On the other hand, this reduction is greater with the increase in the addition content. The depth of penetration of the water is thus minimal in the case of the presence of metakaolin and maximal in the case of sand filler.

(7) The SCC with sand filler behaves in a way very similar to the control concrete with values of migration coefficient that are almost identical to 90 and 180 d. On the other hand, the evolution of this coefficient over time is different in the case of SCCs with metakaolin. At very short terms (3 d), the SCCs with metakaolin exhibit higher values of migration coefficient compared to the control concrete. However, the migration coefficient of these SCCs will quickly drop and then be lower than that of the control concrete.

(8) The penetration depth of the chlorides in all mixtures is higher under tidal than immersion conditions due to the drying of the surface of the concrete. Mixtures with metakaolin show much better resistance to chloride penetration than control concrete and SCC with sand filler in both tidal and immersion conditions, which shows the positive effect of the use of metakaolin.

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