Practical evaluation of rapid tests for assessing the Chloride resistance of concretes containing Silica Fume

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Abstract. The present study is an exploratory research that appraises and compares the performance of four rapid tests for assessing the chloride penetration of concrete; Rapid Chloride Permeability Test (RCPT), Rapid Chloride Migration Test (RCMT), Surface Electrical Resistivity (SR) and Modified Rapid Chloride Permeability Test (MRCPT). Due to the concern raised by some researchers on that the conductivity of the pore solution impinges upon the results on some of these rapid tests, silica fume was used as supplementary cementitious material in proportions of 7.5% and 15%. All four methods exhibited substantial reduction in the chloride permeability of concrete mixtures containing silica fume compared to the control mixture at the ages of 28 and 90 days. The variations in performance caused by silica fume usage were significant for RCPT and SR methods, moderate for RCMT and marginal for MRCPT. Results show that the variation in the conductivity of the pore solution significantly alters the results of SR and RCPT tests, and marginally alters those of MRCPT.

Keywords: concrete; chloride permeability; conductivity; pore solution

1. Introduction

One of the early deterioration of reinforced concrete structures is the corrosion of steel bars resulting in lower durability (Safehian and Ramezanianpour 2013; Ahmadi *et al*. 2014). Carbonation and chloride ions are the main factors affecting the alkalinity of concrete. Chloride ions dissolved in water; which were resulted from the use of deicing salts, ions in the soil, seawater and groundwater could penetrate the concrete and reach the surface of the reinforcement (Ramezanianpour *et al*. 2011). Although the primary mechanism of chloride transport for the near-surface unsaturated concrete is absorption, the accumulation of chlorides in this layer leads to further penetration of chlorides into concrete by diffusion. As a consequence, diffusion becomes

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the most dominant mechanism of chloride transport at higher depths (Atkinson and Nickerson, 1984). Driving force on diffusion of chloride ions is the gradient of ions concentration in the different parts of the concrete (Audenaert *et al.* 2010).

Regarding the importance of measuring permeability of concrete against chloride ions, several methods have been proposed (Hooton and Karkar 2012). The early methods of measuring the diffusion coefficient of chloride ions into the concrete were based on immersion of concrete samples in a saline solution. Then the chloride ion diffusion coefficient is obtained by determining the chloride profile and fitting the Fick's second law (Bagheri and Zanganeh 2012). However, these methods usually take very long time under the normal conditions. Therefore, the use of accelerated methods has gained great attention over the past decades.

One of these approaches was to increase the concentration of dissolved chloride ions, e.g. ASTM C1556 and ASTM C1543 test methods (ASTM 2011, ASTM 2010). However, the diffusion behavior might not follow Fick's law, due to the interference and interaction of the ions (Zhang and Gjørv, 1994).

Another approach is to use an electric potential as the external force to increase the penetration of ions. This approach is widely used (Spragg *et al.* 2013).

In some tests, such as RCMT (Nordtest, 1999), the penetration of chloride ions is accelerated by an external electric field applied to the sample, and the diffusion coefficient is measured. In other tests, such as RCPT (ASTM, 2012), SR (AASHTO, 2011) and MRCPT (Ramezanianpour *et al*. 2012), the resistance of concrete against chloride ion penetration is assessed by measuring the electrical conductivity of concrete.

An important question is the extent to which these tests can be used for predictive purposes. RCMT is one of the quick methods, which has shown better performance with a good correlation with the long term tests (Tang and Sørensen 2001). However, exaggerated improvement in the chloride resistance of concrete containing supplementary cementitious materials, as determined by the RCPT compared to the long-term chloride permeability results, has been reported by researchers (Bagheri and Zanganeh 2012; Riding *et al*. 2008). However, Theoretical and experimental studies indicate that there is a relationship between the electrical conductivity and the permeability of concrete (Streicher and Alexander 1999; Safehian and Ramezanianpour 2015; Wang 2015; Yoon *et al*. 2015). The electrical conductivity of concrete was the expression of the mobility of ions - containing chloride ions - in pore solution of the concrete. Hence it seems that logical relation between electrical conductivity and permeability of the concrete exists. In a specific structure, the higher permeable parts show relatively higher electrical conductivity.

In 2002, the Florida department of transportation started a research program to evaluate all available electrical indicators of concrete chloride penetration resistance (Chini *et al*. 2003). The first project under the research program had the purpose of investigating the possibility of replacing the RCPT method by Surface Resistivity test. Correlations were investigated from both RCPT and SR tests of more than 500 sample sets. The two tests showed a strong relationship for concrete specimens tested at 28 days. Similar results were obtained by other researchers (Ramezanianpour *et al*. 2011; Torrent and Luco 2007; Tang *et al*. 2011). It is worth mentioning that the conductivity of concrete samples affects the results of most of chloride permeability rapid tests (Ramezanianpour *et al*. 2011), the conductivity of the pore solution is a key determinant in the results of these tests. This problem is particularly observed in studying concrete samples containing supplementary cementitious materials, especially active pozzolans such as silica fume (Ramezanianpour *et al*. 2014). The underlying reason is that OH^- ions of the pore solution, which are essential in its conductivity, are consumed in pozzolanic reactions and the conductivity of the

Table 1 Chemical and physical characteristics of cement and silica fume

Chemical Composition* (%)		OPC	SF
	CaO	65.3	0.36
	SiO_2	20.8	94.66
	Al_2O_3	4.3	0.31
	Fe_2O_3	2.2	0.60
	MgO	2.17	0.78
	K_2O	0.63	0.22
	Na_2O	0.36	0.22
Loss on ignition (%)		0.91	1.77
Physical properties	Specific gravity	3.15	2.14
	Fineness** (cm^2/g)	2800	---

* Chemical Composition is specified according to ASTM C114

** Fineness is determined by Blaine apparatus based on ASTM C204

pore solution reduces significantly. Hence, the results of these rapid tests for concrete samples with supplementary cementitious materials may be unreliable. In the method proposed by Streicher *et al.* (Streicher and Alexander 1999), specimens are saturated with 5 M NaCl solution before measuring the conductivity of them. By saturating specimens with a highly conductive solution, they showed virtually the same pore solution conductivity.

As discussed before, there are different methods to assess the chloride permeability of concrete, each with strengths and shortcomings that are contingent on the application, and must be selected according to the particularities of the intended application (Hooton and Karkar 2012). In this research, the performance of four conventional rapid tests i.e. RCPT, SR, MRCPT and RCMT, for assessing concrete permeability against chloride penetration was studied. A total of 12 concrete mixtures were used and cement was replaced with silica fume in some of these mixtures.

2. Experimental program

2.1 Materials and mixture proportions

Twelve concrete mixtures were prepared and tested in the laboratory. ASTM C150 type I Portland cement was used for all concrete mixtures. The amounts of cement used in the mixtures were 350 and 400 kg/m^3. Silica fume (SF) was used as supplementary cementitious material with 7.5% and 15% replacement proportion. Chemical characteristics of the materials are shown in Table 1.

In all cases, crushed coarse aggregate with maximum size of 19mm and natural sand as fine aggregates were used. The coarse aggregates had a specific gravity and a water absorption of 2510 kg/m^3 and 1.90% respectively, and the fine aggregate has a water absorption of 2.75% and a specific gravity of 2570 kg/m^3.

Polycarboxylate Ether based superplasticizer (PCE-SP) with specific gravity of 1.05, solid content of 28% and pH of 6.6 was employed to achieve relatively constant workability. Potable water was used for casting the concrete samples, with water to binder ratios of 0.35 and 0.45. All

Table 2 Mixture proportion and properties of concretes

Mix	W/b	Silica Fume		Cement (kg/m^3)	Water (kg/m^3)	Aggregate (kg/m^3)		PCE-SP* (%)	Fresh Concrete		CS** 28 days (MPa)
		(%)	(kg/m^3)			Coarse	Fine		Slump (mm)	Temp (°C)	
4045SF0	0.45	0	0	400	180	692	1037	0.00	76	22	46.0
4035SF0	0.35	0	0	400	140	732	1099	0.34	89	26	62.3
3545SF0	0.45	0	0	350	157.5	731	1096	0.15	93	20	50.5
3535SF0	0.35	0	0	350	122.5	766	1150	0.55	100	24	65.8
4045SF7.5	0.45	7.5	30	370	180	692	1037	0.25	94	21	58.0
4035SF7.5	0.35	7.5	30	370	140	732	1099	0.60	80	25	78.0
3545SF7.5	0.45	7.5	26.25	323.75	157.5	731	1096	0.53	83	22	60.3
3535SF7.5	0.35	7.5	26.25	323.75	122.5	766	1150	0.80	105	23	78.0
4045SF15	0.45	15	60	340	180	692	1037	0.64	79	20	60.3
4035 SF15	0.35	15	60	340	140	732	1099	0.98	82	22	78.5
3545 SF15	0.45	15	52.5	297.5	157.5	731	1096	0.90	87	22	64.3
3535 SF15	0.35	15	52.5	297.5	122.5	766	1150	1.10	85	24	80.0

* Superplasticizer percentage is presented by Portland cement mass

** Compressive strength

replacements were made by mass. Details of the mixtures and properties of fresh concrete for each mixture are presented in Table 2.

2.2 Testing procedure and specimen preparation

The concrete production was carried out in a 60 l capacity mixer in accordance with ASTM C192. Prior to starting rotation of the mixer, coarse aggregate and mixing of some of the mixing water, SF and SP were added. SF and SP were dispersed in the mixing water before addition. Fine aggregate, cement and rest of mixing water were added with the mixer running.

All the 100×200 mm cylindrical specimens were cast in three layers. Each layer was consolidated on a vibrating table to reduce the air voids. The specimens were kept under a wet towel in the laboratory for 24 hours. After that, they were demolded and cured in lime-saturated water at 23 ± 2 °C until the test day to impede possible leaching of $Ca(OH)_2$ from these specimens.

2.2.1 Surface Electrical Resistivity (SR)

Electrical resistivity of concrete is an inherent attribute whose magnitude is contingent upon its ingredients and moisture (Monfore 1968, Garzon *et al*. 2014). Theoretical and empirical studies evince the existence of an underlying relation between electrical resistivity of concrete and the penetration of chloride ions (Andrade *et al*. 2014). In general, there is an inverse relation between permeability coefficient of chloride ions and the electrical resistivity of concrete (Polder *et al*. 2000). In a given structure, parts with higher susceptibility against penetration of chloride ions have comparatively lower electrical resistivity. Electrical resistivity is also related to the corrosion rate of bars after initiation of corrosion (Hornbostel *et al*. 2013). The flow of ions between anode and cathode is a controlling factor for the corrosion rate (Bockris and Reddy 2000).

One of the most practical methods for measuring the electrical resistivity of concrete is the Wenner four-point method, which is nondestructive, fast and simple (Gowers and Millard 1999). In this method, an array of four equi-distant electrodes is brought into contact with the surface of concrete.

It must be pointed out that the results of SR tests are affected by the chemical composition of the pore solution (Streicher and Alexander 1995). This can influence the results of applying these methods in studying concrete samples with supplementary cementitious materials, due to the low concentration of OH⁻ ions in their pore solution. Nonetheless, mainly due to the simplicity and speed of this test, it is suggested in standards, e.g. AASHTO TP 95 (AASHTO 2011).

In this study, at the age of 28 and 90 days, surface resistivity of three 100×200 mm cylindrical specimens were measured via a four-point Wenner array probe. The set up utilizes four equally spaced surface contacts, where a 40 Hz alternating current is passed through a concrete sample between the outer pair of contacts. The probe array spacing used was 50 mm. The resistivity measurements were taken at four longitudinal locations of the specimen, equally spaced around the circumference.

2.2.2 Rapid Chloride Permeability Test (RCPT)

The RCPT test has been standardized in ASTM and AASHTO and is widely used globally (Stanish *et al*. 1997). In this method, the measured quantity is the total amount of electric charge passed through saturated concrete samples of 10cm diameter and 5cm thickness within a period of 6 hours under 60 V potential difference. Concrete samples are in contact with NaCl solution on one side, with NaOH solution on the other, and by applying potential difference, chloride ions are transported inside the sample through the electric current. This method stands on the presumption that the amount of current that flows is related to the penetration of chloride ions in the pore structure of concrete.

Although RCPT has attained much attention due to its relative simplicity, there are concerns raised about it (Arup *et al*. 1993). In RCPT there is no steady state regime for penetration, i.e., to reach the steady state one has to wait long enough so that the chloride ions have reached the other end of the sample starting from the other, and the waiting time is unduly long, rendering it impractical. Thus in RCPT, measurements start at the outset of the experiment right away. Another pitfall associated with the RCPT method is the heat generated throughout the experiment (Julio-Betancourt and Hooton 2004). Also when pozzolans are used, the characteristics of the pore solution undergo changes that render the RCPT results unreliable (Ramezanianpour *et al*. 2011). Finally, since the applied voltage of 60V is relatively high, the induced current cannot reach the steady state, and the results may not accurately reflect the penetration intensity of chloride ions.

In this study, RCPT was carried out in accordance with ASTM C1202. At the age of 28 and 90 days, three 50 mm thick slices were cut from the middle part of 100×200 mm cylindrical specimens of each mixture. These disks were then conditioned and vacuum saturated and assembled in the test cell. Finally, direct current (DC) by a constant 60 V potential difference was applied to them for a six-hour period.

2.2.3 Rapid Chloride Migration Test (RCMT)

Among the rapid tests for chloride ion penetration that have proved reliable is Rapid Chloride Migration Test (RCMT) (Bagheri and Zanganeh 2012). The general scheme of this method, standardized in NT Build (Nordtest 1999) and AASHTO (AASHTO 2007), resembles that of RCPT. To avoid temperature increase during the experiment, the applied potential difference is
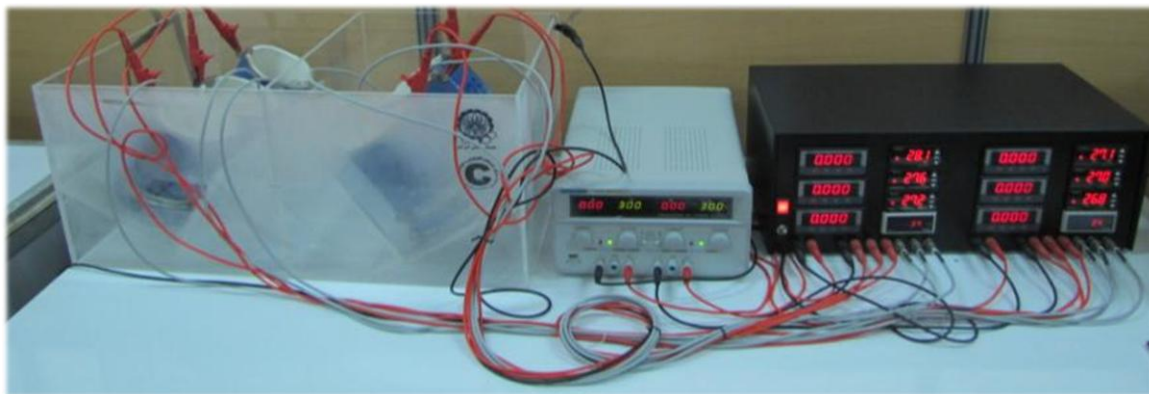
Fig. 1 RCMT set-up and apparatus used

adjusted according to the initial current. The volume of the NaCl solution which is contiguous to the concrete is taken to be large so that the variation in the concentration of chloride ions will be negligible. In this method, to prevent the effects of other ions such as OH⁻ on the results, the penetration depth of chloride ions is measured directly by halving the sample and spraying 0.1 M silver nitrate solution on the freshly split section. However, this method may not eliminate the influence of the pore solution conductivity.

This method was applied to all samples according to NT Build 492 (Nordtest 1999) for each age of the experiment. The preparation of samples was identical to that described for RCPT, with the only distinction being that lime-saturated water was used instead of distilled water.

The conditioned specimens are placed in the rubber ring and the clamps tightened. A 0.3 molar NaOH solution is poured into the rubber ring to cover the upper face of the specimen. The assembly is then placed on the special stand inside the plastic box containing 10% solution of NaCl as shown in Fig. 1.

Then, via the two electrodes situated at the opposing ends of the sample, a potential difference is applied proportional to the initial current, which migrates the chloride ions into the sample.

2.2.4 Modified Rapid Chloride Permeability Test (MRCPT)

Another rapid test for assessing the permeability of concrete against chloride ions is the MRCP test, designed at AmirKabir Univerity (Pilvar *et al.* 2015), whose motivating rationale was to remedy the shortcomings concomitant with the RCPT test. To prepare the samples in this method, they are first dried in the oven at $50\,^{\circ}C$ for 7 days, and then saturated using a 23% NaCl solution. This approach firstly devised by Streicher and Alexander (Streicher and Alexander 1995). In high concentrations of NaCl solution (23%), conductivity is less sensitive to small variations in concentration (Pilvar *et al.* 2015).

MRCPT relies on measuring the conduction of concrete which depends on the intrinsic microstructure and amount of charge carriers in the pore solution, mostly the hydroxyl ions. Therefore, by saturating the concrete specimens with highly conductive solution, the variability of pore water becomes less significant. Different concrete specimens would yield different conductivities because of the differences in their pore structure.

A limitation in the MRCP test stems from slicing smaller size specimens of 100 mm diameter and 25 mm thickness which results in disturbance in the microstructure. Nevertheless, this is

regarded as only surface effect. However, concrete samples with small thicknesses cannot accurately emulate real concrete with coarse aggregate. Nonetheless, MRCPT has several advantages over RCPT, one of which is that the current is at the steady state from the inception. The reading of current is observed quickly as the chloride ion flux and no measurements are required at the downstream reservoir.

In this research, 3 cylindrical samples with 10 cm of diameter and 2.5 cm of thickness are prepared and are placed inside cells, similar to the RCPT method at the ages of 28 and 90 days. Then potential difference of 10V was applied and the flowed current was measured after 1 minute. The conductivity of the samples was calculated using Equation (1), and the results were reported accordingly.

$$\sigma = (i/v) * (t/A) \tag{1}$$

Where σ is the conductivity of the sample [mS/cm], i is the electric current [mA], v is the potential difference [V], t is the thickness of the sample [cm], and A is the cross section area of the sample [cm^2].

3. Results and discussion

3.1 Surface Electrical Resistivity (SR)

The electrical resistivity of concrete depends upon the microstructure of the hardened paste and the chemical compounds of the pore solution (Xia and Li 2013). Electrical resistivity is actually an indicator of the mobility of ions throughout the concrete matrix. It can thus be a plausible merit for the assessment of the permeability of concrete against chloride ions (Torres-Luque *et al.* 2014). On the other hand, through affecting the mobility of ions between cathode and anode regions, the resistivity of the concrete has a key role at the surface of the regions of corrosion in the bars (Yu *et al.* 2014).

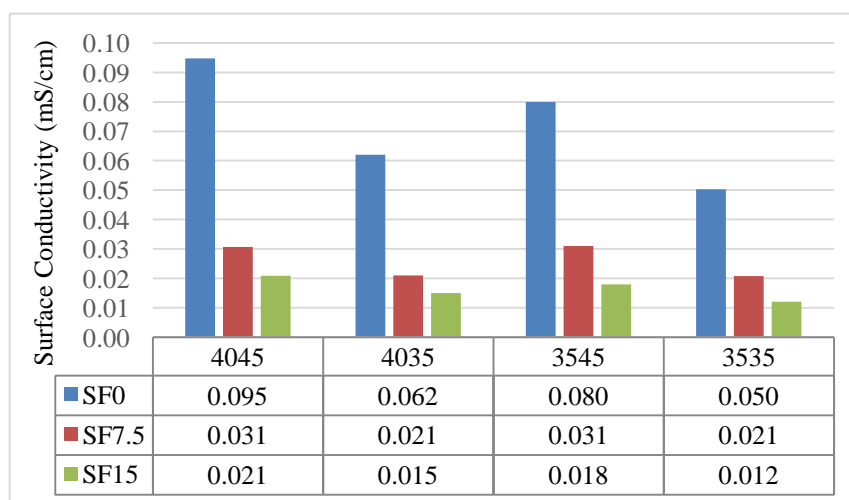|	4045	4035	3545	3535
SF0	0.095	0.062	0.080	0.050
SF7.5	0.031	0.021	0.031	0.021
SF15	0.021	0.015	0.018	0.012

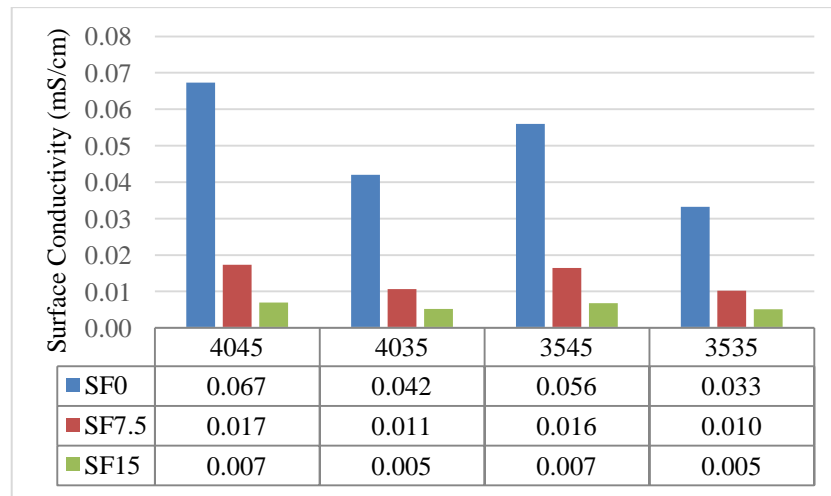Fig. 2 Results of conductivity (inverse of SR), 28 days

Fig. 3 Results of conductivity (inverse of SR), 90 days

To provide convenience of comparison between the results obtained from the surface resistivity test with those of other tests, Figs. 2 and 3 present the conductivity of the samples, which is numerically the inverse of resistivity.

As one would expect, in all samples, for equal amount of cementitious material, the reduction in the water-to-cement ratio and consequently, the reduction of capillary pores and the densification of paste, decreases the conductivity and permeability of concrete.

At a constant water-to-cement ratio, in all samples reduction in the cement content leads to a decrease in conductivity. It can be justified by noting that reducing the cement content, reduces the volume of paste in the concrete as compared to aggregate volume. Furthermore, aggregates act as insulators in the structure of concrete; hence as the ratio of aggregate phase of concrete to paste increases, conductivity diminishes.

At constant water to binder ratio and cement content, replacing cement with silica fume remarkably decreases the conductivity of concrete samples. For instance, in "4045" mixture, 15% replacement of silica fume with ordinary Portland cement decreased the conductivity of concrete samples by 77% and 89% at the ages of 28, and 90 days, respectively. This reduction can be justified by noting that pozzolanic reactions proliferate the tortuosity in the structure of capillary pores and further densifies the pore structure. Furthermore, as the concentration of OH^- ions diminishes in most of these reactions, the conductivity of the pore solution diminishes substantially. It is worth mentioning that OH^- ions have the key role in the conductivity of the pore solution (Bu and Weiss 2014). As a result of these two phenomena, as well as dilution, the conductivity of concrete substantially decreases.

3.2 Rapid Chloride Permeability Test (RCPT)

In this test, the electric charge passed through the sample is measured during the experiment as a determinant of the chloride penetrability of concrete. The results of the RCPT for all samples are presented in Figs. 4 and 5. The overall trend observed in the results is similar to that of the SR.

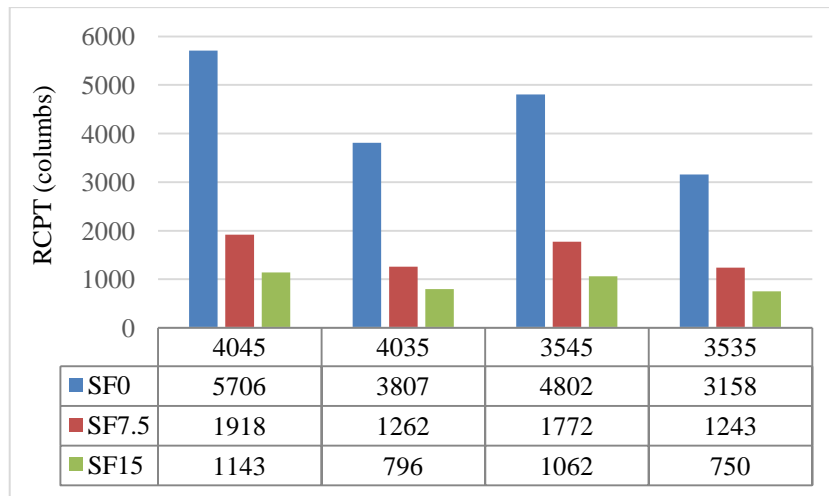The results illustrate that replacing cement with silica fume reduces the permeability of

	4045	4035	3545	3535
SF0	5706	3807	4802	3158
SF7.5	1918	1262	1772	1243
SF15	1143	796	1062	750

Fig. 4 Results of RCPT, 28 days

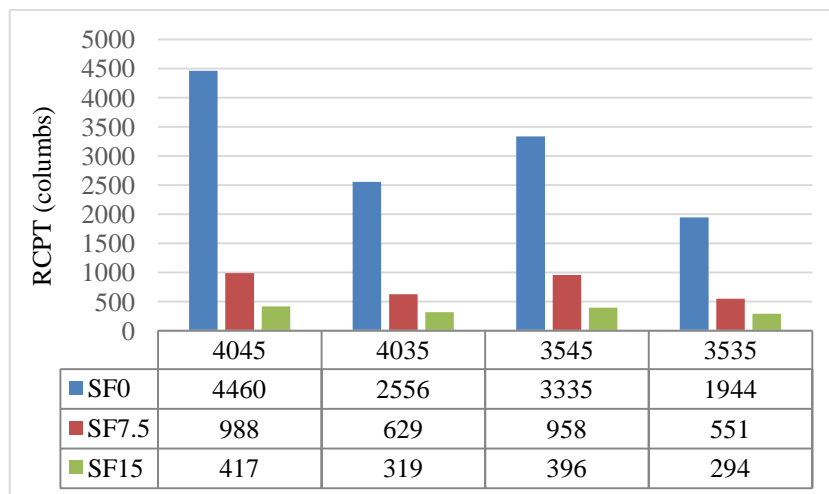|	4045	4035	3545	3535
SF0	4460	2556	3335	1944
SF7.5	988	629	958	551
SF15	417	319	396	294

Fig. 5 Results of RCPT, 90 days

concrete. Replacement of cement with silica fume results in a 90% reduction in the determinant of the total passed charge in "4045" mixture at the age of 90 days. This reduction is more than that of the SR test, while theoretically they should coincide (the total charge equals the area under the current-time curve). The discrepancy arises from the deficiency of RCPT. In RCPT, the current passing through the samples generates heat which in turn increases the current. Consequently, the difference between the total charge in samples with silica fume and the control samples is greater than that one would expect to observe solely due to the difference in their conductivity.

3.3 Rapid Chloride Migration Test (RCMT)

In contrast to RCPT and SR methods, in RCMT the penetration depth of chloride ions is
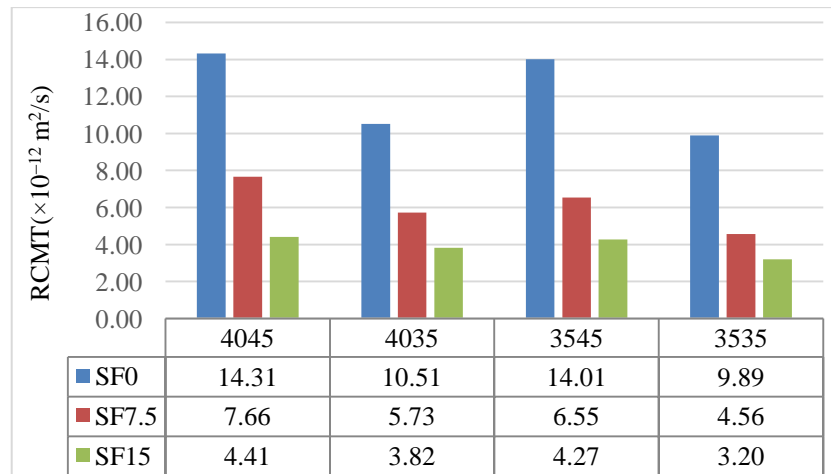
	4045	4035	3545	3535
SF0	14.31	10.51	14.01	9.89
SF7.5	7.66	5.73	6.55	4.56
SF15	4.41	3.82	4.27	3.20

Fig. 6 Results of RCMT, 28 days

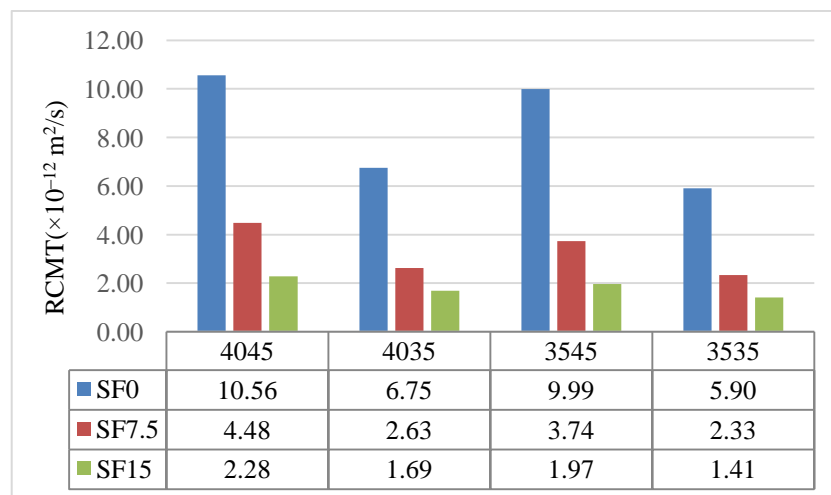|	4045	4035	3545	3535
SF0	10.56	6.75	9.99	5.90
SF7.5	4.48	2.63	3.74	2.33
SF15	2.28	1.69	1.97	1.41

Fig. 7 Results of RCMT, 90 days

measured directly. This is suggestive of the superiority of this method in the assessment of the chloride permeability of concrete, especially when supplementary cementitious materials such as pozzolans are utilized (Madani *et al*. 2014). According to the electro-chemical relation posited in NT Build, the diffusion coefficient of the chloride ions is obtained from the applied potential difference, time, average temperature and penetration depth. The results of this experiment are depicted in Figs. 6 and 7.

As can be observed, the overall trend of the results is in good agreement with those of SR and RCP tests. Although in this method, the difference induced by applying silica fume as compared to control samples is smaller than that of SR and RCPT methods, since this method is less sensitive to variations in the conductivity of the pore solution. The maximum reduction in the diffusion coefficient resulted by applying silica fume, corresponds to "3545" mixture, is 69% and 80% at the ages of 28 and 90 days, respectively.
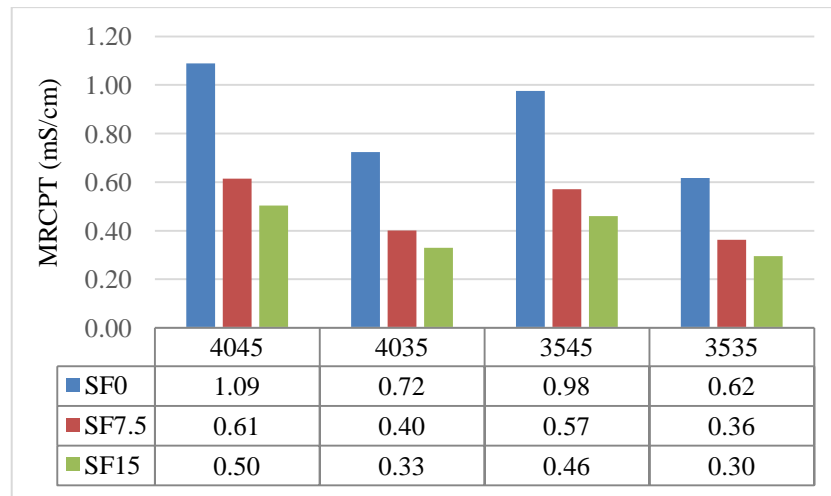
	4045	4035	3545	3535
■ SF0	1.09	0.72	0.98	0.62
■ SF7.5	0.61	0.40	0.57	0.36
■ SF15	0.50	0.33	0.46	0.30

Fig. 8 Results of MRCPT, 28 days

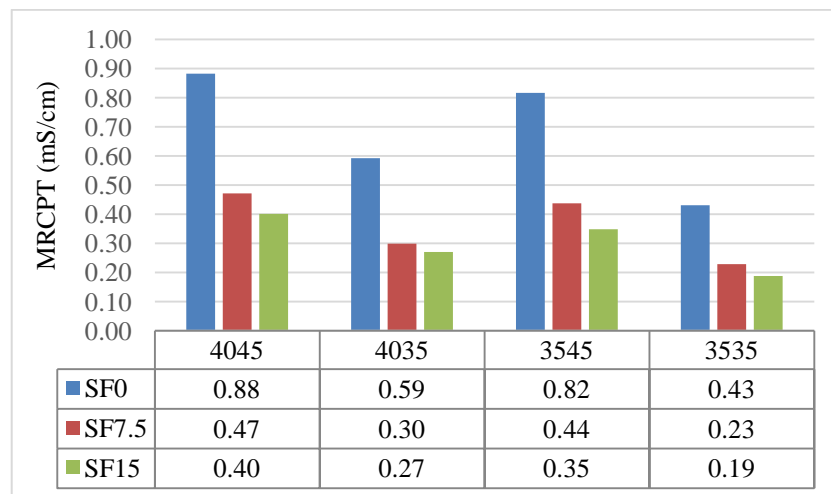|	4045	4035	3545	3535
■ SF0	0.88	0.59	0.82	0.43
■ SF7.5	0.47	0.30	0.44	0.23
■ SF15	0.40	0.27	0.35	0.19

Fig. 9 Results of MRCPT, 91 days

3.4 Modified Rapid Chloride Permeability Test (MRCPT)

In this method, it is claimed that by saturating concrete samples with NaCl solutions having high conductivity, the effect of the conductivity of the pore solution on the results diminishes significantly, and the alteration of the results induced by the variations in the microstructure of capillary pores. Figs. 8 and 9 depict the results of MRCPT for all samples.

It can be observed that applying silica fume reduces the conductivity of the samples, however, this reduction is smaller than that observed in the other three tests. The reduction between the differences of the results of the experiments can be ascribed to the assumption of full saturation of the samples with 23% NaCl solution and the uniformity of the conductivity of the pore solutions of the concrete samples under experiment. This corroborates the claim that this method is less

sensitive towards the variations in the conductivity of the pore solution in different concretes. The maximum reduction observed in the conductivity of concrete samples by applying silica fume is 57% for the "3545" mixture at the age of 90 days. Results of the MRCPT can also be analyzed in terms of the effect of the cement content and water-to-cement ratio, in which the observations and discussions would be similar to those presented for the SR test method.

4. Conclusions

In this research, the performances of four rapid tests for assessing the chloride permeability of concrete were studied. The following conclusions are drawn from the results of this research work: Results of the four tests illustrate that applying silica fume reduces the chloride permeability of concrete. Pozzolanic reactions of silica fume result in the densification of the micro-structure of concrete as well as blocking its capillary pores.

• Due to the consumption of OH^- ions that ensue in the pozzolanic reactions of the silica fume and consequently the substantial reduction in the conductivity of the pore solution, the penetrability of concrete samples incorporating silica fume exhibit a substantial reduction when compared to control samples for SR and RCPT methods. This hints at a shortcoming of these methods in assessing the permeability of concretes with active pozzolans.

• The reduction in the samples with silica fume as compared to control samples is smaller in the RCMT method than those observed for SR and RCPT methods. This can be attributed to the direct measurement of the penetration depth of chloride ions, as well as saturation of the concrete samples with lime-saturated water.

• Among the four methods that were studied, the MRCPT method has the least sensitivity to the variations in the conductivity of the pore solution. It can be contended that MRCPT and RCMT offer more transparent account on the performance and permeability of concretes with silica fume. It can be construed that these methods prove reliable in assessing the concretes with supplementary cementitious materials.

All the four methods captured the effect of water-to-cement ratio and cement content on the permeability of concrete almost equally well, and exhibited comparable performances.

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