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Influence of Zeolite Additive on Chloride Durability and Carbonation of Concretes

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A B S T R A C T

Replacement of cement with pozzolan in the production of concrete not only improves the mechanical properties and durability of concrete but also decreases the amount of consumed cement in construction projects and causes economic and environmental advantages by reducing CO_2 emissions. Zeolite is a natural pozzolanic material and has been found abundantly in nature especially in Iran. In this work, the mechanical and durability properties of concretes containing various amounts of zeolite at various water/binder ratios were investigated. Experimental tests include compressive strength, water permeability, water absorption, electrical resistivity, rapid chloride permeability and carbonation test at different ages.Generally, the results show that zeolite decreases water absorption and water permeability and chloride permeability and increases compressive strength and electrical resistivity compared with that of the control concrete, so it improves chloride durability. However, the usage of zeolite may increase the carbonation depth.

1. Introduction

Concrete is one of the most widely used construction materials, owing to its good durability to cost ratio. The property of cement used in concrete production is the most important factor on concrete performance (Karakurt and Topçu, 2011).

Cement has 1.6 billion tons production annually, so it is the most produced and used binding material in the world (Worrell et al., 2000).Its production consumes large amounts of energy and causes high emission which has negative effect on the environment (Ghrici et al., 2006). 7% of the total CO_2 emission related to the cement industry (Mehta, 2002). Thus, the cement industry has a crucial role in global warming.

Natural and artificial pozzolans, which are materials exhibiting cementitious properties when reacting with calcium hydroxide in the presence of water, have been widely used as partial replacement for Portland cement in blended cements and concrete in many applications due to energy-saving concerns and some environmental considerations (Ramezanianpour at al., 2009a; Rahmani and Ramezanianpour, 2008). The substitution of Portland cement by pozzolana in concrete can decrease sorptivity, porosity and permeability and increase chemical resistance and improve long-term durability (Massazza, 2003; Rodriguez-Camacho and Uribe-Afif, 2002; Papadakis and Tsimas, 2002).

Zeolites derived from either natural or artificial sources are a three-dimensional framework structure containing crystalline aluminosilicates based on repeated units of silicon±oxygen (SiO4) andaluminium±oxygen (AlO4) tetrahedral which are being used as blend materials in concretes (Lea, 1970; Poon et al., 1999).Crystals are a cage like structure with extremely small pores and channels (Ahmadi and Shekarchi, 2010). They have unique characteristics such as large (internal and external) specific surface areas, cation exchange capacity and the ability to lose and gain water(Ahmadi and Shekarchi,

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2010; Colella at al., 2001). Two first characteristics, the ability of ion exchange and high surface area in zeolite pastes lead to the pozzolanic reaction (Perraki at al., 2010; Uzal et al., 2010). Pozzolanic activities of other pozzolanic materials derive from their non-crystalline silica which is the primary phase of these materials (Uzal et al., 2010). They use in many fields such as pollution management, water and air filtration, energy, agriculture, soil amendment, animal hygiene, stock farming and mine metallurgy due to their physical structure and chemical properties (Kurudirek et al., 2010).

The pozzolanic activity of zeolites depends on their chemical and mineralogical composition (Karakurt and Topçu, 2011). When the reactive SiO_2 and Al_2O_3 in zeolite combines with the calcium hydroxide produced by the hydration of cement in concrete , form additional C–S–H gel and aluminates, that improve the microstructure of hardened cement (Poon et al., 1999; Ortega et al., 2000; Caputo at al., 2008).Studies was done by other researchers show that in general, the natural zeolite can improve the compressive strength of concrete and the mechanical properties of cement and concrete composites (Canpolat et al., 2004; Yılmaz, 2007; Feng et al., 1990).Natural zeolite also prevents the chloride penetration , sulfate attack and undesirable expansion due to alkali silica reaction (Janotka and Stevula, 1998; Feng and Hao, 1998; Feng at al., 1992; Niu and Feng, 2005; Ahmadi and Shekarchi, 2010).Only one study was carried out related to carbonation but due to the condition and unexpected results, it is necessary to carry out more experimental tests in this case (Cahit, 2011). Zeolites need more water to produce paste of the same consistency, so superplasticizer has to be added to control the slump (Fragoulis et al. 1997; Niu and Feng, 2005). Because the viscosity of fresh concrete increases with the added zeolite, it can prevent bleeding and segregation in the flowing fresh concrete. However the effects of zeolite minerals on properties of cement are not clearly known.

The objective of this study was to evaluate the effect of clinoptilolite, which is one of the most common zeolite minerals found in nature, on mechanical properties and the durability of concrete using various testing methods. The effects of varying water to total cementitious material ratios on the performance of control concrete and concretes incorporating varying amounts of zeolite at different ages were investigated. For first time, the accelerated carbonation test was done in concrete containing zeolite.

2. Materials

These materials used in this study were as follows:

a) Cement: type 1-425 Ordinary Portland cement (OPC).

b) Zeolite: clinoptilolite type from Abgarm mine southeast of Semnan, Iran.

c) Coarse aggregate: maximum size 19 mm from the Karaj River region .The course aggregates have specific gravity and water absorption of 2540 kg/m^3 and 1.90%, respectively.

d) Fine aggregate: sand in grading zone between A and C in accordance with IRAN standards. The fine aggregate has water absorption of 2.3% and a specific gravity of 2530 kg/m^3.

e) Superplasticizer: polycarboxilate based on a gravity of 1.1 For the purpose of producing high strength and sufficient workability concrete.

The chemical compositions and physical properties of the ordinary Portland cement and zeolite used in the experiments are summarized in Tables 1 and2.

3. Mixture Proportion

Zeolite was in turn used to replace 0%, 10%, 15% by weight of cement. The coarse aggregate content was 40% and fine aggregate 60% of all aggregate while the 19 mm sized coarse aggregates were maximum.The water to total cementations materials ratio W/(C + P) was 0.35, 0.4, 0.45, and 0.5 having a constant total binder (cement + zeolite) content of 350 kg/m^3 for this study. Superplasticizer was added to attain a slump of about 70 to 100 mm.The mixture proportions are given in Table 3.

Table 1: Physical properties in clinoptilolite

Material	**Specific gravity (g/cm^3)**	**Specific surface area (cm^2/g)**	**Remained on sieve (%)**	
			45 µm	90 µm
Clinoptilolite	1.91	10000	0	0
Cement	3.15	3060	20	4.2

Table 2: Chemical composition of cement and natural zeolite

Compound/ Property	Cement	Natural zeolite
Calcium oxide (CaO)	63.00%	1.68%
Silica (SiO_2)	21.95%	67.79 %
Alumina (Al_2O_3)	4.35%	13.66 %
Iron oxide (Fe_2O_3)	3.80%	1.44 %
Magnesium oxide (MgO)	2.00%	1.2 %
Sodium oxide (Na_2O)	0.30%	2.04 %
Potassium oxide (K_2O)	0.77%	1.42 %
Sulfur trioxide (SO_3)	2.43	0.52

Table 3: Mix proportions of concrete

Code	w/c	Zeolite%	w	Coarse aggregate	Fineaggregate	sp%
A 0	0.35	0	122.5	752	1126	0.90
A 10	0.35	10	122.5	752	1126	1.20
A 15	0.35	15	122.5	752	1126	1.30
B 0	0.4	0	140	720	1080	0.45
B 10	0.4	10	140	720	1080	0.80
B 15	0.4	15	140	720	1080	0.95
C 0	0.45	0	157.5	702	1054	0.30
C 10	0.45	10	157.5	702	1054	0.55
C 15	0.45	15	157.5	702	1054	0.65
D 0	0.5	0	175	687	1028	0.00
D 10	0.5	10	175	687	1028	0.20
D 15	0.5	15	175	687	1028	0.35

4. Specimen Preparation, Curing and Testing

4.1 Mixing Procedure

The dry aggregates of concrete were first mixed followed by the addition of 1/3 water.Then the mixture of cement and zeolite was added followed by addition of another 1/3 of water. Superplasticizer was added at the last stage of mixing with the rest of the water. The total mixing time was about 5 min.

4.2 Casting and Curing of Specimens

Concrete specimens were demoded 24 hours after casting, and then placed immediately in a water curing tank. The temperature of water was maintained at 25±2 °C.

Compressive strength at 7, 28, 90, 180, 270 and 300 days of age, three 100mm cube specimens of each concrete mixture were tested for compressive strength.

Water permeability at 28, and 90 days of age, two 150 mm cubic specimens of each concrete mixture were tested for water permeability.In this test, water was forced into the concrete samples from one side for three days and under constant pressures of 0.5 MPa. Then, the samples were split in a plane parallel to the direction of water penetration, and the greatest and moderate depth of water penetration into the concrete sample were measured.
Water absorption at 28, 90 and 270 days of age, two 100 mm cubic specimens of each concrete mixture were tested for water absorption. Specimens were dried in a 50˚C oven for 14 days. The specimen were rested on rods to allow free access of water to the surface and the water level was maintained at 5 ± 1 mm above the base of specimens. The absorption, a_i , was calculated as the change in mass divided by the cross-sectional area of the test specimen after 0, 3, 6, 24 and 72 hours of absorption.

$$a_i = \frac{Mi-M0}{A} \qquad (1)$$

The initial rate of water absorption ($h^{0.5}$x g/cm^2) is defined as the slope of the line that is the best fit to $a_i$plotted against the square root of time ($s^{1/2}$).

The absorption coefficient (S) according to ASTM C1585 was obtained using the following expression:

$$a_i = c + s\sqrt{t} \qquad (2)$$

Electrical resistivity testing was conducted on two 100 x 200 mm cylinders prepared for every concrete mixture at 28, 90, 270 and 300 days after the casting of concretes.

The specimens were submerged in water until the testing age. The four-point electrical resistivity measurement device (Wenner array probe) was used to measure the electrical resistivity of concrete for analyzing the corrosion potential and offers an indication of its permeability. The resistivity measurements were taken at four quaternary longitudinal locations of the specimen.

The Rapid Chloride Permeability Test was conducted in accordance with ASTM C1202 for each mixture. Two specimens of 100 mm in diameter and 50 mm in thickness which had been conditioned were subjected to a 60-V potential for 6 hours. The total charge passed through the concrete specimens was determined and used to evaluate the chloride permeability of each concrete mixtureThe ages of specimens for the tests were 28,90 and 270 days.

Three 50-mm cube specimens of each concrete mixture were tested for carbonation. For this test, fresh concrete mixture was passed through a number 4 sieve to obtain mortar. Specimens were tested in accelerated conditions. They were put in a tank and CO_2 was passed through. After 24 hours, the carbonation depth of the specimens was measured by spraying a 1% phenolphthalein solution on freshly cut surfaces.

5. Results

As can be seen in Table 3, at a high cement replacement levels by zeolite, in order to maintain the similar slump to the control concrete and have the same workability, a higher dosage of superplasticizer was necessary for the concrete with pozzolanic materials especially in low w/c ratios .This can be attributed to the large amount of pores in its frame structure and high surface area (Babak Ahmadi, 2010).Similar results were obtained for the properties of fresh concretes incorporating natural zeoliteby other authors(Ahmadi and Shekarchi, 2010; Chan and Ji, 1999).

5.1. Compressive strength

Figure 1 shows the effect of zeolite on the compressive strength of concrete specimens with varying w/c ratios. As expected, the compressive strength of all concrete specimens increased with the time of curing and decreased with increasing the w/c ratio. It is clear that regardless of replacement level, zeolite decreased concrete strength at 7 days. This reduction was about 6% for 10% replacement and around 9% for 15% replacement. The decrease of strength before 7 days is attributed to the dilution effect (Perraki at al., 2010).

Compressive strength increased at 28 days due to the pozzolanic reactions (Perraki at al., 2010).It is apparent that the optimum replacement level was about 10%, although it did not show considerable difference at 15% replacement. As can be seen in Table 4, for example, the 28-day strengths of zeolite concrete at 10% cement replacement level for C series was 43.17 MPa,12% higher than that of control concrete. The results show that it was possible to obtain a compressive strength as high as 60 MPa after 28 days. Generally, the replacement of cement by zeolite at different levels increased the 28-day compressive strength of concrete about 7 to 12%.In addition, after 28 days, when the cement replacement level by zeolite was 10%, the strength of zeolite specimens were similar to the control concrete and when the cement replacement level by zeolite increases up to 15%, the concrete compressive strength was found to have decreased to a level lower than that of the control concrete. However, the decrease was less than 10% which was not significant.

As a case in point, compressive strength for the B15 mixture after 270 days was 7% lower than the B0 mixture.Between 90 and 300 days, this slight decrease of strength is due to the reduction of the pozzolanic reaction. However, this can be attributed to the experimental errors.The maximum compressive strength in concrete specimens at 300 days was 73.25 MPa corresponding to the A10 mixture and the minimum compressive strength was 50.0MPa corresponding to the D15 mixture.

5.2 Water permeability

One of the most important parameters for evaluating the durability of concrete is water permeability. Table 5 summarizes the results of the water penetration depths in all concrete mixtures. As can be seen, the zeolite concretes provide lower water penetration depth than control concrete.This issue is related to the filler effect, pozzolanic reaction, heterogeneous nucleationand improvement in the pore structure of concrete(Ramezanianpour et al., 2009b). As expected, the depth of water penetration of concrete specimens increased significantly with an increase in w/c ratio due to an increase in porosity.

The maximum depth of water penetration is 22.5 mm for the D0 mixture after 28 days and the minimum was 6.2 mm for the A10 mixture. After 90 days, the maximum depth of water penetration was 14.3 mm for the D0 mixture and the minimum was 6.7 mm for the A10 mixture. In addition to specimens with high w/c ratio (0.45 and 0.5), the depth of penetration decreased by increasing the curing time, because they were more porous and needed more time to fill these pores and disconnect capillary porosity, but specimens with lower w/c ratio (0.4 and 0.35) became dense in the early ages, so their depth of penetration were rarely constant with time.

The BS EN 12390-8:2000 standard advises measuring the maximum depth of penetration, but as seen in Table 5, due to some errors in construction, the preparation or curing of specimens and other unexpected conditions, maximum depth measurement cannot be a real criterion for compareconcretes, especially when the effect of new material is considered. Thus, in this study, both maximum and average depth of water penetration was measured.

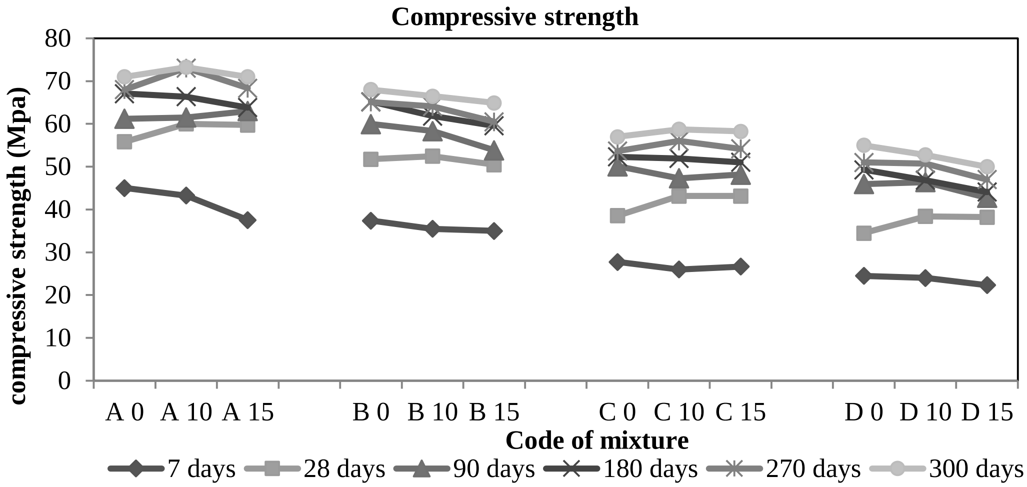

Figure 1: Compressive strength of control and zeolite concretes at different ages with various w/c ratios.

Table 4: Compressive strength of control and zeolite concretes at different ages with various w/c ratios

Number	w/c	Z%	Fc (7 days)	Fc (28 days)	Fc (90 days)	Fc (180 days)	Fc (270 days)	Fc (300 days)
A 0	0.35	0	45.03	55.83	61.23	67.10	68.00	71.00
A 10	0.35	10	43.27	60.00	61.50	66.37	73.07	73.25
A 15	0.35	15	37.55	59.75	63.00	63.85	68.35	71.00
B 0	0.4	0	37.40	51.77	59.97	65.23	65.10	68.00
B 10	0.4	10	35.50	52.47	58.33	61.83	64.10	66.50
B 15	0.4	15	35.00	50.50	53.83	59.57	60.50	64.90
C 0	0.45	0	27.75	38.57	50.10	52.33	53.67	57.00
C 10	0.45	10	26.00	43.17	47.30	51.93	56.00	58.75
C 15	0.45	15	26.67	43.17	48.15	51.03	54.20	58.25
D 0	0.5	0	24.50	34.50	45.97	49.30	51.00	55.00
D 10	0.5	10	24.04	38.40	46.40	46.87	50.75	52.75
D 15	0.5	15	22.33	38.23	42.77	44.10	47.00	50.00

Table 5: The effect of zeolite on the water penetration depth (cm) at various ages

Number	28 day penetration depth		90 day penetration depth	
	Max	Moderate	Max	Moderate
A 0	0.98	0.44	1.26	0.67
A 10	0.62	0.39	0.67	0.43
A 15	0.76	0.46	1.10	0.45
B 0	1.52	0.61	1.19	0.67
B 10	1.00	0.45	1.30	0.60
B 15	0.72	0.38	1.16	0.56
C 0	1.70	0.83	1.02	0.67
C 10	1.43	0.60	1.10	0.53
C 15	1.52	0.62	0.73	0.43
D 0	2.25	1.37	1.43	0.70
D 10	1.61	0.82	1.15	0.65
D 15	1.26	0.54	1.41	0.55

5.3 Water absorption

Table 6 shows the influence of the zeolite on the water absorption of concretes with various w/c ratios at the age of 28, 90 and 270 days. It is clear that reduction in the w/c ratio decreased the water absorption. At all times, the absorption decreased with an increase in zeolite replacement. This is related to pozzolanic reaction and improvement in pore structures and disconnection in capillary pores of concrete.

For example, as seen in Figure 2 after 28 days, the absorption coefficient for the A10 mixturewas 55%lower than that of A0.

5.4 Electrical resistivity

As shown in Figure 3, the electrical resistivity of specimens considerably increased with time. It is clear that mixtures containing zeolite had higher resistivity than control mixtures at various w/c ratios. The resistivity of concrete increased by increasing the zeolite content up to 15%.

Concrete resistivity depends both on the microstructure properties of the concrete and the conductivity of the pore solution (Ramezanianpour et al., 2011).(Shi et al., 1998) showed that the presence of silica fume can alter the composition of the concrete pore solution to the extent that the electrical conductivity of the concrete may be reduced by 90% compare to conventional mixtures with Portland cement alone. Thus, in this study, the same results were observed with zeolite. As can be seen, the electrical resistivity of concrete samples containing zeolite increased 2 to 4 times compared to control samples.

According to the recommendation from FM 5-578, the corrosion rate of reinforcing steel is lower when the resistivity of concrete exceeds 20 kΩcm (Flo).All the zeolite mixtures had resistivity higher than 20 kΩcm. The highest value of electrical resistivity was 99 kΩcm for the A15 mixture after 300 days and the minimum was 10.0 kΩcm for the D0 mixture.

5.5 Chloride permeability

As presented in Figure 4, zeolite considerably decreased the charge passing from specimens and increased the resistivity of concrete against chloride ion permeability at various ages. This was observed mostly in mixtures containing high w/c ratios. As an example, 15% zeolite replacement decreased the chloride permeability ion of mixtures (w/c=0.5) about 59.4 % at 28 days and 70.3 % at 90 days. Similar to the electrical resistivity results, this reduction is related to both pozzolanic reaction and conductivity of the pore solution in concretes containing natural zeolite.

5.6 Carbonation

Figure 5 shows the carbonation depth of specimens .The depth of carbonation increased by increasing the w/c ratio and zeolite replacement. Pozzolanic materials consume the calcium hydroxide of concrete due to pozzolanic reaction. When these materials consume a high amount of $Ca(OH)_2$, the depth of carbonation increases. Thus, this phenomenon was observed in concrete containing natural zeolite .The depth of carbonation also decreased by increasing the curing time due to completing hydration reactions and improving pore structures. (Cahit, 2011) presented another results and said that zeolite decreased the carbonation depth, but it seems to be not true .Because specimens cannot be carbonated in primary ages at normal conditions. Moreover, carbonation test may have errors up to 2 mm and this should not be shown as differences between test results. So their test results were unexpected.

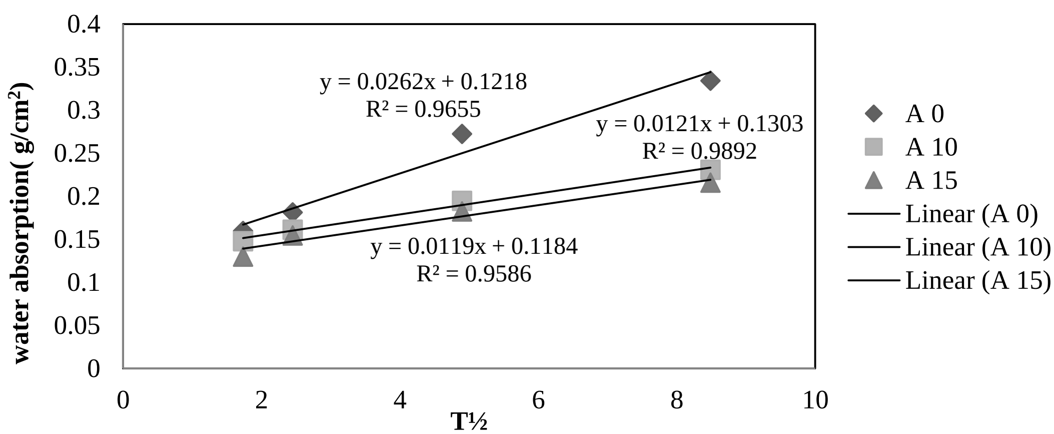

Figure 2: Absorption coefficient for series A at 28 days

Table 6: The effect of zeolite on the water absorption coefficient($h^{0.5}$x g/cm^2) at various ages.

Number	Age of Testing		
	28 days	90 days	270 days
A 0	0.262	0.313	0.537
A 10	0.121	0.128	0.327
A 15	0.119	0.184	0.301
B 0	0.417	0.204	0.336
B 10	0.194	0.104	0.305
B 15	0.219	0.097	0.260
C 0	0.452	0.278	0.549
C 10	0.188	0.266	0.468
C 15	0.144	0.155	0.346
D 0	0.407	0.664	0.807
D 10	0.163	0.673	0.694
D 15	0.271	0.428	0.630

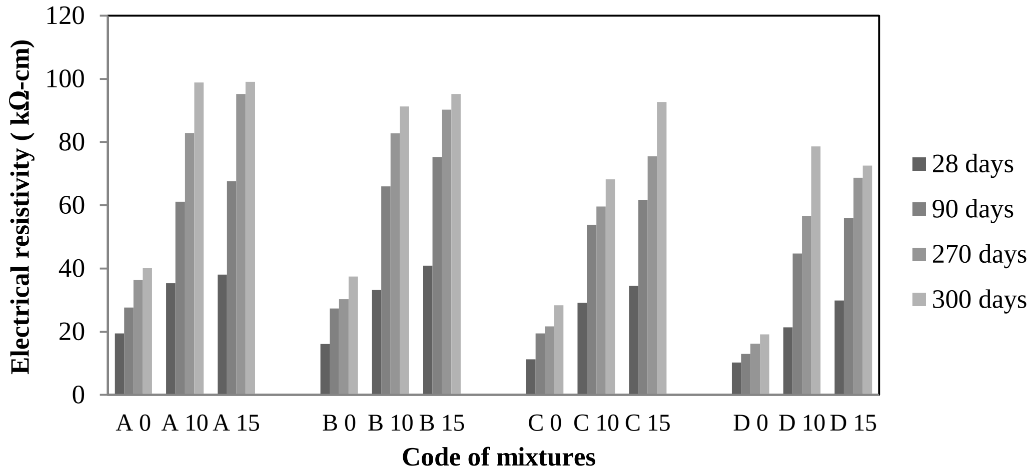

Figure 3: Electrical resistivity of control and zeolite concretes at different ages with various w/c ratios.

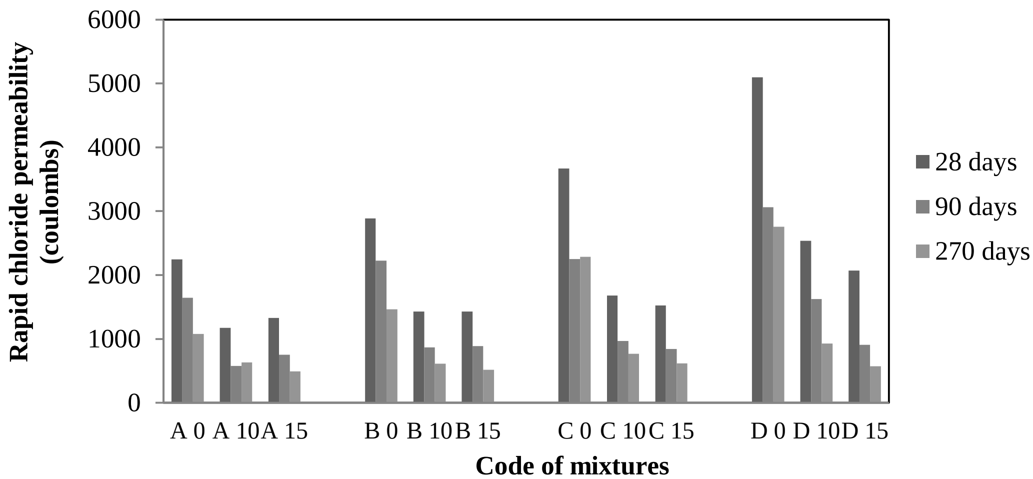

Figure 4: Result of RCPT test of control and zeolite concretes at different ages with various w/c ratios

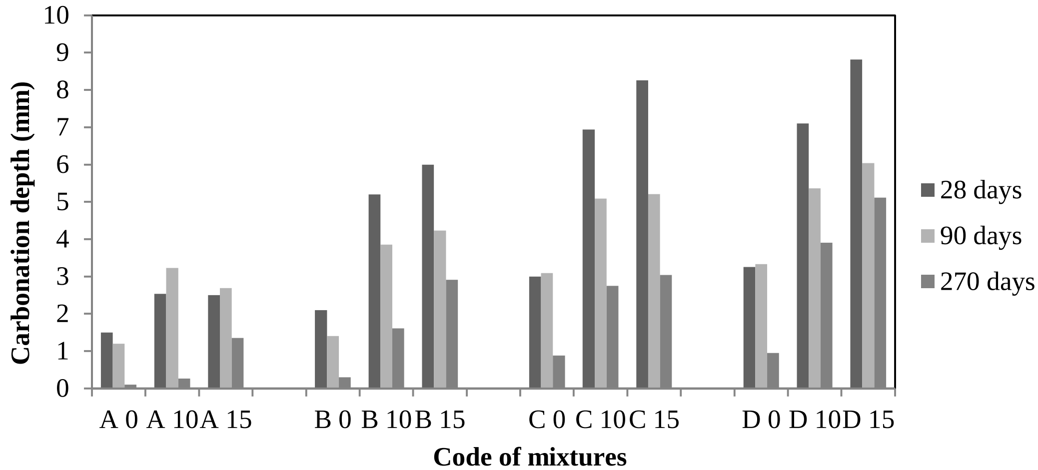

Figure 5: Depth of carbonation in various ages

5.7 Relationships

There are several factors and test methods for evaluating the durability of concrete. In recent years, great attention has been paid to research regarding the relationships of these parameters for concrete because with these relations, it is possible to predict various properties of concrete from one test (Ramezanianpour et al., 2011).On the other hand, results of different experimental test methods can be confirmed by one another. So in this study, the relationships between the results of some experimental tests were examined.

As seen in Figure 6, there was no significant correlation between compressive strength and concrete resistivity (R^2=0.2502) when concrete mixtures were made with various cementitious materials at various ages. However, in the case of similar cementitious materials at same ages (Figures 7 and 8), better correlations (R^2=0.84, 0.96 and 0.94) was be observed between compressive strength and concrete resistivity. Similar results were obtained by other researchers (Ramezanianpour et al., 2011).

One of the main factors in compressive strength is the strength of Interlayer Transition Zone (ITZ) that has no significant effect on concrete resistivity. On the other hand, chemical compounds in solution have a great influence on concrete resistivity while they don't affect compressive strength of concrete (Ramezanianpour et al., 2011). Similar results were observed for correlation between compressive strength and RCPT test.

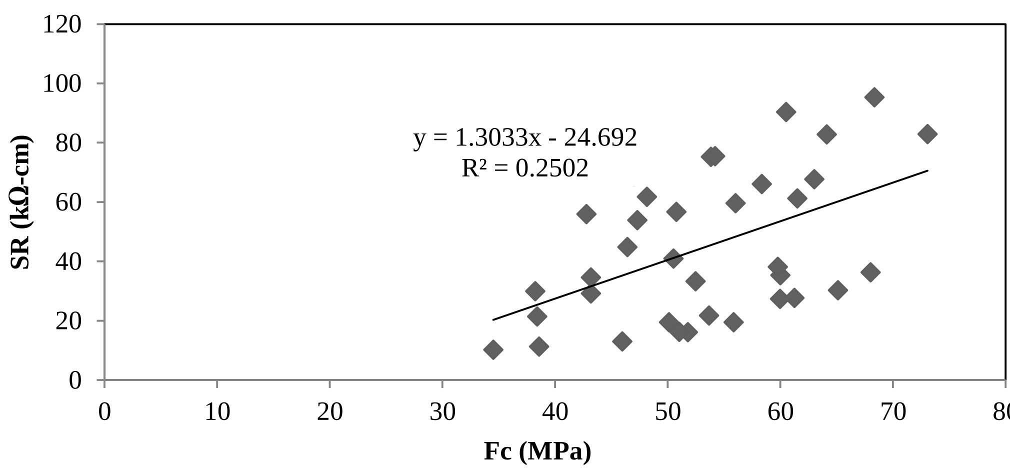

Figure 6: Correlation between compressive strength and electrical resistivity of concrete in all specimens at various ages.

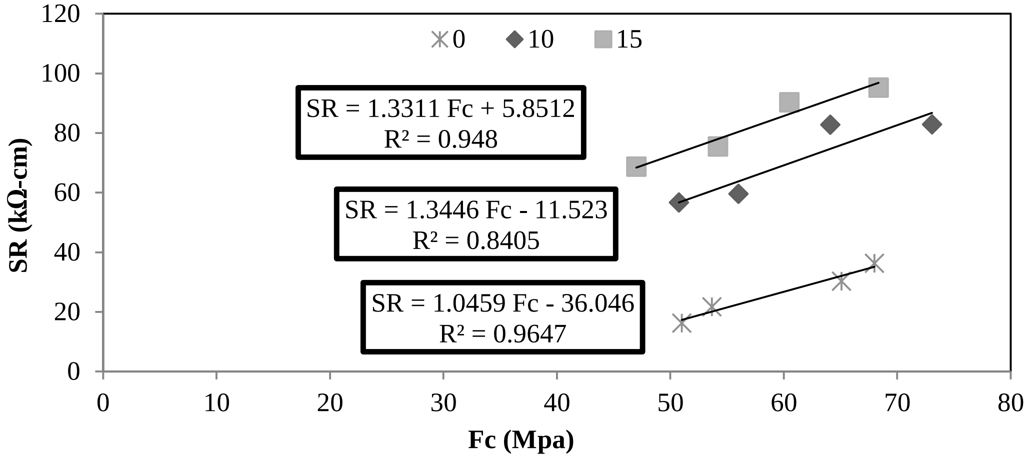

Figure 7: Correlation between compressive strength and electrical resistivity of concrete at 270 days in different zeolite replacement

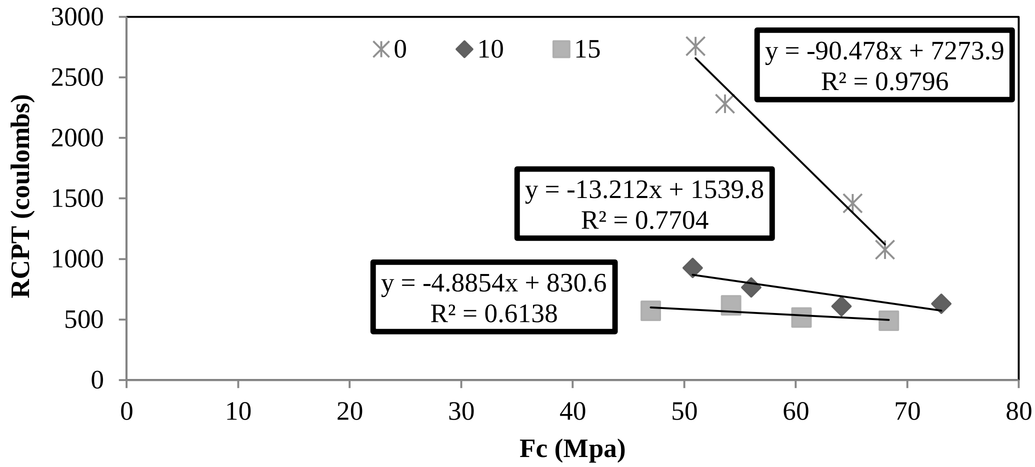

Figure 8: Correlation between compressive strength and RCPT of concrete at 270 days in different zeolite replacement

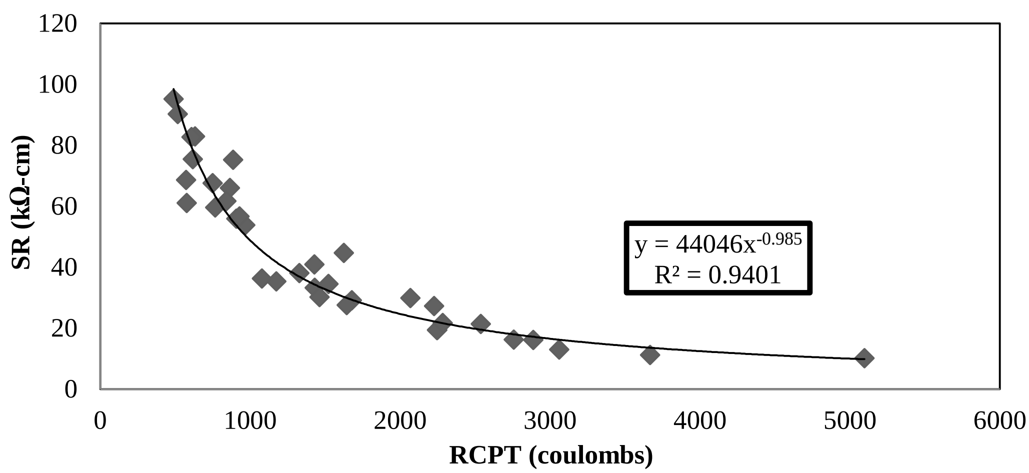

Figure 9: Correlation between RCPT and electrical resistivity of concrete in all specimens at various ages

As shown in Figure 9, the results of this study indicate that there was a strong power relation ($y = ax^b$) between the surface resistivity (SR) and chloride permeability test (RCPT) with a level of agreement (R^2) of 0.94 for a wide range of concrete specimens. This means that changes in the type of measured data (conductivity instead of total charge) is an option that provides an improvement for the RCPT test (Ramezanianpour et al., 2011). Considering the obtained correlations, a new model for correlating SR with RCPT is presented in Figure 9.

As expected, there was no significant relation between electrical resistivity and carbonation or between RCPT and the carbonation of concrete in various mixtures, because effective factors on the results are different. As can be seen in Figure 10 and Figure 11, a good relationship was obtained with the same w/c ratio (R^2=0.99 and 0.97). However when samples with different cementitious materials, w/c ratios or different ages was used, the correlation coefficient was reduced. Similar results were observed for RCPT and carbonation tests. These figures show that results obtained from different tests confirm one another.

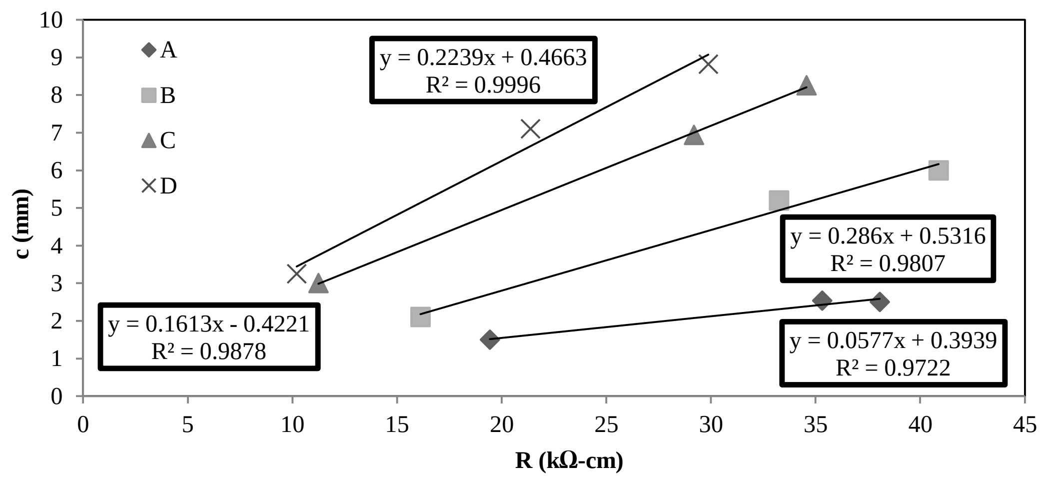

Figure 10: Correlation between carbonation and electrical resistivity of concrete at 28 days for various w/c ratio

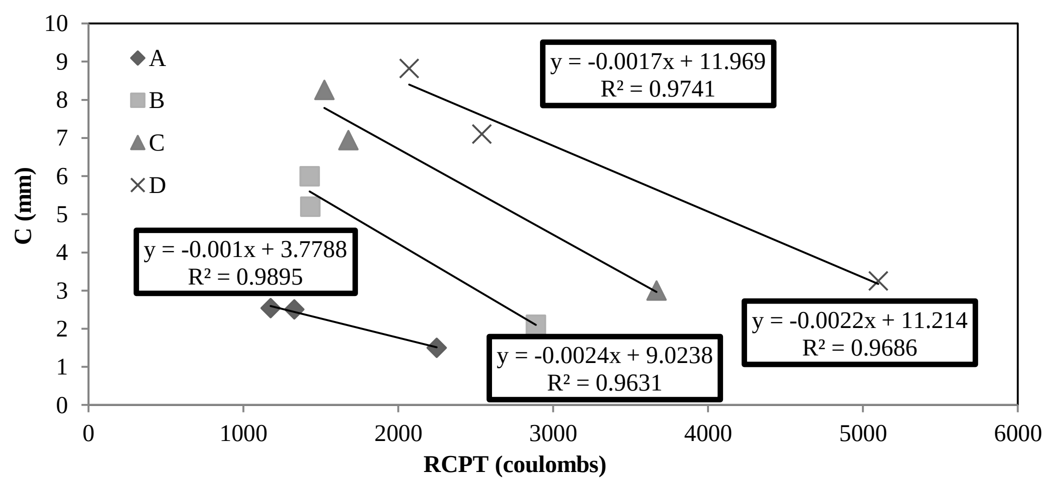

Figure 11: Correlation between carbonation and RCPT of concrete at 28 days for various w/c ratio

6. Conclusions

The following conclusions can be drawn from the obtained experimental data:

i. In general, the use of zeolite decreases slumps of concrete, so a superplasticizer is needed.

ii. The addition of zeolite delays the strength development during the first 28 days, after that age concretes having up to 10% zeolite provide competitive compressive strength with PC concretes.

iii. With increasing w/c ratio, the growth rate of compressive strength decrease at early ages and increase by the time.

iv. Less water absorption and water permeability was observed in the concretes containing up to 15% zeolite.

v. The substitution of cement with 15% natural zeolite reduced the chloride penetration of concrete mixtures according to the RCPT test. The rate of reduction increase with increasing w/c ratio.

vi. The electrical resistivity of concrete samples containing zeolite increased 2 to 4 times compared to control samples. Adding higher percentages of zeolite to cement produces high electrical resistivity.

vii. The addition of zeolite up to 15 % in concretes with W/C = 0.5 delays higher electrical resistivity and lower passing charge in RCPT test , compared PC concretes with W/C = 0.35 , so the addition of zeolite is more effective than the reduction of w/c ratio.

viii. The performance of concrete with cement replacement by zeolite is considerable in RCPT and electrical resistivity test which are in many cases the most important characteristic concerning durability and corrosion prevention.

ix. The depth of carbonation increases with increasing zeolite replacement.

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