

Effect of Using Metakaolin on Chloride Ion Penetration in High Performance Steel Fiber Reinforced Concrete

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Abstract

This paper attempts to reduce the penetrability of high performance steel fiber reinforced concrete to chloride ions originating from external sources, by using High Reactivity Metakaolin (HRM) as a highly active pozzolanic material, in order to prolong the time to initiation of the steel fibers corrosion and to minimize concrete damage that may occur due to the exposure to chloride ion penetration.

According to pozzolanic activity index (P.A.I.), 8% content of HRM was used as a partial replacement by weight of cement with 2% steel fibers by volume of concrete. During the exposure period of 300 days in 4.5% of NaCl solution, the total and free chloride contents (Cl_{total} , Cl_{free}) with the chloride profiles at the ages of 28 and 300 days were investigated. Also the rapid chloride penetrability test (RCPT), compressive and flexural strengths tests were conducted at the ages of 28, 90, 180 and 300 days.

Results showed that the incorporation of 8% HRM caused a reduction in the (Cl_{free}/Cl_{total}) ratio, the chloride penetration depth and the electrical conductivity with percentages of 21%, 40% and 43% respectively after 300 days exposure to chloride solution in comparing with the mix of 0% HRM.

Results also indicated that the losses in compressive and flexural strengths after exposure of 300 days to chloride solution for the mix incorporating 8% HRM were by 5% and 5.8% respectively while they reached 9.5% and 11% respectively for the mix without HRM in relation to the correspondent test specimens cured in tap water.

Key words: Metakaolin, Fiber reinforced concrete

الخلاصة

تهدف هذه الدراسة الى محاولة تقليل نفاذية الخرسانة العالية الاداء المسلحة بالألياف الفولاذية للكلوريدات باستخدام الميتاكاولين العالي الفعالية باعتباره مادة بوزولانية عالية النشاط وذلك من اجل الحد من الصدأ الحاصل في الالياف الفولاذية والضرر الذي قد يحدث في الخرسانة عند تعرضها لنفوذ المحاليل الحاوية على املاح الكلوريدات .

تم استخدام محتوى مقداره 8% من الميتاكاولين العالي الفعالية كأستبدال جزئي من وزن السمنت وذلك أستناداً الى مؤشر الفعالية البوزولانية (P.A.I.)، مع 2% من الالياف الفولاذية نسبة الى الحجم الكلي للخرسانة. تم غمر النماذج الخرسانية في محلول كلوريد الصوديوم وتركيز 4.5% ولفترة تعرض بلغت 300 يوماً تم خلالها ايجاد محتوى الكلوريدات الحرة والكلية ومنحنى توزيع تراكيزها نسبة الى العمق عن سطح النموذج الخرساني عند الاعمار 28 و 300 يوماً، كما تم قياس مقاومة الخرسانة لنفوذ أيونات الكلوريدات (RCPT) وكذلك مقاومتها الانضغاط والانشاء عند الاعمار 28، 90، 180 و 300 يوماً.

أظهرت النتائج ان استخدام 8% من الميتاكاولين العالي الفعالية أدى الى تقليل نسبة محتوى الكلوريدات الحرة الى محتوى الكلوريدات الكلية (Cl_{free}/Cl_{total})، عمق نفوذ تلك الكلوريدات عن سطح النموذج الخرساني وكذلك قابلية الخرسانة لتمرير الشحنة الكهربائية بحدود وصلت الى 21%، 40% و 43% على التوالي بعد فترة تعرض بلغت 300 يوماً لمحلول الكلوريدات بالمقارنة مع الخرسانة الغير الحاوية على الميتاكاولين.

كما أشارت النتائج أيضاً الى ان مقدار النقصان الحاصل في مقاومتها الانضغاط والانشاء للنماذج المعرضة الى محلول الكلوريدات كان بحدود 5% و 5.8% على التوالي للخلطة الحاوية على 8% من الميتاكاولين العالي الفعالية بينما كان النقصان للخلطة التي لا تحتوي عليه بحدود وصلت الى 9.5% و 11% على التوالي بالمقارنة مع نماذج الفحص المطابقة والمعالجة بالماء.

الكلمات المفتاحية: ميتاكاولين ، الخرسانة المسلحة بالالياف

1. Introduction

High Performance Concrete (HPC) has been defined as a concrete that possesses high workability and high strength with long term durability through aggressive environmental action (Russell,1999). Under these circumstances, the use of steel fibers in reinforcing HPC may need more requirements to prolong the time to

initiation of the fibers corrosion, especially with the presence of the harmful chloride solutions and their ingress into the concrete (Tasi and Hwang, 2009).

Even in low concentration, a chloride solution can promote the corrosion of steel in concrete (reinforcement or fibers). The threshold of chloride for corrosion is believed to be within the range of 0.2% - 0.4% (Alonso *et. al.*, 2000). In fact chloride ions in concrete are partially bound with the solid phases (bound chlorides), only the so-called (free chloride) which dissolves in pore water is a risk of steel corrosion (Kurdowski, 2004). Once these ions reach the steel fibers they depassivate the layer surrounding them and in the presence of air and water, the steel fibers commence to corrode. The corrosion products (rust) are greater in volume 400% –600% than the original steel resulting in an expansion and later cracking of the concrete (Marcus, 2002).

Besides promoting of steel corrosion, strong chloride solutions may influence the concrete by dissolving the portlandite (calcium hydroxide) in the hardened cement paste and converting it to soluble chloro – hydroxide hydrates. The loss of calcium hydroxide will make the concrete more porous, and eventually loses a part of its strength (Lee *et. al.*, 2000).

Thus the production of HPC of low permeability is of vital importance in reducing steel corrosion and concrete damage due to chloride ion penetration. However, in terms of the role of the binder, the objective is to come up with options for assessment of performance, one of these options is to use supplementary cementing materials such as HRM.

The use of HRM as a highly active pozzolanic material in conjunction with a high range water reducer (HRWR) may produce a HPC with special feature in resisting Cl^- penetration. The pore – size and grain – size refinement processes due to the chemical reaction between the pozzolanic particles and calcium hydroxide in the hydrated matrix will reduce the concentration of hydroxyl (OH^-) in pore water which leads to a less diffusion of free chloride ions into concrete (Ngala *et. al.*, 1995). Also will form new cementitious compounds, that will reduce the micro cracking of the transition zone leading to a considerable reduction in permeability (Shirazi and Hossain, 2008).

Therefore the use of HRM with HRWR can make steel fiber reinforced concrete more resistant to chloride ion penetration due to the changes in concrete pore structure and in the chemistry of pore solution.

2. Research Significance

Chlorides originating from external sources (i.e. exposure to seawater, chloride solutions and using de-icing salts) can penetrate into concrete by the means: diffusion, capillary absorption and hydrostatic pressure. Diffusion is considered the most familiar method which depends on the concrete pore structure and chemistry of pore solution (Ngala *et. al.*, 1995). The inclusion of supplementary cementitious materials, such as Metakaolin, may have a significant effect in modifying the pore structure and changing the chemistry of pore solution, leading to less concentration of free chloride which is capable of initiating or accelerating corrosion process and damaging concrete (Duchesne and Berube, 1994).

3. Experimental work

3.1. Materials

3.1.1. Cement

An ordinary Portland cement manufactured by the new cement plant of Kufa was used in this work, which complied with (IQS 5, 1984). Its physical properties and chemical composition are shown in Tables (1) and (2) respectively.

Table (1) : Physical properties of cement

Physical property	Test result	Limits of Iraqi spec. No. 5/1984
Specific surface area, m^2/kg	309	≥ 230
Initial setting time, min	110	> 45
Final setting time, hr.	6	≤ 10
Compressive strength		
3 days, N/mm^2	18.6	≥ 15
7 days, N/mm^2	27.3	≥ 23

Table (2) : Chemical composition of cement

Oxide	Content	Limits of Iraqi spec. No. 5/1984
CaO %	60.8	--
SiO ₂ %	21.4	≥ 21
Al ₂ O ₃ %	4.26	≤ 6.0
Fe ₂ O ₃ %	3.91	≤ 6.0
MgO %	2.3	≤ 5.0
SO ₃ %	2.12	≤ 2.8
L.o.I %	1.5	≤ 4.0
I.R %	0.8	≤ 1.5
L.S.F	0.91	0.66 – 1.02

3.1.2. Fine Aggregate

A natural sand with grading limits in zone (3) according to (IQS 45, 1984) was used, its specific gravity, bulk density, absorption, sulfate and total chloride contents are 2.6, 1760 kg/m^3 , 0.8%, 0.21% and 0.03 % respectively. Table (3) illustrates the grading of fine aggregate.

Table (3) : Grading of fine aggregate

Sieve size (mm)	Passing %	Limits of IQS 45/1984
4.75	95	90 – 100
2.36	88	85 – 100
1.18	78	75 – 100
0.6	64	60 – 79
0.3	24	12 – 40
0.15	4	0 – 10

3.1.3. Coarse Aggregate

A crushed coarse aggregate of 10 mm maximum size was used, its specific gravity, bulk density, absorption, sulfate and total chloride contents are 2.62, 1640 kg/m^3 , 0.47%, 0.05% and 0.035% respectively. Table(4) shows the grading of coarse aggregate which conforms to (IQS 45, 1984).

Table (4) : Grading of coarse aggregate

Sieve size (mm)	passing %	Limits of IQS 45/1984
14	100	100
10	98	85 – 100
5	16	5 – 25
2.65	0.6	0 – 5

3.1.4. Steel Fibers

Hooked end steel fibers (commercially known as Dramax – ZC) were used throughout this work. Table (5) illustrates fiber properties.

Table (5) : Properties of steel fibers

Fiber length	30 mm
Equivalent diameter	0.5 mm
Aspect ratio	60
Tensile strength	1100 N/mm ²
Dosage	20 – 50 Kg/m ³

3.1.5. High Range Water Reducer (HRWR)

Melment L10/40 (sulphonated melamine formaldehyde condensate) was used as HRWR according to (ASTM C494/C494M -13 Type F,2013).Its technical description is given in Table (6).

Table (6) : Technical description of Melment L 10/40

Appearance	Clear to slightly milky
Density, g./cm ³	1.25 – 1.27
Subsidiary effect	Hardening accelerator
pH value (20 ⁰ C)	9.0 – 11.4
Solids in aqueous solution	Approximately 40%
Total chloride content	0.003%
Dosage recommendation, % by weight of binder	0.5 – 2.5

3.1.6.1 High Reactivity Metakaolin (HRM)

The Dwekhla Iraqi kaolin was used in this work after calcining at 700⁰ C for 1 hr (Caldarone *et. al.*, 1994). Its chemical analysis is given in Table (7) and its specific gravity and fineness (Blaine) were 2.62 and 19000 cm²/g. respectively.

Table (7) : Chemical analysis of HRM

Oxide	Content %	Limits of ASTM C618 /1989 pozzolan class N
Al ₂ O ₃	36.83	≥ 70
SiO ₂	57.45	
Fe ₂ O ₃	1.41	
CaO	1.12	-
MgO	0.03	-
Na ₂ O	0.50	-
SO ₃	0.08	≤ 4
L.O.I	1.54	≤10

3.1.6.2. Pozzolanic Activity Measurement

In order to find the suitable content of HRM as a partial replacement by weight of cement, the Pozzolanic Activity Index (P.A.I.) was tested in accordance with (ASTM C 311,2013) for three cement mortars incorporating 8%,12% and 20% of HRM. The (P.A.I.) was determined as follows:

compressive strength of the tested mortar

$$\text{P.A.I. \%} = \left(\frac{\text{compressive strength of the reference mortar}}{\text{compressive strength of the tested mortar}} \right) * (100)$$

Results, as given in Table (8), indicated that the incorporation of 8% HRM as a partial replacement by weight of cement would give the highest (P.A.I.).

Table (8) : Pozzolanic activity index for tested mortars

Type of mortar	HRM% by wt. of cement	P.A.I. %
Reference	0	-
Tested m1	8	147.6
Tested m2	12	141.0
Tested m3	20	130.2

3.2. Concrete Mixes

The plain reference concrete mixture was designed to give a 28-day characteristic compressive strength of 60 N/mm² according to (ACI 211.4 R –98, 2004), with proportions of (1:1.16:1.72). The w/c ratio was 0.31 to obtain a slump of (100±5) mm. Four mixes having the same slump were prepared throughout this study as illustrated in Table (9). After 24 hrs. of casting, specimens were immersed, up to 300 days, in 4.5% of sodium chloride solution (Abdul Kareem,2007) while the reference specimens were cured in tap water.

Table (9) : Details and curing condition of concrete mixes

Mix	HRM Kg/m ³	Cement Kg/m ³	Sand Kg/m ³	Gravel Kg/m ³	Water Kg/m ³	HRWR % by wt. of cement	W/C	St. fibers % by vol. of concrete	Curing solution
M _{1W}	-	550	638	946	170.5	1.8	0.31	-	Tap wa.
M _{1C}									NaCl
M _{2W}	44	506	638	946	167.2	1.8	0.304	-	Tap wa.
M _{2C}									NaCl
M _{3W}	-	550	638	946	182	1.8	0.326	2	Tap wa.
M _{3C}									NaCl
M _{4W}	44	506	638	946	177.1	1.8	0.322	2	Tap wa.
M _{4C}									NaCl

3.3. Tests Performed

3.3.1. Chloride Content Tests

Two test methods were performed to find the total and free chloride contents as follows:

3.3.1.1. Total Chloride Content Test (Cl_{total})

The acid – soluble chloride content (Cl_{total}), including bound in the solid phases and free chlorides in the pore solution, was dissolved by extraction in nitric acid to be investigated according to (ASTM C1152/C1152M – 04, 2012).

3.3.1.2. Free Chloride Content Test (Cl_{free})

The water-soluble chloride content in the pore solution was extracted by leaching with water to be investigated according to (ASTM C1218/C1218M–99, 2008).

3.3.1.3. Sample Requirements

Ten powdered samples were drilled from two portions selected on the middle part of two opposite faces of 150 mm concrete cube to be used for each test. From each portion five samples were taken as representative of the concrete at the depths (5– 15), (15–25), (25–35) and (35–45) mm. The five samples of each face were oven – dried, ground to pass a No. 20 sieve and mixed with the correspondent powdered parts of the other face to make one part of at least 25 g.mass. A sample having a mass of approximately 10 g. was selected from each part to be tested.

3.3.1.4. Testing Age

According to the instructions of acid-soluble specification (ASTM C1152/C1152M-04,2012) for testing to meet water-soluble specification limits, tests should be performed on specimens at least 28 days old. Therefore both tests were carried out at the ages of 28 days as an initial testing age and 300 days as a final testing age for only the mixes M_{1W} , M_{1C} , M_{2W} , and M_{2C} . Table (10), Fig (1) and Fig (2) give the chloride content analysis and chloride profiles of these mixes.

3.3.2. Rapid Chloride Penetrability Test (RCPT)

The electrical conductance of concrete, expressed as the total charge in coulomb (ampere second) passed during a certain time interval through a concrete cylinder, was measured to provide a rapid indication of concrete resistance to chloride ion penetration. This test method was performed in accordance with (ASTM C1202, 2003) on three replicate concrete discs with diameter of 102mm (4 in) and length of 51mm (2 in) at the ages of 28, 90, 180 and 300 days for only the mixes M_{1W} , M_{1C} , M_{2W} , and M_{2C} . Fig (3) shows the results of this test.

3.3.3. Compressive Strength Test

The compressive strength of 150 mm concrete cubes was measured in accordance with (B.S.1881:part 116,1989) at the ages of 28, 90, 180 and 300 days for only the mixes M_{3W} , M_{3C} , M_{4W} and M_{4C} . Fig (4) shows the test results.

3.3.4. Flexural Strength Test

Prismatic concrete specimens of standard dimensions (100x100x400) mm were used to determine the modulus of rupture at the ages of 28, 90, 180 and 300 days. The test was carried out in accordance with (B.S. 1881: part 118, 1989) for only the mixes M_{3W} , M_{3C} , M_{4W} and M_{4C} . Fig (5) shows the results of this test.

4. Results and Discussion

4.1 Chloride Content Analysis

Table (10), Fig (1) and Fig (2) show the (Cl_{free} , Cl_{total}) contents and chloride profiles at the ages of 28 and 300 days for the mixes M_{1W} , M_{1C} , M_{2W} and M_{2C} . It is observed that the final values of chloride content (free or total) for the mixes cured in tap water (M_{1W} and M_{2W}) did not show major differences from the initial values and that can be attributed to the weak effect of tap water on washing out chloride ions from concrete pores. This observation agreed with the results which obtained by (Hooton and Hong,2000). Results also indicate that the free and total chloride contents and the ratio between them (Cl_{free}/Cl_{total}) for the mix incorporating 8%HRM (M_{2C}) were less than those of the mix with 0%HRM (M_{1C}) by about 24%, 14% and 12% respectively at age of 28 days and 39%, 23% and 21% respectively at age of 300 days. Moreover, it is found from Figures (1) and (2) that the penetration depth of chloride ions for the mix M_{2C} at the ages 28 and 300 days was less by about 20% and 40% respectively than that of the mix M_{1C} .

From the present chloride content analysis, it can be concluded that HRM has a significant effect in reducing the percentage contents of the free and total chlorides for the mix M_{2C} which makes this mix more resistant to chloride ion penetration than the mix M_{1C} . This may be related to the characteristics of the chemical products of the pozzolan-lime reaction which have higher molecular weight silicate chains than those in the structure of the calcium silicate hydrate (C-S-H) phases present in hardened paste of Portland cement. These products tend to subdivide the pore structure of the concrete leading to less permeability (Zhang *et. al.*,1999). In addition to that, the use of HRM will provide an additional alumina source which combines with free chloride to form insoluble salt and that will reduce the activity of these ions leading to a lower ratio of (Cl_{free}/Cl_{total}) for the mix M_{2C} than the mix M_{1C} (Duchesne and Berube, 1994).

4.2. Rapid Chloride Penetrability Test (RCPT)

The effect of the age and the inclusion of HRM on the total charge passing through concrete specimens are clarified in Fig.(3). The results show a significant difference between the total charge passing through the mixes incorporating 8% HRM (M_{2W} and M_{2C}) and that passing through the mixes with 0% HRM (M_{1W} and M_{1C}). There was a decrease by (25,27)% at age 28 days and (62,43)% at age 300 days in the total charge passing through the mixes M_{2W} and M_{2C} relative to the mixes M_{1W} and M_{1C} respectively. These differences are related to the difference in the electrical conductivity of the concrete mixes which depends on many factors such as: pore structure of concrete and chemistry of pore solution which are the main factors that influence the concrete electrical conductivity (Duchesene and Berube,1994).The inclusion of HRM may change these factors by providing an additional alumina source that can improve the pore structure and reduce the alkalinity of pore solution by incorporating more alkalis into hydration products than they release to the pore solution leading to less concentration of conductive ions (Shi, 2004).

Relating to the results of the present test, it can be noticed that the exposure to external chloride ions for both mixes M_{1C} and M_{2C} increases the electrical conductivity of concrete but with different rates depending on the content of HRM. This increase may be related to the fact that the ingress of chloride ions into the concrete will enhance the ability of the electrolytes to transmit the charge through concrete pores (Liu, 1996).

4.3. Compressive Strength Test

The compressive strength of the mixes containing 2% steel fibers (M_{3W} , M_{3C} , M_{4W} and M_{4C}) are shown in Fig.(4). From the strength patterns, it is observed that the exposure to NaCl solution caused a long term loss in compressive strength of the mixes M_{3C} and M_{4C} . The loss was by about 3.6% and 2.4% respectively at age 28 days and reached 9.5% and 5% respectively at age 300 days in relation to control mixes M_{3W} and M_{4W} . The initial loss in compressive strength especially at age 28 days can be due to the influence of chloride solution on the concrete but with time the corrosion of steel fibers may be initiated by the presence of free chlorides especially for those located in the outermost 40 mm of the concrete for the mix M_{3C} and 25 mm or less for the mix M_{4C} whereas the free chlorides present at high concentration as shown in the profile of Fig.(3). Besides promoting corrosion of steel fibers, chloride ions can influence the concrete by:1-Producing chloroaluminate which causes concrete deterioration by decalcification. 2- Interacting with calcium silicate hydrates and forming porous C-S-H due to involving many complex reactions. 3- Promoting the leaching of calcium hydroxide by converting it into soluble chloro-hydroxide hydrates (Kurdowski,2004;Carde and Francois,1997). The formation of stable, insoluble cementitious compounds due to the pozzolan – lime reaction, not only can reduce the excess amount of $Ca(OH)_2$ available to react with chlorides, but also can modify the pore structure of C-S-H phase leading to less influence of chloride ions on the concrete (Caldarone *et. al.*,1994).Thus results show that the inclusion of HRM makes the loss in compressive strength for the mix M_{3C} more pronounced than that of the mix M_{4C} .

4.4. Flexural Strength Test

As in compressive strength, Fig.(5) shows also a long term loss in flexural strength of the mixes M_{3C} and M_{4C} due to exposure to saline solution, but with more final loss than that in compressive strength. The loss was by about 11% and 9.5% respectively at age of 300 days comparing to correspondent control mixes M_{3W} and M_{4W} . This increase in the loss may be due to the fact that by contrast with the compressive strength, flexural strength of fiber reinforced concrete is highly

dependent on tensile strength of reinforcing fibers in controlling crack growing and transferring fracture energy around crack tips (Tasi and Hwang, 2009). Thus any loss in the tensile strength of steel fibers due to fibers corrosion, that may be initiated with time by the presence of free chlorides, will bring more loss in flexural strength than that in compressive strength. Furthermore the inclusion of HRM can prolong the initiation of steel fibers corrosion by controlling the transport of chloride ions through concrete pores (Shi, 2004) leading to less loss in flexural strength. Therefore it can be noticed that the loss in flexural strength of the mix M_{4C} was less pronounced than that of the mix M_{3C} .

Table (10): Chloride content analysis for the mixes with 0% fiber content

Cl _{free} / Cl _{total}		Average chloride content % by wt. of binder for a depth of (5 - 45) mm from concrete surface				Mix
		Free chloride (Cl _{free})		Total chloride (Cl _{total})		
300 days	28 days	300 days	28 days	300 days	28 days	
0.114	0.118	0.024	0.025	0.211	0.212	M _{1W}
0.183	0.146	0.098	0.041	0.535	0.280	M _{1C}
0.110	0.114	0.023	0.024	0.209	0.210	M _{2W}
0.145	0.128	0.060	0.031	0.414	0.241	M _{2C}

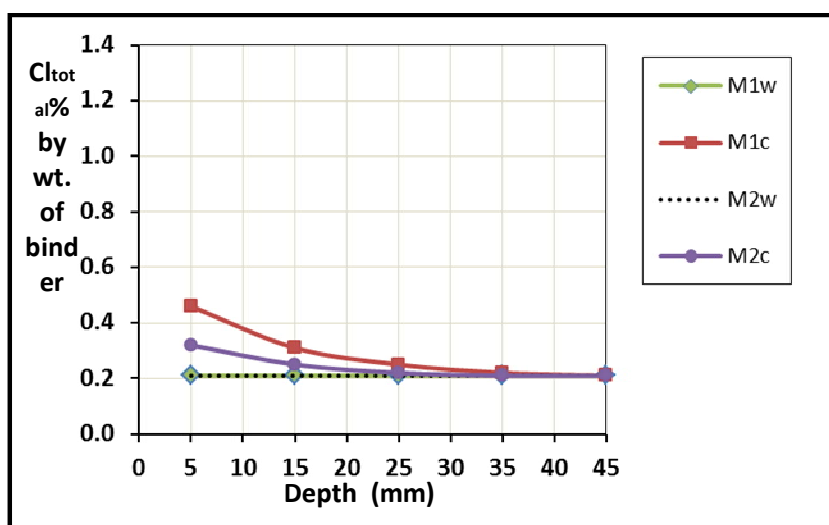


Fig (1) : Profile of total chloride content at 28 - day age

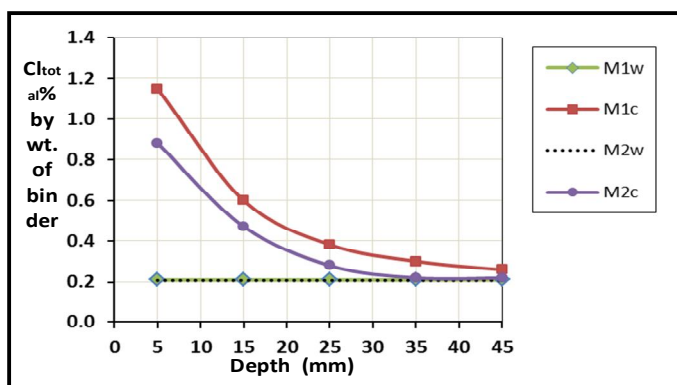


Fig (2) : Profile of total chloride content at 300 - day age

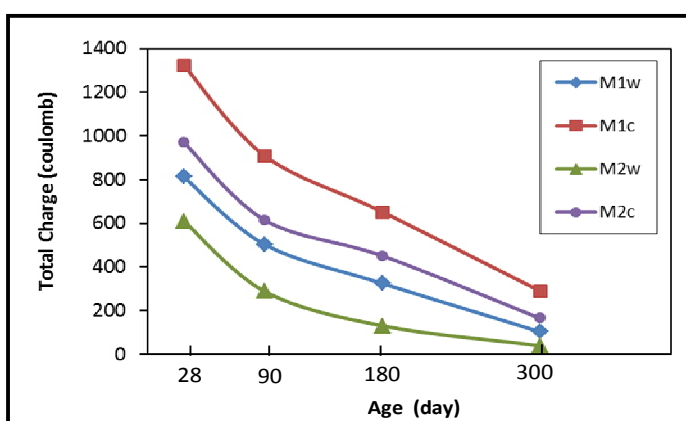


Fig (3) : Total charge passing through concrete specimens of different mixes with time

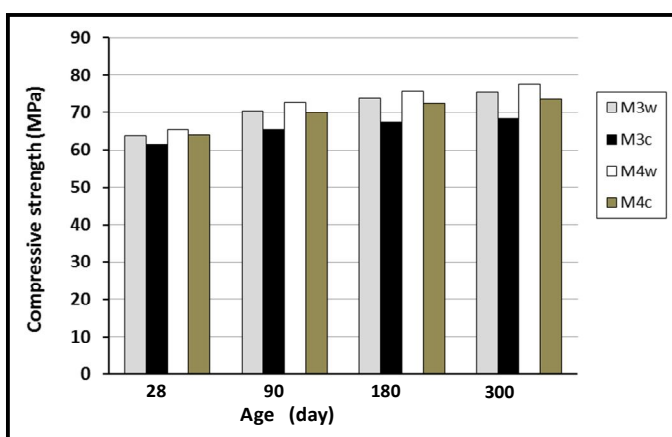


Fig (4) : Compressive strength of mixes at different ages

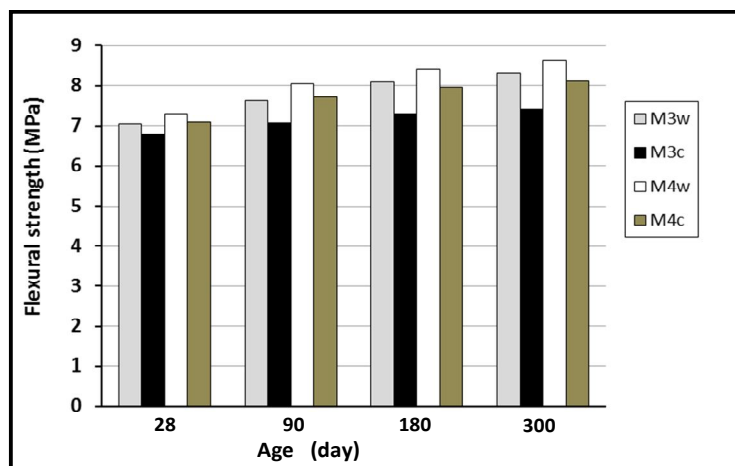


Fig (5) : Flexural strength of mixes at different ages

5. Conclusions

Based on the test results, the following conclusion can be extracted :

1. The exposure to external chloride source increases both free and total chlorides inside the concrete specimens. The use of HRM could be beneficial in controlling the rate of Ingress of chloride ions and their transport through concrete.
2. The incorporation of 8% HRM has reduced the ratio between (Cl_{free}/Cl_{total}) and the penetration depth of chlorides by 21% and 40% respectively after 300 days exposure to chloride ions relative to the mix with 0% HRM. This may be related to the additional alumina source provided by Metakaolin which serves to subdivide the pore structure and to combine with free chloride to form insoluble compound that reduces the activity of chloride ions.
3. The concrete mix with 8% HRM has always a less electrical conductivity than that of 0% HRM. The decrease was by (25-62)% for the mixes cured in tap water and (27-43)% for the mixes cured in sodium chloride solution at ages (28-300) days respectively. This decrease could be due to the reduction in alkali concentration or lower pH value in pore solution for the mix with 8% HRM relative to that without HRM.
4. The losses in compressive and flexural strengths after 300 days exposure to sodium chloride solution were more pronounced in the mix with 0% HRM than that with 8% HRM comparing to control mixes. This indicates that the concrete with HRM could be less susceptible to sodium chloride attack than that without HRM.
5. It is recommended to use 8% HRM as a partial replacement of the cement in conjunction with HRWR. This replacement could be beneficial in rising the resistance of high performance steel fiber reinforced concrete to chloride ion penetration in case of exposure to external chlorides.

6. References

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