

MIGRATING VS ADMIXED CORROSION INHIBITORS FOR STEEL IN PORTLAND, POZZOLONA AND SLAG CEMENT CONCRETES UNDER MACRO CELL CONDITION

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الخلاصة:

لقد تم تقويم خاصية تعطيل الأداء لمضادات الهجرة و مضادات المزلج للفولاذ المغروس في أنواع الإسمنت المسلح OPC, PPC, PSC في ظروف تآكل الخلية الكلي. وقد تم التحكم على مدار سنة من التعرض في عوامل تآكل الخلية الكلي كفرق الجهد السالب، تيار الخلية و التيار الكلي التكاملي، حيث تم تقويم سرعة التآكل للفولاذ المغروس باستخدام أطيايف الأشعة فوق البنفسجية والأشعة تحت الحمراء. وقد أظهرت جميع الدراسات أن مضادات الهجرة للتآكل تعمل على تقليل سرعة التآكل في الفولاذ المستخدم في أنواع الخرسانة المختلفة تحت ظروف الخلية الكلي بشكل أفضل من مضادات تآكل المزلج.

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ABSTRACT

The inhibitive performance of migrating and admixed inhibitors for steel embedded in ordinary Portland cement (OPC), Portland pozzolona cement (PPC), and Portland slag cement (PSC) concretes were evaluated under macro cell corrosion condition. Macrocell corrosion parameters, such as anode potential, macro cell current, and total integrated current, were monitored over a one year exposure period. The corrosion rate of steel embedded in different concretes was assessed by the gravimetric weight loss method. The surface of the specimens was further examined by UV and FTIR spectroscopy. All the studies revealed that migrating corrosion inhibitor (MCI) performed better than admixed inhibitor in reducing the corrosion rate of steel embedded in different concretes under macrocell condition.

Key words: migrating corrosion inhibitor, admixed inhibitor, macrocell corrosion, types of cement, adsorption

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1. INTRODUCTION

Corrosion of steel in concrete is one of the major causes of premature deterioration of reinforced concrete structures leading to structural failures. Various methods are being adopted to extend the service life of reinforced concrete structures, such as coating the concrete surface, coating reinforcements, cathodic protection, electrochemical removal of chloride, realkalization, and the use of corrosion inhibitors. Among the available methods, use of corrosion inhibitors seems to be more attractive because they are cost-effective and easy to handle [1]. Admixed inhibitors are added to fresh concrete (new structures) and migrating inhibitors are usually proposed for repair systems. While admixed inhibitors have been commercially available since the 70's, migrating corrosion inhibitors for concrete structures were proposed only in the last 5 to 10 years [1]. Both inorganic and organic inhibitors, such as zinc oxide [2], molybdates and borates [3,4], carboxylate ions [5, 6], quaternary ammonium salts, and many other organic compounds [7,8], were studied. Nitrite based inhibitors are considered as one of the most effective products available in the market. Nitrite acts as a passivator due to its oxidizing properties, and its inhibitive effectiveness is related to the $[\text{NO}_2^-]/[\text{Cl}^-]$ molar ratio that should be higher than 0.8 to 1 to prevent corrosion [5,9]. Organic corrosion inhibitors act by adsorption on the metal surface forming an organic layer that may inhibit both the anodic and cathodic processes and they are considered as mixed inhibitors [10,11]. Although a detailed review has been published in 2001 [1], the efficiency of commercial organic corrosion inhibitors is not yet well understood. Laboratory tests showed conflicting results about the inhibiting action of these products, both in simulated concrete environments and in actual concrete.

Amine and ester based admixed inhibitors have dual actions in concrete, as the amine compound acts as an inhibitor whereas the carboxylate ester compound acts as a pore-blocking agent which blocks the ingress of the chlorides ions [12–14]. Certain types of inhibitors, though showing higher corrosion resistant properties in simulated concrete environments, failed to maintain mechanical properties, such as setting time, workability, and compressive strength.

The objective of the present investigation is to evaluate the inhibitive performance of migrating and admixed inhibitors for steel embedded in ordinary Portland cement (OPC), Portland pozzolona cement (PPC), and Portland slag cement (PSC) concretes under macro cell corrosion condition.

2. EXPERIMENTAL

2.1 Materials Used

Ordinary Portland cement (OPC) conforming to IS:8112-1989 (equivalent to ASTM – Type-I cement), Portland pozzolona cement (PPC) conforming to IS:1489 – Part-I, and Portland slag cement (PSC) conforming to IS:455 were used. The chemical composition of the cements used is given in Table 1. Graded river sand passing through a 1.18 mm sieve with a fineness modulus of 2.85 and a specific gravity of 2.55 was used as a fine aggregate. The different size fractions of coarse aggregate (20 mm down graded and 12.5 mm down graded) were taken and recombined to a specified grading. TMT (thermo-mechanically treated) rebar of size 12 mm and potable water were used for casting the reinforced concrete. The mix design used for casting was 1:1.80:3.28 (W/C = 0.51).

Table 1. Chemical Composition of Cements

Compound	Wt%		
	OPC	PPC	PSC
Silicon-di-oxide (SiO_2)	20–21	28–32	26–30
Aluminium oxide (Al_2O_3)	5.2–5.6	5.0–8.0	9.0–11.0
Ferric oxide (Fe_2O_3)	4.4–4.8	4.9–6.0	2.5–3.0
Calcium oxide (CaO)	62–63	41–43	44–46
Magnesium oxide (MgO)	0.5–0.7	1.0–2.0	3.5–4.0
Sulphur-tri-oxide (SO_3)	2.4–2.8	2.4–2.8	2.4–2.8
Loss on ignition (LOI)	1.5–2.5	3.0–3.5	1.5–2.5

2.2. Inhibitors Used

The admixed inhibitor consisting of 0.5% sodium citrate+0.5% sodium stannate, analytical grade, was used. The migrating inhibitor consisted of equal proportions of amino alcohol, amines, and nitrites. The various control and corrosion inhibiting systems studied are given in Table 2.

Table 2. Control and Corrosion Inhibiting System

Abbreviation	System
A	Ordinary Portland cement (OPC)
B	OPC + Migrating corrosion inhibitor (MCI)
C	OPC + Admixed inhibitor
D	Portland pozzolona cement (PPC)
E	PPC + MCI
F	PPC + Admixed inhibitor
G	Portland slag cement (PSC)
H	PSC + MCI
I	PSC + Admixed inhibitor

2.3. Preparation of Rectangular Concrete Specimens

A rectangular concrete specimen of size 279 mm x 152 mm x 114 mm was designed for macro cell corrosion studies [15]. A TMT rebar of 12 mm diameter and 300 mm length was used as both anode and cathodes in the same concrete. The top mat of rebar acts as anode and the bottom mat of rebars act as cathode. The anode to cathode area ratio was maintained at 1:2 in order to induce accelerated corrosion of the embedded anode. The schematic view of a macro cell specimen is given in Figure 1. In both the anode and cathodes, 250 mm was embedded inside the concrete and the remaining length was used for electrical connection with proper insulations.

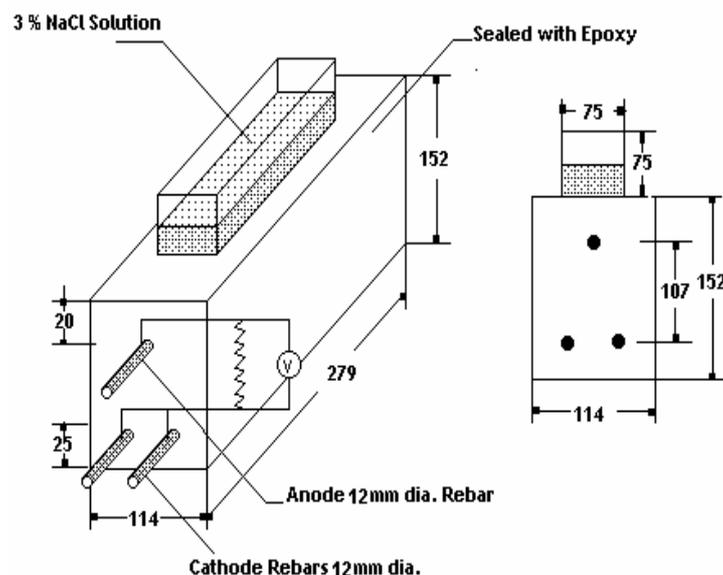


Figure 1. Schematic view of macro cell specimen

During casting, admixed corrosion inhibitors were added for uniform distribution. The specimens were mechanically vibrated. After 24 hours of setting, the specimens were demoulded and cured in distilled water for 28 days. On the other hand, migrating corrosion inhibitor (MCI) was applied on the concrete surface by the spraying method. This process was repeated 3 times at a time interval of 24 hours. Both systems were subjected to alternate wetting and drying cycles. One cycle consists of 2 weeks of wetting in 3% NaCl and 2 weeks of drying. Measurements were carried out during wetting cycles as macro cell current showed maximum magnitude due to the low resistivity of concrete. All the concrete specimens were subjected to 12 complete cycles of exposure period.

2.4. Macrocell Parameters

2.4.1. Potential vs. time behavior of steel in concrete

The open circuit potential of TMT steel anode was monitored for every cycle under alternate wetting and drying conditions using saturated calomel electrode (SCE) for an exposure period of 12 months.

2.4.2. Macro cell current vs time behavior of steel in concrete

The magnitude of macro cell current flow between anode and cathode was measured using a high-impedance (20 M Ω) voltmeter. The top mat and bottom mat rebars were connected by a 100- Ω resistor and macro cell current was obtained from the relation $I=V/100$. Macrocell current was monitored once in every cycle until the average macro cell current measured for the control specimens attained 10 μ A or greater. The tests were continued up to 12 complete cycles of exposure to ensure the presence of sufficient corrosion for visual examination.

2.4.3. Total integrated current vs time behavior of steel in concrete

The total integrated current for each system was calculated as per ASTM G:109-92:

$$TC_j = TC_{j-1} + [(t_j - t_{j-1}) \times (i + i_{j-1}) / 2] \quad (1)$$

where TC = total corrosion (coulombs)

t_j = time (seconds) at which measurements of the macro cell current is carried out, and

i_j = macro cell current (amps) at time t_j .

2.4.4. Qualitative and quantitative estimation of corrosion

At the end of the exposure period, the concrete specimens were split open and the anode (rebar) was rated for rusted area and expressed in terms of percentage of area rusted. The corrosion rate of TMT steel anodes embedded in the concrete specimen was determined by the gravimetric weight loss method. The final weight was measured after cleaning the rebars in inhibited hydrochloric acid as specified in ASTM G1-90. From the initial and final weight, the corrosion rate in mmpy was calculated using Equation 2 [16]:

$$\text{Corrosion rate} = \frac{87.6 W}{D A t} \quad (2)$$

where W is the metal loss in mg

D is density of Fe in g/cm³

A is area of the steel in cm², and

t is time in hours.

2.4.5. Chemical analysis for alkalinity and free chloride contents

The core samples collected near the anode were crushed mechanically and powdered. Then 100 gm of the powdered sample was shaken with 100 ml of double-distilled water in a conical flask using Microid flask shaker for 1 hour. The extract was then filtered through a Whatman filter paper No. 42. The extract prepared from the powdered sample was then analyzed for alkalinity and free chloride contents as per standard procedures [17,18].

50-cc of the filtered solution was placed in a 100-ml beaker and the alkalinity of the sample was measured in terms of pH using a standard calibrated pH meter.

20-cc of the filtered solution was placed and the free chloride content was estimated by standard silver nitrate using potassium chromate as an indicator. The amount of chloride present was expressed in terms of parts per million (ppm) on the basis of weight of sample taken for analysis.

2.5. Surface Examinations

At the end of the exposure period, the rebar specimens were taken out and concrete samples adhered on the surface were collected, powdered, and then subjected to UV-visible absorption spectroscopy and Fourier transform infrared spectroscopy (FTIR).

3. RESULTS AND DISCUSSION

3.1. Potential vs Time Behavior of Steel in Concrete

The electrochemical characteristics of half-cell potential of steel measured periodically with reference to SCE with time for OPC, PPC, and PSC concretes and their inhibited systems are shown in Figures 2, 3, and 4, respectively.

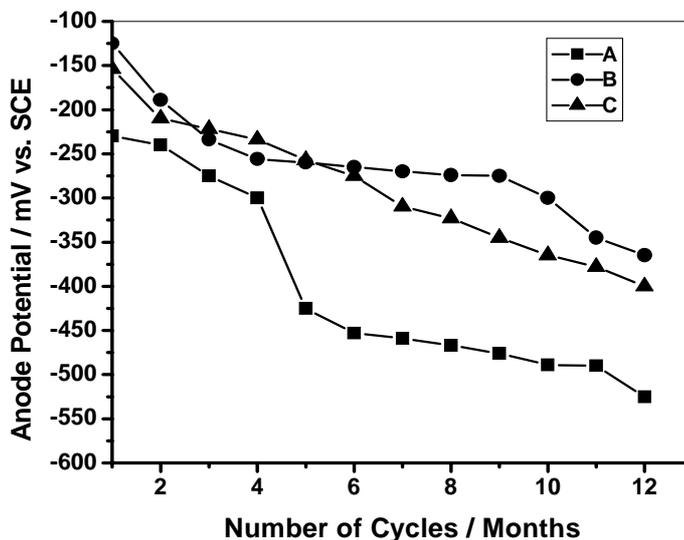


Figure 2. Potential vs number of cycles of exposure for steel in OPC concrete under macro cell condition [A - Control; B - Migrating; C - Admixed]

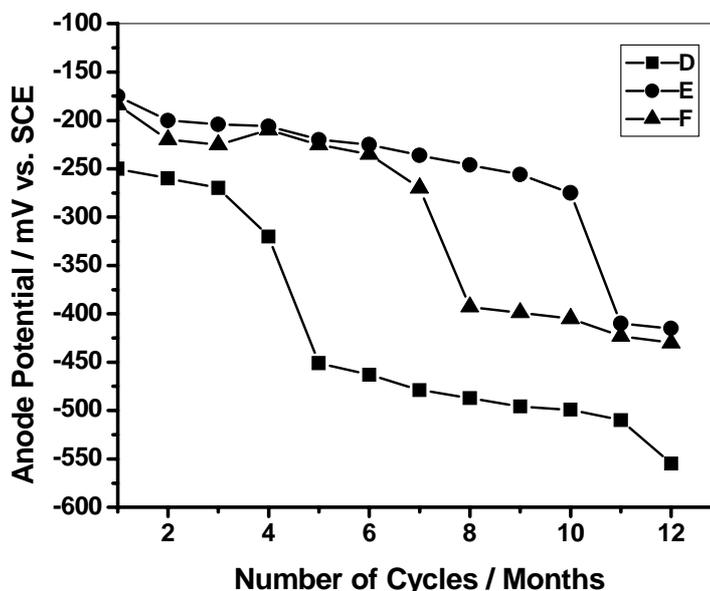


Figure 3. Potential vs number of cycles of exposure for steel in PPC concrete under macro cell condition [D - Control; E - Migrating; F - Admixed]

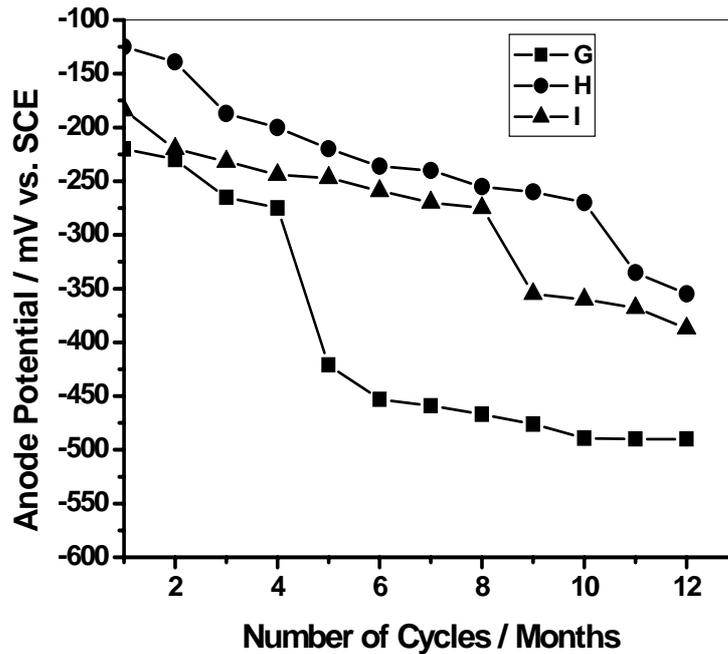


Figure 4. Potential vs number of cycles of exposure for steel in PSC concrete under macro cell condition [G - Control; H - Migrating; I - Admixed]

It is observed from Figure 2 that steel embedded in OPC control concrete (system A) showed active condition within 3 cycles of exposure. On the other hand, migrating inhibitor (system B) and admixed inhibitor (system C) maintained their passive condition of embedded steel up to the 9th and 6th cycles of exposure, respectively. The migrating inhibitor maintained its passivity for a longer period of time when compared to the admixed inhibitor in OPC concrete under macro cell condition.

It is observed from Figure 3 that steel embedded in PPC control concrete (system D) showed active condition within 3 cycles of exposure. However, the migrating inhibitor (system E) enhanced the passive condition of steel by 3 times and the admixed inhibitor (system F) enhanced the passive condition of steel by 2 times when compared to control concrete.

It is observed from Figure 4 that steel embedded in PSC control concrete (system G) showed active condition within 4 cycles of exposure. The passive condition of embedded steel was enhanced by 2.5 times for migrating inhibitor (system H) and 2 times for admixed inhibitor (system I) indicating the better performance of the migrating as well as admixed corrosion inhibitors.

Among the inhibited systems, MCI prolonged the passive condition of embedded steel in all three concretes. The better inhibitive performance of MCI is due to the fact that MCI, which contains basically $-NH_2$ and $-NO_2$ groups, maintained the OCP of embedded steel in a passive region only. The better performance of PPC and PSC was due to the increased chloride binding capacity of blended concrete. The chloride ions get adsorbed on the surface of the pozzalonic material and chemically react with its aluminate phase forming insoluble chloroaluminate complexes as follows: $3CaO \cdot Al_2O_3 \cdot CaCl_2 \cdot 10H_2O$. Use of blended cement is thus beneficial in restricting the mobility of chloride ions within the hydrated cement paste.

3.2. Macro Cell Current vs Time Behavior of Steel in Concrete

The macro cell current or galvanic current measured periodically with time for steel in OPC, PPC, and PSC concretes is shown in Figures 5, 6, and 7, respectively. From these figures, it is observed that all the control concretes (systems A, D, and G) showed a higher magnitude of macrocell current flow at the end of the 12th cycles of exposure which indicated the severe corrosion of the embedded steel. The macrocell currents measured were found to be 62 μA , 60 μA , and 58 μA for OPC, PPC, and PSC control concretes, respectively. Migrating inhibitor (systems B, E, and H) showed considerable reduction in the magnitude of current flow as 54.8%, 60%, and 62%, respectively. On the other hand, admixed inhibitor (systems C, F, and I) showed a slight decrease in the macrocell current for all the concretes. The reduction in the magnitude of current flow observed was 4.8%, 18.3%, and 39.6% for admixed OPC, PPC, and PSC concretes, respectively. This observation clearly proved that in the case of MCI, the enhanced inhibition was due to the formation of an insoluble stable complex film through the process of chemisorption on the metal surface. The film integrity was also confirmed by potential vs. number of cycle's behavior.

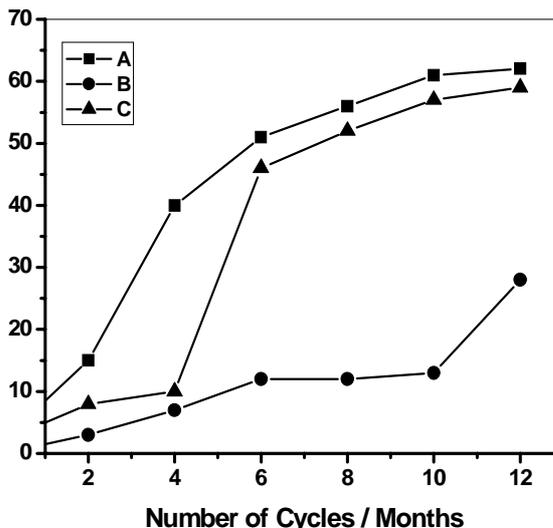


Figure 5. Macro cell current vs number of cycles of exposure for steel in OPC concrete under macro cell condition [A - Control; B - Migrating; C - Admixed]

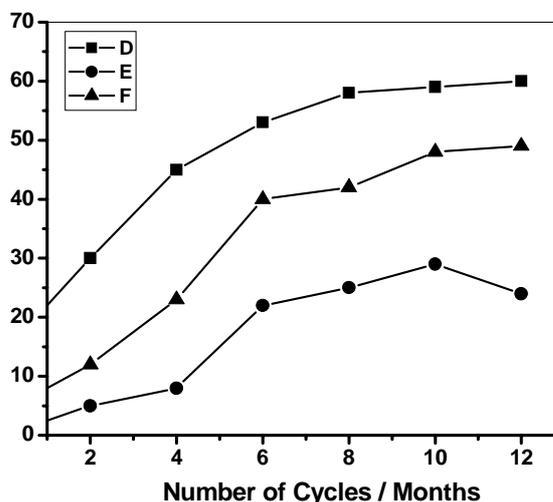


Figure 6. Macro cell current vs number of cycles of exposure for steel in PPC concrete under macro cell condition [D - Control; E - Migrating; F - Admixed]

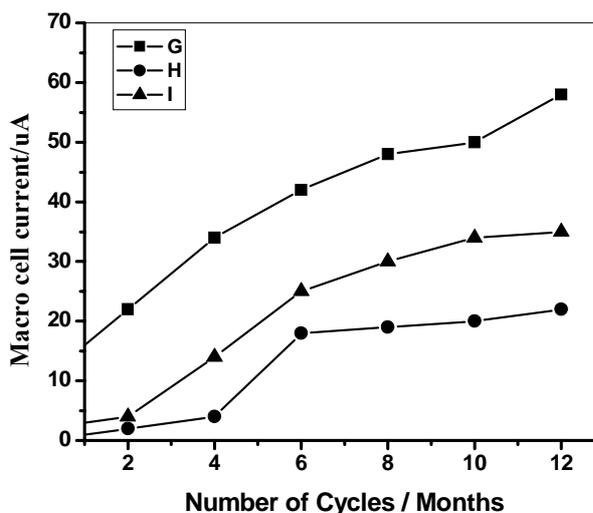


Figure 7. Macro cell current vs number of cycles of exposure for steel in PSC concrete under macro cell condition [G - Control; H - Migrating; I - Admixed]

The relative amounts of chloride and complexing anions available at the reinforcing steel determine whether the ferrous ions react first with the chlorides or the complexing anions. In the case of the OPC system, the availability of

chloride ions are available to occupy the defect position and react with ferrous ions and begin the corrosion process. However, in the case of PPC and PSC concretes, the availability of free chloride is significantly reduced due to the formation of chloroaluminate complexes. Hence, the relative amount of inhibitive anions more readily reacts with ferrous ions than chlorides. With the result, considerable reduction in the magnitude of current flow was observed in PPC and PSC concretes.

3.3. Total Integrated Current vs Number of Cycles

The total integrated current calculated with time using Equation 1 for OPC, PPC, and PSC concretes are illustrated in Figures 8, 9, and 10, respectively. From the figures, it is observed that at the end of exposure (12th cycle) all the control concretes (systems A, D, and G) showed higher coulombs value as observed earlier. Migrating inhibitor (systems B, E, and H) showed considerable reduction in the coulombs value when compared to admixed inhibitor (systems C, F, and I). For example, in the case of PSC concretes, 609 coulombs for control concrete, 335 coulombs for admixed inhibitor, and 226 coulombs for migrating inhibitor were measured.

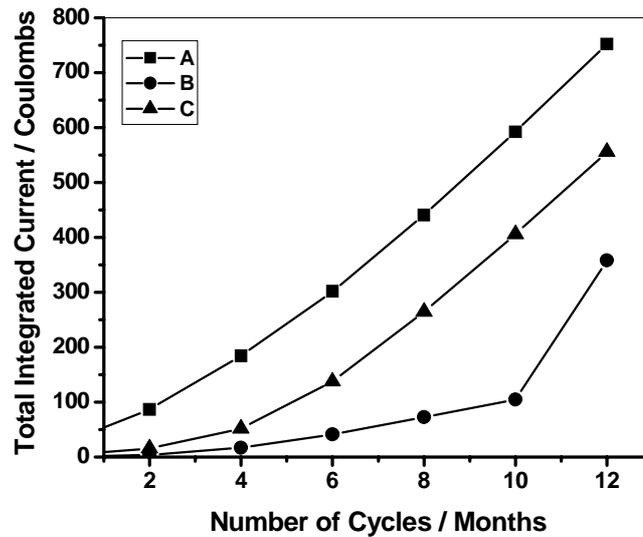


Figure 8. Total integrated current vs number of cycles of exposure for steel in OPC concrete under macro cell condition [A - Control; B - Migrating; C - Admixed]

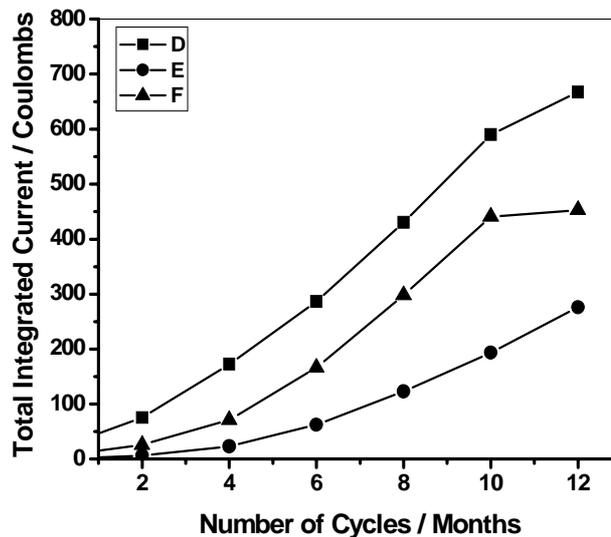


Figure 9. Total integrated current vs number cycles of exposure for steel in PPC concrete under macro cell condition [D - Control; E - Migrating; F - Admixed]

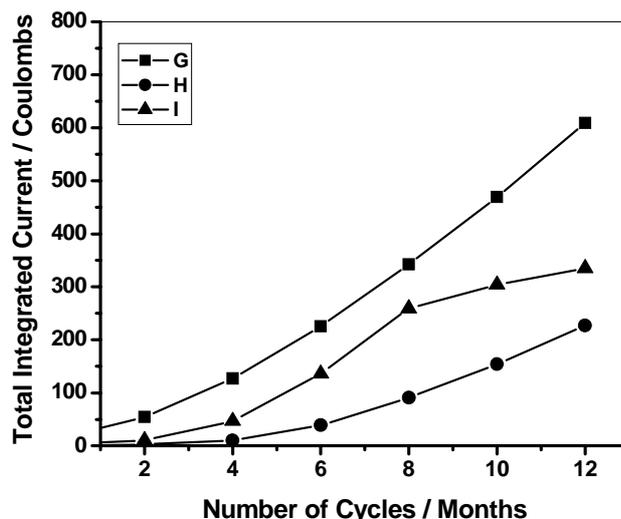


Figure 10. Total integrated current vs number of cycles of exposure for steel in PSC concrete under macro cell condition [G - Control; H - Migrating; I - Admixed]

3.4. Qualitative and Quantitative Estimation of Corrosion of Steel

Alkalinity (pH), free chloride contents (ppm), and corrosion rate (mmpy) measured at the end of the exposure period for various systems are given in Table 3. At the end of exposure, it is observed that the alkalinity of the control concretes were affected, but both migrating and admixed inhibiting systems maintained their alkalinity even in the presence of aggressive environments.

Table 3. Qualitative and Quantitative Estimation of Corrosion of Steel in OPC, PPC and PSC Concretes

Systems	Alkalinity (pH)	Free chloride contents (ppm)	Corrosion rate (mmpy)
OPC			
Control	11.45	4667	0.2044
Migrating	12.16	3750	0.1878
Admixed	12.08	3813	0.1892
PPC			
Control	11.46	3257	0.1810
Migrating	12.40	2862	0.1621
Admixed	12.33	3229	0.1751
PSC			
Control	11.46	2019	0.1396
Migrating	12.40	1813	0.1027
Admixed	12.31	2017	0.1188

Among the three concretes, PSC showed considerable reduction in free chloride contents. The migrating inhibiting system showed the least free chloride contents when compared to the admixed inhibiting system and control concrete.

For example, the free chloride contents estimated for the various concretes are as follows:

	OPC	>	PPC	>	PSC
Control	4667 ppm	>	3257 ppm	>	2019 ppm
MCI	3750 ppm	>	2862 ppm	>	1813 ppm
Admixed	3813 ppm	>	3229 ppm	>	2017 ppm

As a higher amount of chlorides was estimated for the OPC system, the corrosion rate was also found to be higher when compared to PPC and PSC concretes. Of all the concretes, PSC concrete showed the least corrosion rate of embedded steel in concrete. The corrosion rate of steel embedded in the migrating inhibiting system was found to be lower than the admixed inhibiting system. For example, in the case of PSC concrete, the corrosion rates were found to be 0.1396 mmpy, 0.1188 mmpy, and 0.1027 mmpy for control, admixed inhibitor, and migrating inhibitor, respectively. These data confirmed the fact that PSC concrete showed the superior performance even in the presence of aggressive macrocell conditions. The better performance of the migrating inhibiting system is due to the fact that MCI acts as a mixed type inhibitor. The presence of a lone pair of electrons in nitrogen atom is capable of forming a co-ordinate bond with metals and, thereby, enhancing the adsorption process. The adsorption process increases the over potential of metal and slows down the corrosion reaction. Two important pre-requisites in order to ensure a successful adsorption are the surface area which the molecules occupy and the necessary binding energy.

In the present investigation, MCI is water soluble and possesses necessary binding energy to metal surfaces. So it can easily displace surface water. This establishes an anchoring point that prevents the corrosive species reaching the metal surface.

3.5. Surface Examinations

3.5.1. UV-visible absorption spectroscopy

UV absorbance spectrum on concrete sample adhered on the steel surface is shown in Figure 11. UV absorbance spectrum indicated that λ_{\max} at 300 nm corresponds to the amino group [19]. It strongly supported that MCI adsorbed on the rebar surface and acted as a passivating layer and, thereby, protected the rebar from aggressive environments.

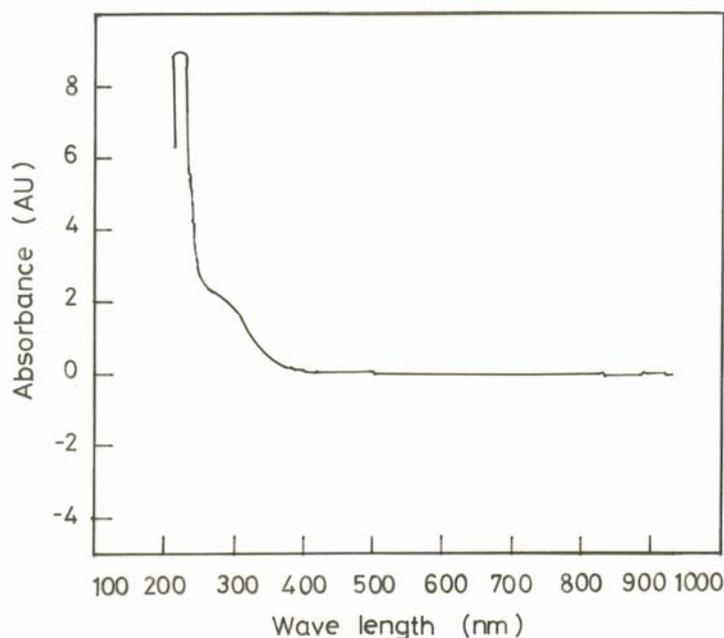


Figure 11. UV-vis. absorption spectrum on concrete sample adhered to steel surface

3.5.2 FTIR spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR) on concrete sample adhered on the steel surface is shown in Figure 12. The observed peak at the 3292 cm^{-1} indicated the -NH stretching vibration of amino group [20]. The presence of nitrite group was confirmed by the observed peak at 1409 cm^{-1} -NO stretching vibration. The -CO stretching and -OH in plane deformation at 1020.5 cm^{-1} confirmed the presence of alcohol group. The presence of -NH_2 , -NO_2 and -OH groups at the steel-concrete interface occurred due to the diffusion of migrating corrosion inhibitor. It is suggested that the MCIs, the primary inhibiting component, are an amino carboxylate or amino alcohol. These

inhibitors are able to diffuse through concrete to the underlying rebar where they act to suppress both the anodic and cathodic corrosion reactions by forming a monolayer film at the steel–concrete interface. This film acts as a barrier to the passage of aggressive ions (Cl⁻) and other species required for corrosion reactions.

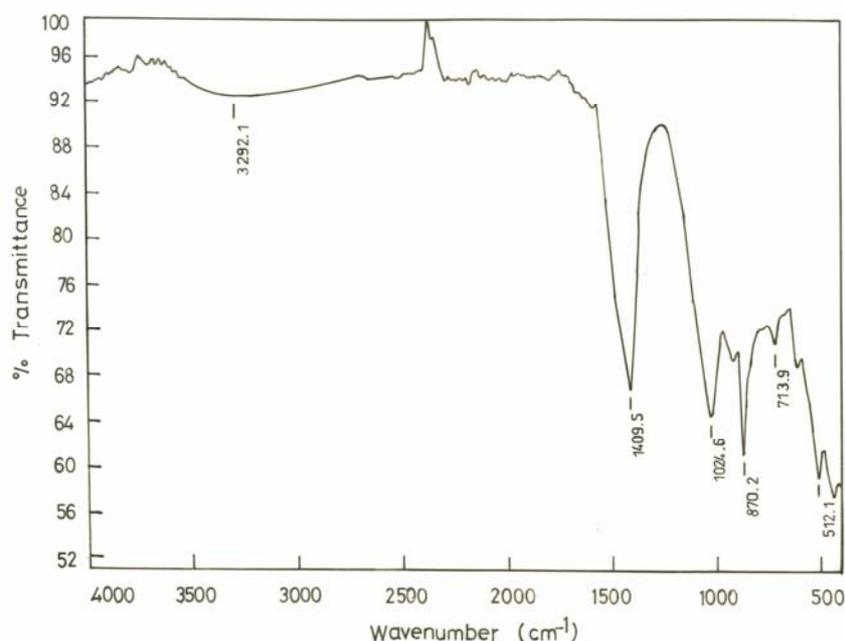
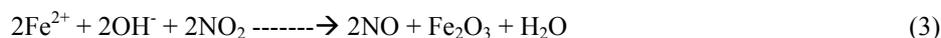


Figure 12. FTIR spectrum on concrete sample adhered to steel surface

It is a well known fact that amines are very good corrosion inhibitors for steel. Additional nitrites provide passive protection to the steel as follows:



The individual and the combined influence of all the above properties exhibited better corrosion resistance properties to the concrete as well as embedded steel.

4. CONCLUSIONS

The following conclusions were drawn from the present investigation:

- Alkalinity of the concretes was not affected by adding either MCI or admixed inhibitors. Among three concretes, PSC performed well under macro cell condition.
- Migrating inhibiting system enhanced the passive condition of embedded steel in concrete by three times and admixed inhibiting system enhanced the passive condition by 2 times when compared to the respective control concretes.
- Migrating inhibiting system showed 60% reduction in macrocell current, whereas admixed inhibiting system showed only 40% reduction in macrocell current in the case of PSC concretes.
- The prolonged passive condition of embedded steel, lowest macrocell and integrated current, lower free chloride contents, lowest corrosion rate, and visual observation data confirmed the better corrosion resistance of steel embedded in migrating corrosion inhibiting system.

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