

Improving the durability of concrete by using inhibitors

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Abstract

Corrosion of steel in concrete is one of the major causes of premature deterioration of reinforced concrete structures, leading to structural failure. Various methods are being used to extend the service life of reinforced concrete structures, which include surface coatings to concrete surface, coatings to the reinforcement, cathodic protection, chloride removal and corrosion inhibitors. Of these methods, the use of corrosion inhibitors is found to be one of the effective methods to control rebar corrosion. An attempt has been made to study the performance of the anodic inhibitors (sodium nitrite and zinc oxide), cathodic inhibitors (mono ethanol amine, diethanol amine and tri ethanol amine) and mixed inhibitors to control rebar corrosion. Compressive strength test, tensile strength test, chloride diffusion test and macro cell corrosion test were conducted by varying the type of inhibitors and varying the concentration of inhibitors. The addition of inhibitors not only increased the compressive strength of the concrete but also improved the corrosion resistance properties.

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Keywords: Inhibitors; Strength; Chloride diffusion; Macro cell corrosion

1. Introduction

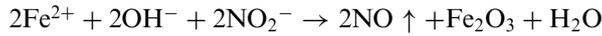
The use of inhibitors in concrete is an alternative option for preventing the corrosion of steel in concrete in the presence of chloride ions [1,2]. Corrosion inhibitors can be divided into three types: anodic, cathodic and mixed depending on whether they interfere with the corrosion preferentially at the anodic or cathodic sites or whether both are involved [3]. The use of corrosion inhibitors in concrete has been reviewed by Treadaway and Russel [4], Craig and Wood [5], Griffin [6], Slater [7] and most recently by Berke [8]. Earlier studies carried out on numerous inhibitors with most attention focused on sodium nitrite, potassium chromate, sodium molybdate and fluorophosphite [9,10]. Calcium nitrite is the first corrosion inhibitor admixture commercialized on a large scale for reinforced concrete [11]. Calcium nitrite inhibits

corrosion by reacting with ferrous ions to form a protective ferric oxide film [12,13]. Craig and Wood [14] studied sodium nitrite, potassium chromate, and sodium benzoate using the polarization technique and found that sodium nitrite was the most effective corrosion inhibitor, but it had harmful effects on concrete strength. Similar results were also reported by Treadaway and Russel [15], and found that sodium nitrite inhibited corrosion of steel bars in the presence of chlorides, whereas sodium benzoate did not. Rosenberg et al. [16] studied the effect of calcium nitrite as an inhibitor in reinforced concrete. They used polarization techniques for evaluation of the inhibitors and reported that the relative corrosion rates for samples soaked in saturated sodium chloride solution for 90 days with 2% and 4% admixed calcium nitrite were about a factor of 15 times lower than those without the calcium nitrite admixture. Berke [17] reported that, after 4 years of accelerated corrosion with chloride levels at the steel exceeding 7 kg/m^3 (12 lb/yd^2), the only uncorroded specimens were those which contained calcium nitrite. Berke and Sundberg [18] showed that the use of calcium nitrite

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and micro silica should significantly improve the durability of reinforced concrete in marine environment. Studies also showed that calcium nitrite, an anodic inhibitor, modifies the oxide film on the steel bar to be more protective than the film that naturally forms in concrete. The inhibitive action of calcium nitrite depends on its reaction with Fe^{2+} ions according to the following reaction:



Calcium nitrite competes with the chloride ions for ferrous ions produced in concrete and incorporates them into a passive layer on the iron surface, thus preventing further corrosion. Long-term corrosion studies showed that in spite of the decrease in concrete resistivity, corrosion rates were significantly reduced. Likewise, increases in AASHTO T277 chloride permeability values with calcium nitrite were clearly shown to have no negative effects on actual chloride ingress into the concretes [19]. It has been reported that the long-term performance of ZnO as a corrosion inhibitor reduced the concrete porosity and also chloride content at rebar level [20]. In the present investigation anodic, cathodic and the combination of two inhibitors were evaluated for their corrosion resistance by adopting various techniques.

2. Experimental detail

2.1. Materials used

Ordinary Portland cement:	Conforming to IS 8112–1989
Graded fine aggregates:	Local clean river sand (fineness modulus of medium sand equal to 2.46) conforming to grading zone III of IS-383—1970 was used.
Graded coarse aggregates:	Locally available well graded aggregates of normal size greater than 4.75 mm and less than 12 mm.
Inhibitors used:	Control(OPC)-System 1 Sodium Nitrite-System 2 Zinc Oxide-System 3 Mixed ($\text{NaNO}_2 + \text{ZnO}$)-System 4 Monoethanolamine-System 5 Diethanolamine-System 6 Triethanolamine-System 7
Percentages of inhibitor used:	1%, 2% and 3% by weight of cement.

2.2. Techniques used

1. Compressive strength test (as per BS—1881: Part 116: 1983)

2. Splitting tensile test (ASTM-C496-90)
3. Pull-out test (IS 2770—1967—Part-1)
4. Rapid chloride ion penetration test (ASTM-C1202)
5. Impressed voltage test
6. Potential-time behaviour studies (ASTM-C876)
7. Macrocell corrosion studies (ASTM-G109)
8. Gravimetric weight loss method

2.2.1. Compressive strength

100 mm × 100 mm × 100 mm concrete cubes were cast using 1:2.29:3.90 mix with W/C ratio of 0.53. Specimens were cast with and without inhibitors ranging from 1% to 3% by weight of cement. During moulding, the cubes were mechanically vibrated. After 24 h, the specimens were removed from the mould and subjected to water curing for 7, 14, and 28 days. After curing, the specimens were tested for compressive strength using AIMIL compression testing machine of 2000 kN capacity at a rate of loading of 140 kN/min. The tests were carried out on triplicate specimens and average compressive strength values were recorded.

2.2.2. Splitting tensile test

Concrete cylinders of size 150 mm diameter and 300 mm long were cast using 1:2.29:3.90 mix with W/C ratio of 0.53. The same procedure as indicated above was followed for casting and curing of the specimens. The tests were carried out on triplicate specimens and the average splitting tensile strength values were recorded.

2.2.3. Pull-out test

Cold twisted deformed (CTD) bars with size 12 mm diameter and 450 mm long were used for steel-concrete bond strength determination. The rod was placed concentrically in the concrete cube of size 100 mm × 100 mm × 100 mm using a concrete mix of 1:2.29:3.90 with W/C ratio equal to 0.53. Specimens were cast with and without inhibitors ranging from 1% to 3% by weight of cement. The rebars were projected down for a distance of about 10 mm from the bottom face of the cube as cast and projected upward from the top up to 300 mm height in order to provide an adequate length to be gripped for application of load. The same procedure as indicated above was followed for casting and curing of the specimens. After the curing period, the steel-concrete bond strength was determined using a universal testing machine (Model: UTE-60) of capacity 60 t. The bond strength was calculated from the load at which the slip was 0.25 mm divided by the embedded area of steel rebar. The tests were carried out in triplicate specimens and average bond strength values were obtained.

2.2.4. Rapid chloride ion penetration test (RCPT)

Concrete disc of size 85 mm diameter and 50 mm thickness with and without inhibitors ranging from 1%

to 3% were cast and allowed to cure for 28 days. After 28 days of curing the concrete specimens were subjected to RCPT test by impressing 60 V. The concrete disc is placed in between two halves of the PVC container of diameter 90 mm and sealed. One side of the container is filled with 3% NaCl solution (connected to the negative terminal of the power supply). Other side is filled with 0.3N NaOH solution (connected to the positive terminal of the power supply). Current is measured at an interval of every 30 min up to 6 h. Chloride contamination and temperature at every 30 min was also monitored. From the results using current and time chloride permeability is calculated in terms of coulombs at the end of 6 h.

2.2.5. Impressed voltage test

In impressed voltage technique [22] the concrete specimen is immersed in 5% NaCl solution and embedded steel in concrete is made anode with respect to an external stainless steel electrode serving as cathode by applying a constant positive potential of 12 V to the system from a DC power source. The variation of current is recorded with time until cracking occurred. Cylindrical concrete specimens of size 50 mm diameter and 100 mm height were cast using 1:2.29:3.90 mix ratio ($W/C = 0.53$) with centrally embedded rebar containing ordinary Portland cement with and without inhibitors ranging from 1% to 3% were cast and allowed to cure for 28 days. After curing, the specimens were subjected to impressed voltage test. For each specimen, the time taken for initial crack and the corresponding maximum anodic current was recorded. Triplicate specimens were used for this study.

2.2.6. Potential–time behaviour studies

Concrete specimens of size 100 mm × 100 mm × 100 mm were cast with 12 mm diameter rebar of 120 mm long rebar were embedded at a cover of 25 mm from one side of the cube specimen. The rebars were cleaned with pickling acid and degreased before embedded in concrete. The concrete specimens were cast with and without inhibitors ranging from 1% to 3%. All the triplicate specimens were cured in distilled water for 28 days. After 28 days of curing the cubes were taken out and dried for 24 h and subjected to alternate wetting and drying in 3% NaCl solution in order to accelerate chloride penetration and reinforcement corrosion. One cycle consists of 7 days immersion in 3% NaCl solution and 7 days drying in open atmosphere. Open circuit potential measurements (OCP) [21] were monitored using a voltmeter with a high input impedance of 10 M. Saturated calomel electrode (SCE) was used as a reference electrode. The positive terminal of the voltmeter was connected to the working electrode (rebar) and the common terminal was connected to the reference electrode. OCP was measured in three different locations very near to the rebar and the average potentials recorded. The cycle is continued for

150 days. From the results, potential vs. time plot is drawn using the average potentials obtained.

2.2.7. Macro cell corrosion studies

A rectangular concrete prism of size 279 mm × 152 mm × 114 mm was designed according to ASTM G109-92 for macro cell corrosion studies. CTD rebar of size 12 mm diameter, 300 mm length was used as anode and embedded in the concrete and taking electrical connection by screwing 6 mm diameter rod on the anode and the edges were properly insulated from the aggressive environments. The rebars were cleaned as mentioned in OCP. The top mat rebars act as anode and the bottom mat rebars act as cathode. The anode to cathode area ratio was maintained as 1:2 in order to induce accelerated corrosion. Concrete specimens were prepared using 1:2.29:3.90 mix with a W/C ratio of 0.53. The specimens were mechanically vibrated during casting. After 24 h the specimens were demoulded and cured in distilled water for 28 days. After the curing period was over, all the concrete specimens were ponded with 3% NaCl wetting cycles immediately. One alternate wetting and drying cycles consist of 3 days wetting in NaCl solution and 3 days drying in open atmosphere. The measurements were carried out under wet condition as macro cell current showed maximum magnitude due to the low resistivity of concrete. Tests were conducted on a minimum of triplicate specimens and the average values were recorded. Macro cell current flow between anode and cathode was measured using a high input impedance voltmeter. The top and bottom mat rebars were connected by a 100 Ω resistor and macro cell current was obtained from the relation $I = V/100$. Current was monitored once in every cycle until the average macro cell current of the control specimen is 10 μ A or greater.

2.2.8. Weight loss method

Cylindrical concrete specimens of size 55 mm in diameter and 60 mm in height, were cast using different types of inhibitors at 1%, 2% and 3% concentrations. Rebars, 12 mm in diameter and 50 mm long, were embedded in the center of each specimen. The initial weight of the rebar samples was taken in 4-digit electronic balance for gravimetric weight loss measurements. Concrete specimens were cast using 1:2.29:3.90 concrete mix with a W/C ratio of 0.53. During casting the specimens were mechanically vibrated. After 24 h, the specimens were demoulded and cured for 28 days in distilled water to avoid any contamination. After the curing period was over, all the specimens were completely immersed in 3% NaCl solution. The specimens were maintained in the same condition for 15 days and then subjected to drying for another 15 days. Therefore, one alternate wetting and drying cycle consisted of 7 days immersion in 3% NaCl solution

and 7 days drying in open air at room temperature. All the concrete specimens were subjected to six complete cycles. Tests were conducted on a minimum of three replicate specimens and the average values are reported. The corrosion rate was assessed from the difference in weight loss values between initial and final exposure period as reported elsewhere [23]. The corrosion rate is calculated using the following equation:

$$\text{Corrosion rate (mmpy)} = \frac{87.6W}{DAT},$$

where *W* is the weight loss in milligram, *D* the density of the material used, *A* the area of the specimen (cm²) and *T* is the test period in hours.

3. Results and discussion

3.1. Compressive strength

Fig. 1 shows the average compressive strength results of different inhibitor added concrete after 28 days of curing.

From Fig. 1 it is observed that, in 1% inhibitor addition, zinc oxide is showing a maximum compressive strength of 39.30 N/mm². In 2% inhibitor addition the mixed (NaNO₂ + ZnO) inhibitor system is showing a maximum compressive strength of 46.60 N/mm². In 2% inhibitor addition, sodium nitrite added concrete is showing a compressive strength of 46.0 N/mm². ZnO added concrete is showing a compressive strength of 45.00 N/mm². With 3% inhibitor addition all the inhibitor added concretes are showing a lesser compressive strength than the control concrete.

In 1% inhibitor addition the order of increase in compressive strength is found to be as follows:

ZnO > Diethanolamine > Control > Monoethanol > Sodium Nitrite > Mixed > Triethanol amine

In 2% addition the order of increase in compressive strength is as follows:

Mixed > NaNO₂ > ZnO > Diethanol amine > Monoethanol amine > Control > Triethanol amine.

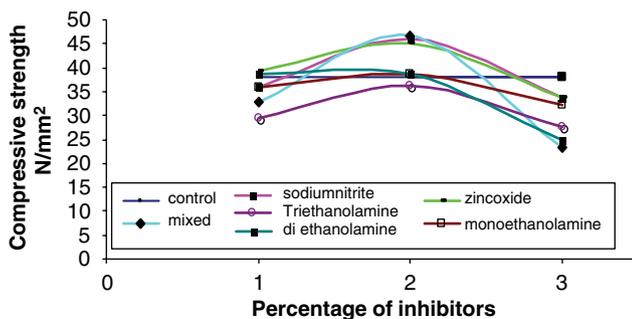


Fig. 1. Compressive strength at 28 days vs. percentage of inhibitors

In 3% inhibitor addition the order is found as follows: Control > ZnO > NaNO₂ > Monoethanol amine > Triethanolamine > Mixed > Diethanolamine.

From Fig. 1 it is observed that with 3% inhibitor addition a decrease in compressive strength values is observed which may be due to the retarding effect of the inhibitor addition, which increases the setting time effect. From compressive strength measurements it is observed that 2% is the optimum percentage addition of inhibitor to the concrete.

3.2. Splitting tensile strength

Figs. 2–5 show the splitting tensile strength of different types of inhibitor added concrete. With 1% inhibitor addition, all the inhibitor added systems show a tensile strength of more than 4.0 N/mm². With 2% inhibitor addition, except diethanolamine and triethanolamine all the systems show a tensile strength higher than the control concrete.

With 3% addition, all the inhibitor added systems show lower strength values than the control concrete. Fig. 5 shows the splitting tensile strength vs. percentage of inhibitor addition. From the figure it can be clearly inferred that, as the percentage of inhibitor level increases, the split tensile strength decreases.

3.3. Bond strength

Fig. 6(a)–(f) shows the comparison of bond strength for each inhibitor with various concentration. It can be seen that the addition of 1% of inhibitor gives a significant increase in bond strength.

3.4. Rapid chloride penetration test (RCPT)

Fig. 7 shows the chloride diffusion results of different percentage of inhibitor added concrete. From the figure

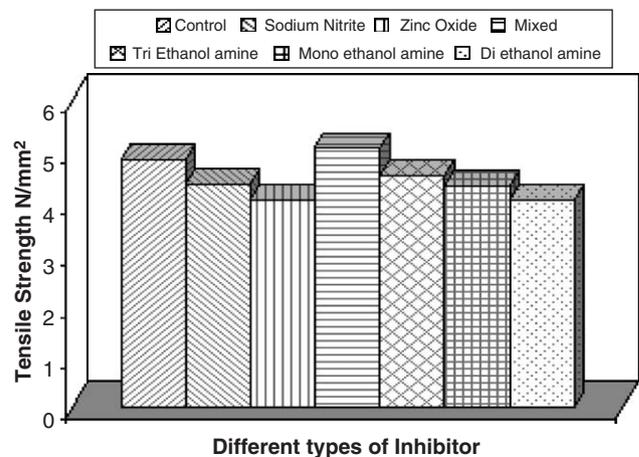


Fig. 2. Splitting tensile strength of 1% inhibitor added concrete.

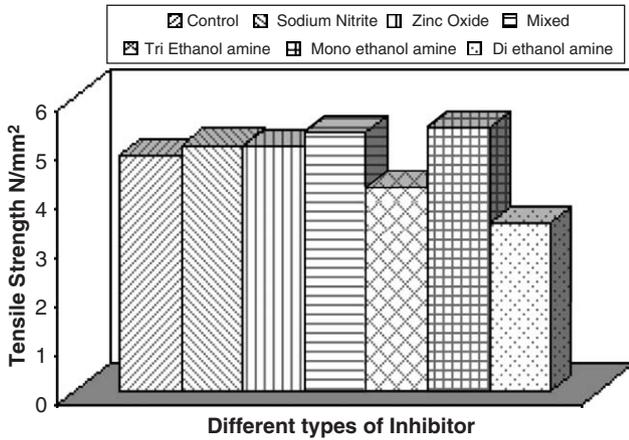


Fig. 3. Splitting tensile strength of 2% inhibitor added concrete.

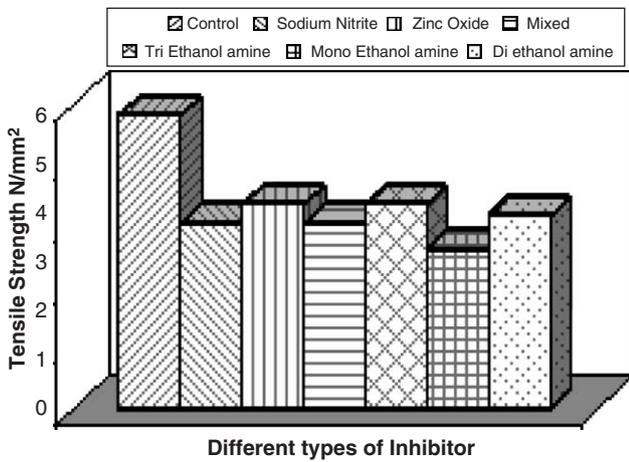


Fig. 4. Splitting tensile strength of 3% inhibitor added concrete.

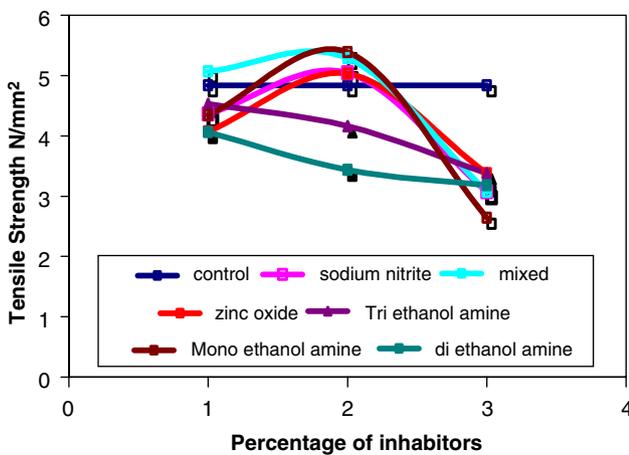


Fig. 5. Splitting tensile strength vs percentage of inhibitors.

it is observed that, zinc oxide and mixed inhibitor systems show lower coulomb values than the other systems.

3.5. Impressed voltage test

Fig. 8 shows the time to cracking with different types of inhibitors. From the figure it is observed that the time to cracking is maximum in the case of 2% inhibitor addition. Zinc oxide and mixed systems show a longer time to cracking, indicating the lower permeability of the concrete when compared to control and other systems.

3.6. Potential–time behaviour

Fig. 9 shows the open circuit potential measurement for 1% inhibitor added concrete immersed in 3% NaCl solution over a period of 120 days. From the figure it is observed that, all the system are showing a potential value more than -275 mV vs. SCE indicating the active condition of the rebars. When compared to control concrete all the inhibitor added concretes are showing a lower potential value than the control concrete. With 1% inhibitor addition zinc oxide, sodium nitrite, mixed inhibitor and monoethanolamine are behaving in a similar manner throughout the test period.

Fig. 10 shows the open circuit potential vs. time behaviour of 2% inhibitor added concrete in 3% NaCl solution under alternate wetting and drying conditions. In 2% inhibitor added concrete all the systems are showing a more negative potential than -275 mV vs. SCE indicating the active condition of the rebar. All the systems are found to perform equally irrespective of the percentage of inhibitor addition.

Fig. 11 shows the open circuit potential vs. time behaviour of 3% inhibitor added concrete in 3% NaCl solution under alternate wetting and drying conditions. With 3% inhibitor addition, except control concrete all the inhibitor added systems show a lower potential than the control concrete. Zinc oxide and mixed systems also show a more negative potential indicating the active condition of the rebar. With ZnO, the potentials are more negative (indicating the Zn precipitation) and remain so for a much longer time because of set retardation, which depends on ZnO content.

3.7. Macrocell current vs. time behaviour

Fig. 12 shows the macrocell current vs. time behaviour of 1% inhibitor added concrete under macrocell corrosion conditions. Except the control system all the inhibitor-added systems are showing a lower macrocell current indicating the passive condition of the rebar up to 60 days. But beyond 60 days a sharp rise in macrocell current observed indicating the active condition of the rebar. Zinc Oxide and mixed inhibitor systems show a lower macrocell current compared to the other systems.

Fig. 13 shows the macro cell current with number of days of exposure for 2% addition of inhibitor. It can be seen that up to 30 days of exposure all the systems,

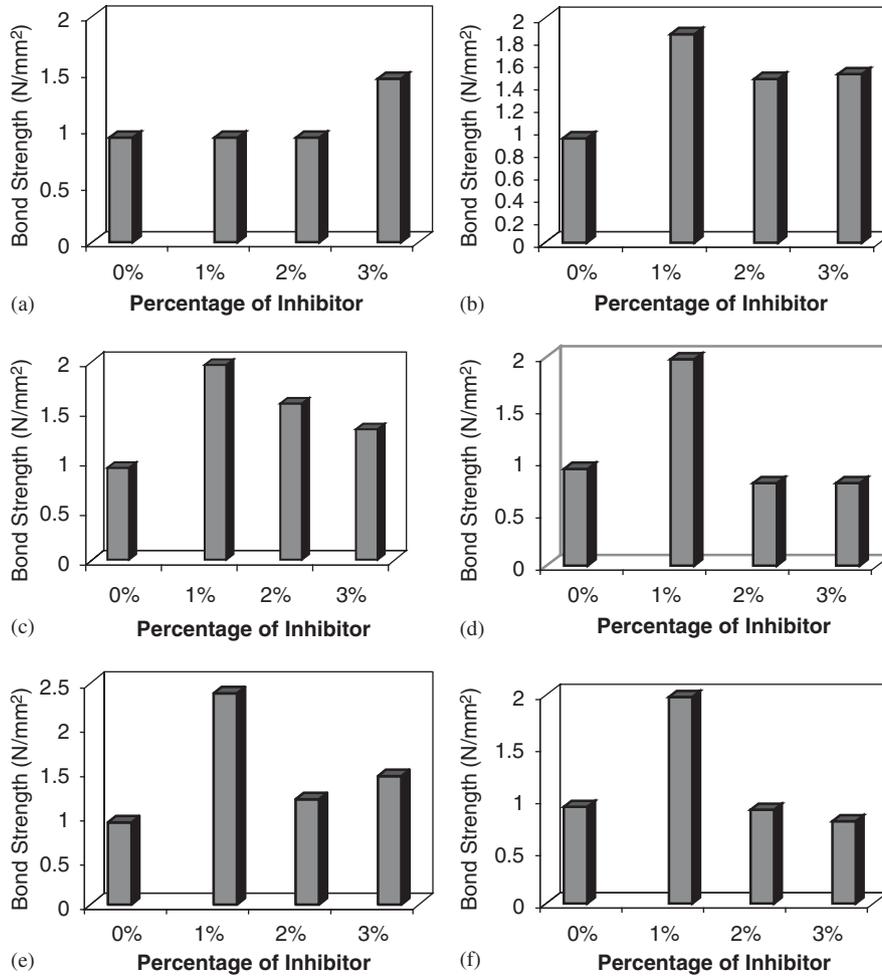


Fig. 6. Comparison of bond strength of different Inhibitor added concretes (a) control vs. sodium nitrite; (b) control vs. zinc oxide; (c) control vs. mixed; (d) control vs. triethanolamine; (e) control vs. monoethanolamine; (f) control vs. diethanolamine.

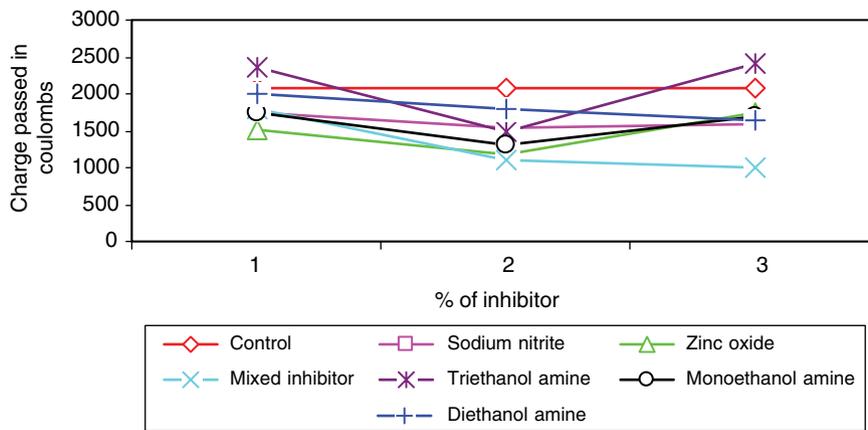


Fig. 7. Rapid chloride penetration test.

including control system, show a negligible macrocell current, indicating that the chloride has not reached the steel up to 30 days. But after 30 days there is a sudden shift in the macro cell current. Zinc oxide and mixed inhibitor, show a macro cell current less than 150 μ A

after 180 days of exposure. When compared to 1% inhibitor system, 2% inhibitor system shows a higher macro cell current.

Fig. 14 shows the macro cell current with the number of days of exposure for 3% addition of inhibitor. From

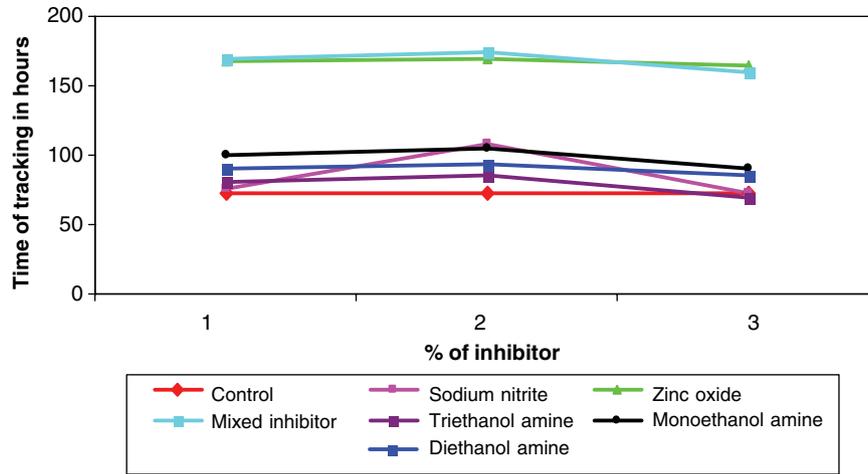


Fig. 8. Impressed voltage test.

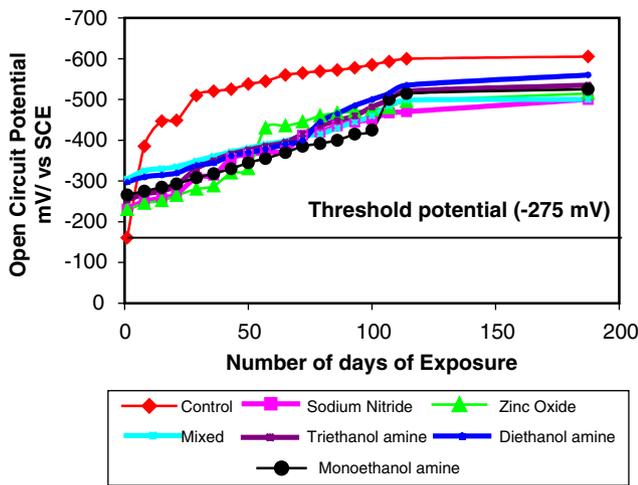


Fig. 9. Potential-time behaviour of 1% inhibitor admixed concrete in 3% NaCl solution.

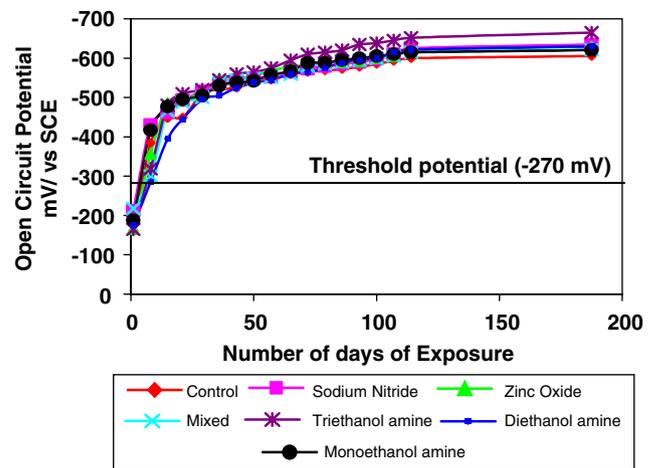


Fig. 11. Open circuit potential measurement of 3% inhibitor admixed concrete in 3% NaCl solution.

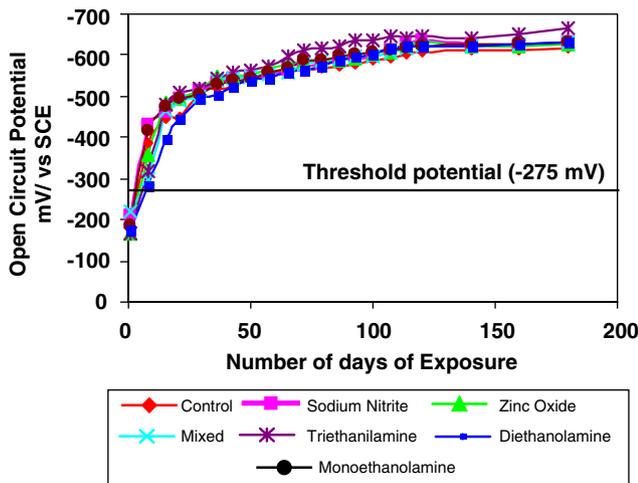


Fig. 10. Open circuit potential measurement of 2% inhibitor admixed concrete in 3% NaCl solution.

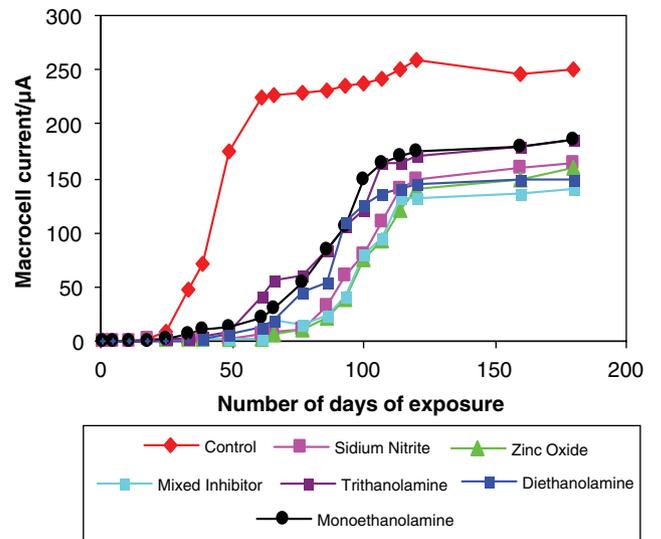


Fig. 12. Macrocell current vs. number of days of 1% exposure for inhibitor admixed concrete under macrocell condition.

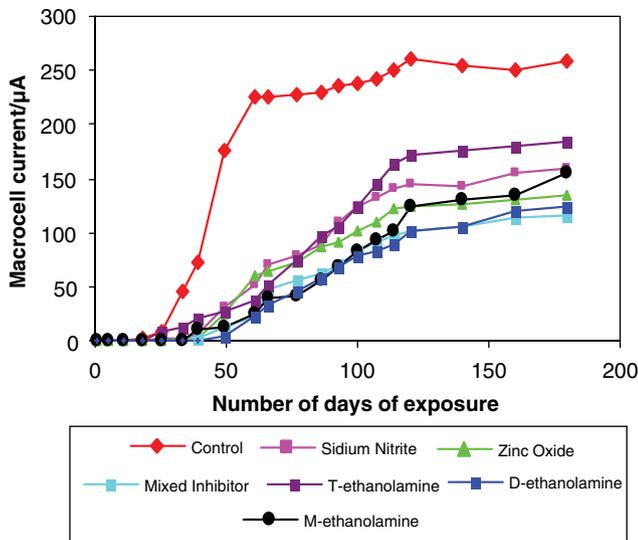


Fig. 13. Macrocell current vs. number of days of exposure for 2% inhibitor admixed concrete under macrocell condition.

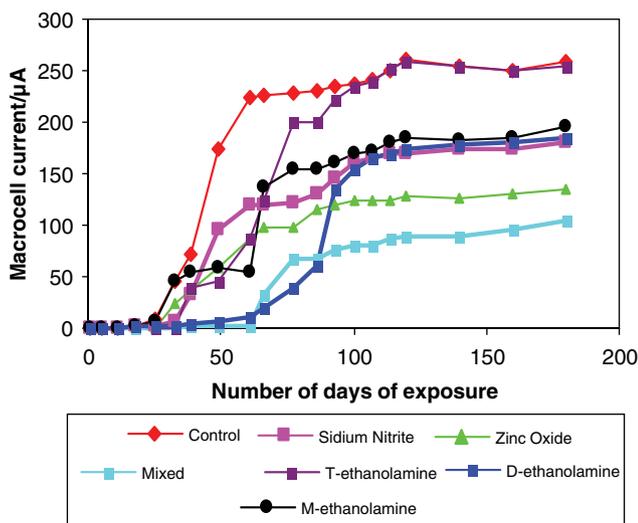


Fig. 14. Macrocell current vs. number of days of exposure for inhibitor-admixed concrete under macrocell condition.

the figure it is seen that up to 40 days of exposure all inhibitors show a negligible macro cell current, indicating that the chloride has not reached the embedded rebar. But the control concrete shows a current of 75 µA even after 40 days of exposure. When compared to control concrete all the type of inhibitors show a lower current, indicating the lower activity of the steel rebars.

Zinc oxide and mixed inhibitor systems performed well in all the investigations, which may be due to the reason that zinc oxide acts as a cathodic inhibitor. When used in water it can precipitate compounds both on the cathodic and anodic areas, as well as within the concrete itself. It can be expected that zinc oxide reacts with concrete and forms $(Ca(Zn(OH)_3)_2 \cdot 2H_2O)$, causing passivation of steel in concrete [15]. This product

Table 1
Gravimetric weight loss of rebar due to corrosion

System	Corrosion rate (mmpy)		
	1%	2%	3%
Control	0.0093	—	—
NaNO ₂	0.0023	0.0093	0.0250
ZnO	0.0022	0.0023	0.0024
Mixed	0.0009	0.0012	0.0037
Triethanolamine	0.0117	0.0038	0.0071
Monoethanolamine	0.0171	0.0042	0.0096
Diethanolamine	0.0203	0.0034	0.0083

formation helps to reduce the porosity and maintain the passivity of steel in concrete when it is exposed to a medium of high chloride ion content. Sodium nitrite is an anodic inhibitor, which forms a stable passive layer even in the presence of chloride ions.

3.8. Gravimetric weight loss method

The average corrosion rate calculated in mmpy for rebar embedded in concrete after 180 days of exposure in 3% NaCl solution with different type and percentage of added inhibitors from 1% to 3% by weight of cement are given in the following table.

Table 1 shows the weight loss measurements of different inhibitor added systems at various percentages. From the table it is observed that in 1% inhibitor addition, mixed, zinc oxide and sodium nitrite exhibit a lower corrosion rate values than the control and other systems. With 2% inhibitor addition also, both zinc oxide and mixed systems show the lowest corrosion rate values of 0.0023 and 0.0012 mmpy, respectively. With 3% inhibitor addition also all the systems show higher corrosion rates than 1% and 2% added systems. On the basis of gravimetric measurements, the optimum percentage was found to be 2%. When comparing all the inhibitors zinc oxide and mixed systems are found to perform better than the other systems.

4. Conclusions

The following conclusions can be drawn from the above investigations:

- Compressive strength measurements revealed that, all the inhibitor admixed concretes at 2% addition level showed a maximum compressive strength.
- Zinc oxide and zinc oxide + sodium nitrite admixed concrete systems have shown a higher compressive strength values than the other systems.
- Splitting tensile and bond strength measurements revealed that the tensile strength of the concrete is not at all affected by the addition of inhibitors.

- Chloride diffusion studies revealed that all the inhibitor admixed concretes are ranked as very low permeability concretes.
- Impressed voltage test revealed that the inhibitor admixed concrete has taken more time to cracking, instead the control concrete cracked in 72 h.
- Open circuit potential measurements revealed that the zinc and mixed inhibitor added systems are found to perform better than the control and other inhibitor added systems.
- Macro cell current measurements also revealed that the, inhibitor admixed systems have shown lower macro cell current compared to the control concrete.
- From the results it is observed that the optimum percentage addition of inhibitor is found to be 2% by weight of cement.
- The results obtained from this study are in good agreement with the results of other researchers [20]. It has been reported that when ZnO was added, the steel remained in passive state reducing Cl^- content at reinforcement level, which allows $\text{Ca}(\text{NO}_2)_2$ to act efficiently on the reinforcement, passivating it.

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