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Durability of Concrete Incorporating Corrosion Inhibitors Exposed to a Salt Solution of $CL^{-}+SO_4^{-2}$

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Abstract:

Laboratory investigations were performed in order to assess the effectiveness of three types of inhibitors, calcium nitrite, ethanolamine and Sika ferro gard 901 (commercial inhibitor) with 1%, 2% and 3% concentration by weight of cement for each inhibitor to retarding corrosion of steel embedded in concrete. Concrete specimens were used to assess the effects of corrosion inhibitors on the compressive and tensile strength of concrete and corrosion of reinforcement.

Some of the specimens were subjected to wetting and drying cycles and reinforcement corrosion was evaluated by measuring corrosion potentials and corrosion current density. Other concrete specimens were immersed in the salt ($Cl^{-}+So_3^{-2}$) solution and reinforcement corrosion was accelerated by impressing an anodic potential of +12 V from a DC power supply and measuring the time-to-cracking of the concrete specimens. The results indicated that the concrete specimens incorporated corrosion inhibitors of calcium nitrite and Sika ferro gard 901 did not adversely affect the compressive and tensile strength of concrete. Furthermore, the time-to-cracking in specimens contains those two inhibitors (calcium nitrite and Sika ferro gard 901) were higher. Two percent of calcium nitrite followed by three percent of Sika ferro gard 901 were efficient in delaying the initiation of reinforcement corrosion and reducing the rate of reinforcement corrosion current density in the concrete specimens, while all the percentages of ethanolamine corrosion inhibitor were ineffective to delay corrosion of the rebar under the conditions of the study and it's adversely affect the strength.

Keywords: concrete, strength, corrosion inhibitors, corrosion potential, current density

ديمومة الخرسانة المخلوطة بمثبطات التآكل والمعرضة إلى محلول ملحي من الكلوريدات والكبريتات

المستخلص: فحوصات مختبريه أنجزت لتقييم فعالية ثلاثة أنواع من المثبطات (نتريت الكالسيوم و ايثانول امين و سيكا فيرو كارد 901 (مثبط تجاري)) بنسب 1%و2%و3% من وزن السمنت لكل مثبط لتأخير التآكل في الحديد المطمور في الخرسانة. تم استخدام نماذج خرسانية لتقييم تأثير مثبطات التآكل على مقاومة الانضغاط ومقاومة الشد وتأكل حديد التسليح.

تم تعريض بعض النماذج إلى دورات من الترطيب والتجفيف لغرض تقييم التآكل في حديد التسليح من خلال قياس فرق جهد التآكل وقياس كثافة تيار التآكل (بطريقة فقدان الوزن). نماذج اخرى غمرت في المحلول الملحي الحاوي على أملاح الكلوريدات والكبريتات وتم تسريع التآكل في حديد التسليح عن طريق تسليط فرق جهد أنودي بمقدار 12 فولت وحساب وقت ظهور التشققات في النماذج الخرسانية. أشارت النتائج إلى أن النماذج الخرسانية الحاوية على مثبطات التآكل نتريت الكالسيوم وسيكا فيروكارد 901 لم توثر سلبا على مقاومة الانضغاط ومقاومة الشد. بالاضافة الى ذلك ، فان وقت ظهور التشققات في النماذج الحاوية على هذين المثبطين (نتريت الكالسيوم و سيكا فيروكارد 901) كان أعلى . استخدام 2% من نتريت الكالسيوم و3% من سيكا فيروكارد 901 كان الأكثر فعالية في تأخير بدء التآكل وكذلك في تقليل كثافة تيار التآكل لحديد التسليح في النماذج الخرسانية، بينما كانت جميع النسب المستخدمة للمثبط ايثانول امين غير فعالة في تأخير التآكل تحت ظروف الدراسة و له تأثير ضار على المقاومة.

1. Introduction

Corrosion of steel in concrete is one of the major causes of premature deterioration of reinforced concrete structures, leading to structural failure and the useful service-life of the structures is drastically reduced because of this phenomenon[1]. When these structures are exposed to aggressive substance containing chloride, the corrosion faults are frequently induced by the presence of chloride ions, these ions cause localized breakdown of the passive film that initially forms on the steel due to the high pH of the concrete interstitial electrolyte. Once corrosion has initiated accumulation of corrosion products occurs on the steel surface. Since these products occupy a volume several times larger than that of the original steel[2], thus, the result is, an increase of the internal tensile stresses that generally induce cracking and spalling of the concrete cover. This situation facilitates further intrusion of aggressive agents and the consequent acceleration of the corrosion process.

There are several sources of chlorides, chlorides incorporated in the concrete when it is mixed (e.g. From salty aggregated and salty mixing water) and chlorides penetrating into concrete from the environment (e.g. From sea water, salty ground water and sea spray) [3]. To minimize the corrosion processes a number of procedures can be assessed such as coating of concrete surface, surface treatment of the rebars, cathodic protection, chloride extraction and use of corrosion inhibitors.

ACI 116R-85 defines a corrosion inhibitors as a chemical compound, either liquid or powder, can be mixed within the fresh concrete as an admixture, usually in very small concentrations, in order to reduce the risk of steel corrosion in reinforced concrete[4].

The corrosion inhibiting admixtures should not be as an alternative to the design specifications for durable concrete, but to increased protection against corrosion [5]. Corrosion inhibitors can be divided into three types: anodic (e.g. Calcium nitrite, Sodium nitrite, Sodium benzoate, Sodium chromate), cathodic (e.g. sodium hydroxide, sodium carbonate, Phosphates, silicate and polyphosphates), and mixed (e.g. amine and aminoalcohol), depending on whether they interfere with the corrosion reaction preferentially at the anodic or cathodic sites or whether both are involved[6].

Several studies have been accomplished to evaluate the effectiveness of chemical admixtures in inhibiting reinforcement corrosion[7–9]. Early studies were concentrated on sodium nitrite, potassium chromate, sodium benzoate, and stannous chloride. Later work concentrated mainly on calcium nitrate. Craig and Wood[7], 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[8] who found that sodium nitrite inhibited corrosion of steel bars in the presence of chlorides, whereas sodium benzoate did not. Rosenberg et al. [10], 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 a 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.

Tomazawa et al. [11], also supported the effectiveness of calcium nitrite as a corrosion inhibitor in concrete. In accelerated tests with wetting and drying cycles at 80°C, calcium nitrite was found to be an effective inhibitor for long-term exposures even in marine environments.

Collins et al.[12], evaluated several inhibitors including (1) a calcium-nitrite-based inhibitor, (2) a monofluorophosphate-based inhibitor, (3) sodium tetraborate, (4) zinc borate (5) a proprietary oxygenated hydrocarbon produced from an aliphatic hydrocarbon, (6) a proprietary blend of surfactants and amine salts (MCI2020 which migrate through concrete), and (7) a proprietary alkanolamine inhibitor (MCI 2000). The results of the study, which involved monitoring of corrosion, compressive strength and resistivity, showed that the calcium-nitrite-based inhibitor was the most promising to mitigate corrosion in a repaired structure after removal of chloride-contaminated old concrete. On the other hand, both borate compounds were found to retard the setting of Portland cement.

Prowell et al.[13], evaluated some of the inhibitors studied by Collins et al.[12] and conducted ponding experiments where they monitored corrosion by measuring the half-cell potential, linear polarization resistance, and chloride ion concentration for a period of 325 days. They reported that two proprietary inhibitors Alox 902 and MCI 2020 were the best performers.

Berke and Hicks [14], published long-term data to show the levels of chloride that a given level of calcium nitrate can protect. They also indicated that once corrosion initiates, the rates are lower with the addition of calcium nitrate.

Jamil et al.[15], conducted electrochemical impedance measurements in order to obtain information on the corrosion behavior of reinforcing steel in the presence of a penetrating amino-alcohol corrosion inhibitor. The investigation was performed in solutions contaminated with chlorides, in the presence of the inhibitor. The electrochemical results indicated that the inhibitor is able to penetrate through mortar, minimizing steel corrosion.

The significance of using corrosion inhibitors in aggressive exposures is ascribable to the fact that data are lacking in the performance of reinforced concrete that is subject to both chloride and sulfate salts.

This investigation was conducted to evaluate the effectiveness of three different types of corrosion inhibitors in reducing reinforcement corrosion in concrete subjected to chloride and sulfate salts with percentage equivalent to those present in soil and underground water in the southern parts of Iraq. The performance of the selected inhibitors in reducing reinforcement corrosion was evaluated by adopting various techniques.

2. Experimental Works

2-1: Materials and Mixes

Sulfate resistance cement (Type V) was used, the chemical composition and physical characteristics are given in tables 1 and 2, which indicate compliance with the requirements of Standard Iraqi Specification 5/1984[16].

Table 1: Chemical composition of cement*

Chemical components	CaO	Sio_2	Al_2O_3	Fe_2O_3	**Lime Saturated Factor**	MgO	SO_3	L.O.I	**Insoluble residues**	C_3A
Used cement, %	62	24	4	5	0.75	4.8	1.78	2.7	0.92	2.15
Limit of Iraqi specification No.5/1984, %	-	-	-	-	0.66-1.02	≤ 5	≤ 2.5	≤ 4	≤ 4	≤ 3.5

Table 2: Physical characteristics of cement[*]

Physical properties	Test results	Limit of Iraqi specification No. 5/1984, %
Setting time, min. -Initial setting	121	≥ 45
Compressive strength, MPa		
3 days	19	≥ 15
7 days	23.5	≥ 23

[*]: This test was done by construction laboratory at College of Engineering- University of Thi Qar.

Natural sand of 4.75 mm maximum size complying with the Standard Iraqi Specification 45/1984[17] has been used. The sieve analysis is given in table 3. The coarse aggregate is crushed gravel with a maximum size of 20 mm has been used and its comply with Iraqi specification 45/1984, table 4. Tap water was used for mixing and curing operations**.** Steel deformed bars, φ 12 mm, conforming to ASTM-A615/A 615-06[18] specifications were used. The mechanical properties of steel are shown in table 5.

Table 3: Grading of sand

Sieve size, mm	10	4.75	2.36	1.18	0.6	0.3	0.15
Percentage passing	100	99	90	75	52	15	2
Limits of Iraqi Specification No. 45/1984 (Zone 2)	100	90-100	75-100	55-90	35-59	8-30	0-10

Table 4: Grading of gravel

Sieve size, mm	37.5	20	10	4.75
Passing, %	100	99	57	2
Limits of Iraqi Specification No. 45/1984 (5-20) mm	100	95-100	30-60	0-10

Table 5: Mechanical properties of steel bars*

Test results for steel bars		ASTM-A615/A 615-06 requirements
Nominal diameter, mm	12	-
Yield strength, MPa	566	≥ 420
Tensile strength, MPa	637	≥ 620
Elongation,%	10	Min. 9

*: This test was done by construction laboratory at College of Engineering- University of Thi Qar.

The mix proportion by weight of cement, fine and coarse aggregate were (1:1.72:2.8). The ingredient proportions are kept constant throughout the work. The cement content was 390 kg, w/c ratio was 0.52 for all concrete specimens.

Three types of inhibitors were used in this work (calcium nitrite (inorganic compound), ethanolamine(organic compound) and sika ferro gard 901(combination of organic and inorganic inhibitors)) with percentage of 1%, 2% and 3% by weight of cement for each inhibitor.

2-2: Preparation of The Salt Solution (Cl^{-} + So_4^{-2})

Pure NaCl, $CaCl2.2H2O$ and $MgSo4.7H2O$ with concentration of 4.5%, 0.55% and 1.79%, respectively, were used in preparing the solution to give a concentration of Cl^{-} equal to 30000 ppm and So_4^{-2} equal to 7000 ppm to simulate sulfate and chloride salts in soil and underground water in the southern parts of Iraq according to the report of the National Center for Geological Survey [19]. Potable water was used as a solvent for these salts.

2-3: Specimens Details

100 mm x 100 mm x 100 mm concrete cubes were prepared for compression strength, concrete cylinders 150 mm diameter and 300 mm high were cast for tensile strength and reinforced concrete specimens of 100 x 100 x 100 mm with a centrally placed 12 mm diameter reinforcing steel bar of 50 mm long were cast for electrochemical measurements (corrosion assessment). Copper wire is connected to the steel rebar embedded in concrete for electrical connections. The specimens were cast with and without inhibitors ranging from 1% to 3% by weight of cement. Three specimens for each percentage of the corrosion inhibitors were tested, the specimens were covered with a nylon sheet to minimize evaporation during 24 hours after casting. Thereafter, the specimens were removed from the mould and cured in potable water for 27 days.

3. Techniques Used

3-1: Compressive Strength

After curing (24 hr. in air and 27 days in potable water) the specimens of compressive strength were tested according to BS 1881: Part 116: 1983[20].

3-2: Tensile Strength

ASTM C496-96[21] was adopted to test the concrete specimens for tensile strength after curing.

3-3: Half-Cell Potential (Corrosion Potential)

In normal conditions, the evolution of salt ingress is slow and has an evolution rate measured in years. In order to accelerate this process. After 28 days of curing, the specimens were exposed to sulfate and chloride solution using the wetting and drying cycles, consisting of 8 days immersed in the solution followed by 7 days drying period in an open atmosphere. The corrosion potentials were measured according to ASTM C876[22]. Corrosion potentials were measured using a copper– copper sulfate reference electrode (CSE) and a high impedance voltmeter. The positive terminal of the voltmeter was connected to the working electrode (rebar) and the common terminal was connected to the reference electrode (CSE). The cycle in continued for 120 days. From the results, potential with time plot is drawn using the average potentials obtained. According to ASTM C 876 potential values more negative than -350 mV CSE indicate more than 90% probability of corrosion activation. Figure (1) illustrates the test method.

3-4: Accelerated Corrosion

In this technique[23] the reinforced concrete specimens, after 28 days of curing, is placed in the salt solution of ($Cl^{-} + So_4^{-2}$) and accelerate the corrosion of steel embedded in concrete by impressing a +12 V fixed anodic potential from a DC power supply until cracking of the specimens. The steel bar in the concrete specimens was connected to the positive terminal of a DC power supply to be anode. 100 x 100 mm stainless steel plate was placed in the solution tank and connected to the negative terminal of a DC power supply to be cathode.

For each specimen, the time taken for initial crack was recorded. Figure (2) illustrates the electrical connection. Battery, charger power 12V and AC conductor were used to overcome main electrical power outage.

Figure1: corrosion potential measurement

Figure 2: Electrical connection for applying a +12 V fixed anodic potential to accelerate the corrosion

3-5: Corrosion current density by weight loss method [24]

For the determination of corrosion rate by using of (weight loss) measurement, the initial weight of the rebar samples was taken in 4-digit electronic balance. After the curing period was over, all the specimens were completely immersed in chloride and sulfate solution for 8 days and then subjected to drying for another 7 days in open air at room temperature (wetting and drying cycles). All the concrete specimens were subjected to eight complete cycles.

After testing, the concrete specimens were broken and the reinforcing steel bars were removed. The procedure stated in ASTM G1-03[25] was adopted for the cleaning of corroded steel bars and for the determination of mass loss. The corrosion rate was calculated using the following equation given in ASTMG1-03[25].

$$\text{Corrosion rate (mm/year)} = \frac{(K \times W)}{(A \times T \times D)}$$

Where:

K: a constant equal to 8.76×10^4

W: mass loss in grams

A: actual corroded area of steel bar in cm^2 after removal from the specimen and visually examining

T: time of exposure in hours

D: density of steel ($7.85\ g/cm^3$).

Using Faraday's law, the corrosion rate in (mm/year) obtained from mass loss measurement was converted to corrosion current density (μA/cm^2) by assuming uniform corrosion occurred over the steel surface by the following equation [24].

$$\text{Corrosion rate (mm/year)} = \frac{(0.00327 \times a \times Icorr.)}{(n \times D)}$$

Where:

Icorr.: corrosion current density in μA/cm^2

a: atomic weight of iron (55.84 amu)

n: no. of electrons exchanged in the corrosion reaction (2 for iron)

D = density of steel (7.85 g/cm^3).

4. Results and Discussion

4-1: Compressive Strength

Figure (3) shows the average compressive strength of the control concrete specimens (0% corrosion inhibitor) and those containing the corrosion inhibitors after 28 days of curing. The experimentally obtained results showing the value of compressive strength of the specimens incorporating ethanolamine corrosion inhibitors was less than the control specimens and the decrease was about 3.5-13.8%, depending on percentage of inhibitor, the maximum reduction was about 13.8% when 3% of this corrosion inhibitor was added and that is may be due to the retarding effect of the inhibitor and the air content might be slightly increased. This reduction was also confirmed during another research project [26].

From Figure (3) it is indicated that there is a slight increase in compressive strength when the percentage of Sika ferro Gard 901 corrosion inhibitor increased and that indicates there is no adverse effect on the compressive strength of concrete.

The strength for concrete containing 2% of calcium nitrite corrosion inhibitor was about 10% higher than for control specimens. In 3% calcium nitrite there was a reduction in the rate of increasing. K.Y. Ann et al.[27], indicates that an increase in dosage of corrosion inhibitor may not guarantee the properties of concrete. The cause of the reduction in compressive strength is not well known.

4-2: Splitting Tensile Strength

Figure (4) shows the splitting tensile strength with a percentage of inhibitor addition and it's illustrate the same behavior for compressive strength with slightly different in reduction or increasing in value of tensile strength. With the 1 % inhibitor addition, there was no adverse effect of tensile strength except ethanolamine inhibitor there was a slight reduction than the control concrete. With 2% of calcium nitrite inhibitor addition, the tensile strength was about 5% higher than the control concrete. With the 3 % inhibitor addition, the figure illustrates that, as the percentage of calcium nitrite and ethanolamine inhibitor level increase, the split tensile strength decreases but there is a slight increase with Sika ferro gard 901 inhibitor.

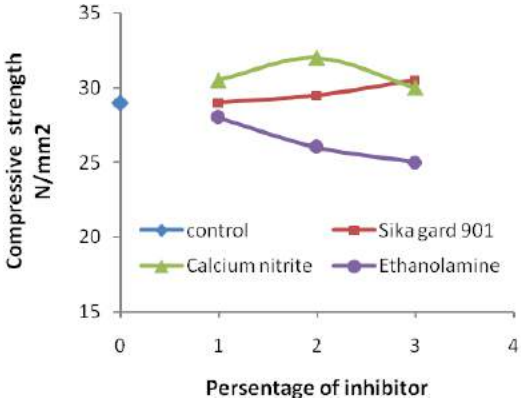

Figure 3: Compressive strength at 28 days of concrete specimens incorporating corrosion inhibitors

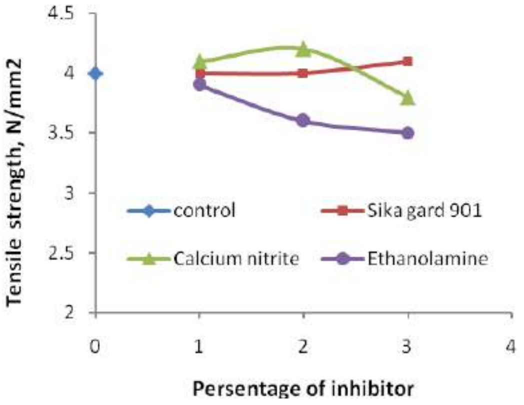

Figure 4: Splitting tensile strength at 28 days of concrete specimens incorporating corrosion inhibitors

4-3: Half-Cell Potential (Corrosion Potential)

The corrosion potentials on steel in the concrete specimens incorporating with 1% of the selected corrosion inhibitors are shown in Figure (5). From the figure it is observed that the corrosion potentials on steel in the all concrete specimens were more negative than – 350 mV CSE after about 30 days of exposure indicating the corrosion activation of the rebar based on the ASTM C 876 criteria.

Figure (6) shows the corrosion potential corrosion with time for 2% inhibitor added concrete in the solution under alternate wetting and drying conditions and it illustrates that concrete specimens containing calcium nitrite were less negative -350 mV CSE indicating the passivity of reinforcing steel even after 120 days of the test period and it was -274 mV CSE. Concrete specimens incorporating Sika ferro gard 901 were showing a more negative potential than -350 mV CSE after 60 days of exposure. Control specimens and that incorporating ethanolamine inhibitor were more negative -350 mV CSE after 30 days of exposure and they were -552 and -605 mV CSE respectively after 120 days of exposure indicating the corrosion activation.

Figure (7) shows the corrosion potential with time for 3% inhibitor added concrete in the salt solution under alternate wetting and drying conditions. In 3% inhibitor added, the control concrete specimens and incorporating with calcium nitrite and ethanolamine are showing a more negative potential than -350 mV CSE after 30 days of exposure indicating the active condition of the rebar while concrete specimens with Sika ferro gard was less negative -350 mV CSE along of the period of exposure. The corrosion potentials on steel in the control, Sika ferro gard 901, calcium nitrite and ethanolamine concrete specimens were -552, -316, -410 and -626 mV CSE respectively after 120 days of exposure. Comparing with control specimens, the potentials in the concrete specimens incorporating sika gard 901 and calcium nitrite were less negative (more positive) than the control concrete specimens.

Figures (6) and (7) can be shown the potentials in the concrete specimens incorporating 3% calcium nitrite were unexpectedly more negative than those incorporating 2% calcium nitrite under the conditions of this study, they were after 120 days of exposure -274 and -410 mV CSE for 2% and 3% calcium nitrite respectively. The same result was obtained by Al-Amoudi et al. [22] when they were used concrete specimens prepared with sea water and incorporating calcium nitrite with 2% and 4%.

Figures (5-7) show that the corrosion potential of ethanolamine inhibitor was decreasing and it's more negative than control when increased the percentage of inhibitor, it was after 120 days of exposure conditions -573, -605 and -626 mV CSE with 1%, 2% and 3% of ethanolamine inhibitor respectively, that is means this inhibitor ineffective to delay corrosion of the rebar under the conditions of the study.

4-4: Accelerated Corrosion

The time to cracking of control concrete specimens and those incorporating with corrosion inhibitors are shown in figure 8. From this figure, it can be observed that the time to cracking is maximum in the Specimens incorporating calcium nitrite and Sika gard 901 corrosion inhibitors addition, they were ranged (90-98) hours, indicating the low permeability of the concrete when compared to control and ethanolamine specimens. There was no clear effect to the percentage of inhibitor.

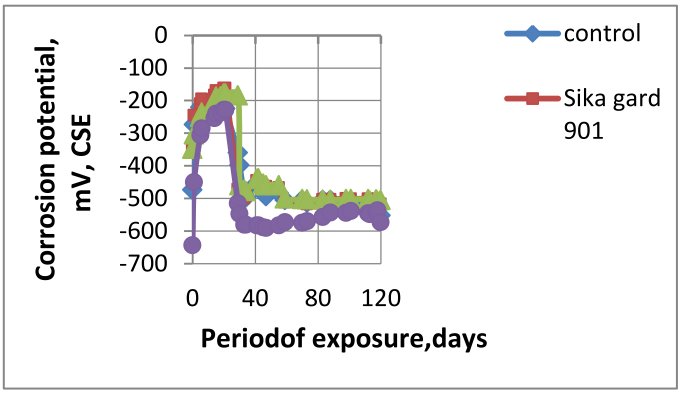

Figure 5: Corrosion potential on steel in the concrete specimens incorporating with 1% inhibitor

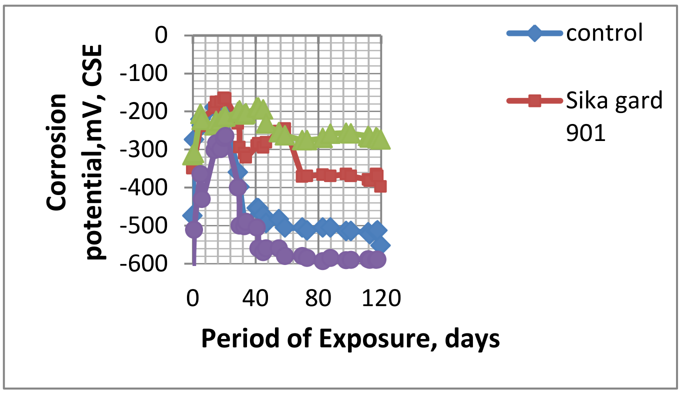

Figure 6: Corrosion potential on steel in the concrete specimens incorporating with 2% inhibitor

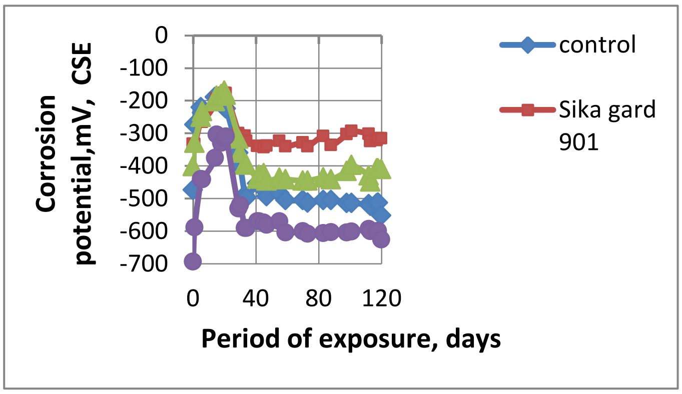

Figure 7: Corrosion potential on steel in the concrete specimens incorporating with 3% inhibitor

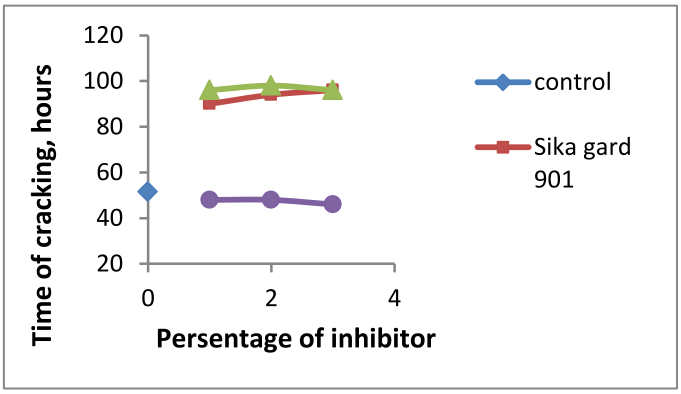

Figure 8: Time of cracking for concrete specimens incorporating inhibitors

4-5: corrosion Current Density by Weight Loss Method

The average corrosion current density calculated in µA/cm^2 for rebar embedded in concrete after 120 days of exposure in the solution with different type and percentage of added inhibitors from 1% to 3% by weight of cement are shown in figure (9).

Figure 9 shows the corrosion current density (Icorr) values in the concrete specimens with 1% corrosion inhibitors were in the range of (0.714–0.886) µA/cm^2 while in the concrete specimens with 2% corrosion inhibitors were in the range of (0.602–0.963) µA/cm^2. The Icorr values in the concrete specimens with 3% corrosion inhibitors were in the range of (0.696–0.963) µA/cm^2 and 0.911 µA/cm^2 for control. The effective corrosion inhibitors were calcium nitrite and Sika ferro gard 901 in the study conditions, the minimum Icorr values were noted in the concrete specimens with 2% calcium nitrite inhibitor, followed by those incorporated with 3% Sika gard 901 inhibitor, The decrease in the Icorr was 33.9% and 23.6%, respectively, comparing with control specimens.

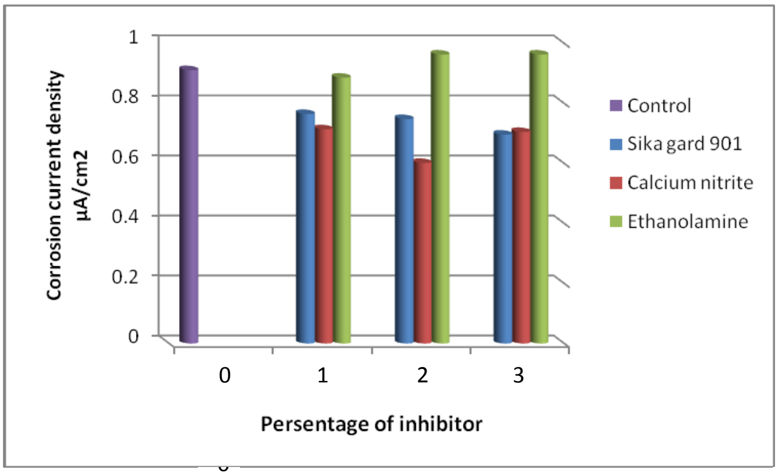

Figure 9: Corrosion current density for concrete specimens incorporating inhibitors

5- Conclusions

1. There is no adverse effect on the compressive and tensile strength of concrete specimens incorporating calcium nitrite and Sika ferro gard 901 corrosion inhibitors. The results showing maximum increasing were about 10% and 5% in the compression and tensile strength respectively, when 2% of calcium nitrite inhibitor was used to compare with the control specimens and there was a slight increase with specimens containing Sika ferro gard 901.
2. Corrosion potential studies revealed that the best performance was shown by 2% of calcium nitrite corrosion inhibitor followed by 3% of Sika ferro gard 901 corrosion inhibitor, the corrosion potentials were -274 and -316 mV CSE, respectively, and they were less negative (more positive) than -350 mV CSE indicating the passivity of reinforcing steel.
3. Ethanolamine corrosion inhibitor ineffective to delay corrosion of the rebar under the conditions of the study and there was an adverse effect on the compressive and tensile strength.
4. Calcium nitrite and Sika ferro gard 901corrosion inhibitors increased the time to cracking of concrete specimens to about 90% higher comparing with control specimens.

5. The minimum corrosion current density values were obtained from the concrete specimens with 2% calcium nitrite inhibitor, followed by those incorporated with 3% Sika ferro gard 901 inhibitor, the percentage reduction in the values of corrosion current density was 33.4% and 23.6% than that in the control specimens, respectively.

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