

Inhibition of Chloride Induced Corrosion in RCC Structures with Calcium Nitrate

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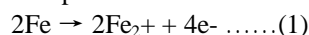
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ABSTRACT: This study attempts to find the inhibition effect of Calcium Nitrate in a chloride excess environment when RCC is most susceptible to corrosion. To achieve these objectives the methodology adopted is accelerated corrosion technique with lollipop casts to accelerate the reactions involved and obtain the results in a shorter period of time. In the first set, the sample with Calcium Nitrate showed 90% inhibition towards chloride induced corrosion while in the second set the sample with Calcium Nitrate showed 100% inhibition towards corrosion which might be due to the lower percentage of NaCl in seawater. For the split tensile strength test, in the first set, the sample with Calcium Nitrate failed at 30.9% more load than the sample without Calcium Nitrate. In the second set, the sample with Calcium Nitrate failed at 18.1% more load than the sample without Calcium nitrate. The reason for the lower tensile strength is the un-bonding of concrete with the corroded rebar due to the expansion of the rust products

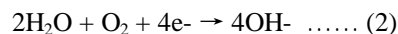
KEYWORDS: Calcium Nitrate; Corrosion Inhibitor; Rebar corrosion.

I. INTRODUCTION

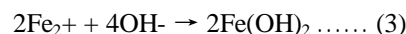
Corrosion of reinforcing steel and other embedded metals is the leading cause of deterioration in concrete. Corrosion is an electrochemical process involving the flow of charges (electrons and ions). At active sites on the bar, called anodes, iron atoms lose electrons and move into the surrounding concrete as ferrous ions. This process is called a half-cell oxidation reaction, or the anodic reaction, and is represented as:



The electrons remain in the bar and flow to sites called cathodes, where they combine with water and oxygen in the concrete. The reaction at the cathode is called a reduction reaction. A common reduction reaction is:



To maintain electrical neutrality, the ferrous ions migrate through the concrete pore water to these cathodic sites where they combine to form iron hydroxides, or rust:



Exposure of reinforced concrete to chloride ions is the primary cause of premature corrosion of steel reinforcement. The intrusion of chloride ions, present in de-icing salts and seawater, into reinforced concrete can cause steel corrosion if oxygen and moisture are also available to sustain the reaction. Calcium nitrite was originally used as a corrosion inhibitor in Japan. The scarcity of suitable aggregates forced the Japanese to use Marine sand in concrete [6]. That sand contained chlorides which promoted corrosion of reinforcing steel. Research by the Japanese led to the use of calcium nitrate which was determined to be an effective corrosion inhibitor that did not adversely affect the properties of concrete unlike calcium Nitrite.

II. RELATED WORK

Corrosion of steel is a global problem for the construction industry causing crores of rupees of loss through repair and maintenance needed to keep the facilities functional. Corrosion is one of the main causes for the limited durability of steel-reinforced concrete. Significant studies have been made into the mechanism of corrosion and different ways to

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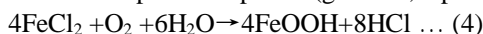
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prevent it. Corrosion can occur due to Carbonisation, Sulphates, Chlorides, Bacteria, Sea Water and many other reasons, but chloride induced corrosion is by far the most common reason for corrosion of rebar and degradation of concrete[05]. Calcium nitrite was originally used as a corrosion inhibitor in Japan the scarcity of suitable aggregates forced the Japanese to use marine sand in concrete. That sand contained chlorides which promoted corrosion of reinforcing steel. Research by the Japanese led to the use of calcium nitrate which was determined to be an effective corrosion inhibitor that did not adversely affect the properties of concrete unlike calcium Nitrite [06].

Detwiler et al. (1991) in the paper revealed that another influence on the pore structure is the temperature that is experienced at the time of casting. High-temperature curing accelerates the curing process so that at young concrete ages, a high- temperature cured concrete will be more mature and thus have a better resistance to chloride ion penetration than a normally-cured, otherwise identical [07]. McGrath. P (1996) in the dissertation report states that the rate of ingress of chlorides into concrete depends on the pore structure of the concrete, which is affected by factors including materials, construction practices, and age[08]. Belaid F et al. (2000) presented the performed X-ray diffraction analysis on steel bars and identified compounds Fe_3O_4 , Fe_2O_3 , γ -FeOOH (lepidocrocite) which are responsible for the passive film formed on iron in alkaline media, such as the one in concrete [09]. Bertolini et al. (2004) study established that there are three main types of pores are identified: gel pores (ranging from 1 to 10 nm); capillary pores (ranging from 10 nm to 10 μ m) and macro pores and air voids (from 10 μ m up to several mm). Because of the small size, gel pores do not significantly contribute to transport processes and usually are not considered with regard to durability of the reinforcement [10].

Nelson (2013) reported evidence of soluble iron-chloride complexes, i.e. green rust, generally represented in the Figure 1 below as $FeCl_2$ (although other forms have been proposed) The mechanism is believed to involve the formation of these compounds at the pit, diffusion to hydroxide and oxygen richer areas and formation of solid rust products, releasing the chloride ions to further attack the passive layer with the rust product $FeOOH$ representing one of several possible species (goethite, lepidocrocite or akaganeite) [12].



Akshata Shetty et al. (2014) have studied the effect of corrosion on the ultimate load carrying capacity of a beam by corroding the beams to different levels of corrosion and then testing them for their ultimate load carrying capacity. They found that the Ultimate Load steadily decreased as the level of corrosion increased [13].

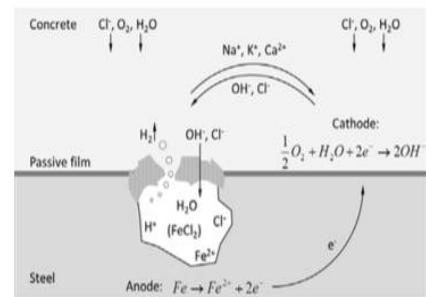


Figure 1 Mechanism of de-passivation by chloride ions

III. OBJECTIVE AND SCOPE

- The inhibition effect of Calcium Nitrate in an environment where Chloride ions are in excess.
- To check if a dosage of 4% Calcium Nitrate would be enough in a chloride excess environment.
- To access if any strength reduction is caused due to the un-bonding of rebar with concrete as a result of corrosion products.

The results to be obtained from this study can be used for further long term studies on Calcium Nitrate and find applications in the construction industry. This study emphasizes on the behaviour of rebar in a chloride excess environment and can be very helpful to predict the rebar corrosion of coastal structures which are constantly exposed to chloride rich water.

IV. METHODOLOGY

The mix design was performed based on design procedure specified in IS 456-2000 and based on the guidelines specified in ACI 544 3R- 1993 for the design of fibre reinforced concrete. ASTM C-143 has been referred to for performing slump cone test. The Mix design is as per IS 10262 – 2009. Four concrete cylinders were cast, two of them with 4% Calcium Nitrate by weight of cement and two of them without. The cylindrical casts were 15cm in diameter and 20cm in height. The samples were divided into two sets of two samples each. Both the sets contained one sample

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with Calcium Nitrate and one sample without Calcium Nitrate. The first set of samples was let dry for a week before accelerated corrosion test was done in a tub with 5% NaCl solution. The second set of samples was kept in the accelerated corrosion tub immediately after removing it from the tank and the test was conducted with seawater from Malpe beach, Udupi.

• **Mix Design Procedure**

The Mix Design is based on standard mix design procedure for M30 concrete as per IS10262-2009. The target strength was 42 MPa and the slump obtained was 8 mm. The mix proportions of specimen prepared as shown in Table 1.

Table 1: Mix Proportions

Max nominal aggregate size	20 mm	Max water content (10262-table 2)	186 litres
Min cement content	310 kg/m ³	Max cement content	540 kg/m ³
Target strength mean	42 N/mm ²	Characteristic strength	30 N/mm ²
Water cement ratio	0.42	Cement content (160/.42)	380 kg/m ³
Volume of coarse aggregate (Table 3 of IS 10262)	62%	Volume of fine aggregate	38%

Table 2: Mix Results for 1 Cubic meter concrete

1.	Vol of concrete	1.0 m ³
2.	Vol of cement 0.12 m ³	380 kg
3.	Vol of water 0.16 m ³	160 kg
4.	Vol of all in aggregates	0.718 m ³
5.	Vol of coarse aggregate – 0.445	1283 kg (20 mm = 924 kg, 10mm = 359 kg)
6.	Vol of fine aggregate – 0.273	711 kg
7.	Calcium chloride should be 4% weight of cement	6.4 kg

The mix design results achieved for M30 grade of concrete as shown in Table 2. The aggregates and cement were first weighted in the lab and then mixed with a trowel when dry and then the water was added slowing from a measuring cylinder. Once the concrete was mixed properly it was poured in the cylindrical cast in three stages of layers/tamping cycles with the reinforcement in the center and then kept on a vibrating machine for about 30 sec. If the level of the concrete exceeded what was needed (20 cm) then some of the concrete was removed and the cast was vibrated again.

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4.1 Material properties

4.1.1. Cement

Cement used for the mix was JK Cement brand OPC grade 53 cement. The properties of cement are as shown in Table 3.

Table 3: Properties of Cement

Sp. Gravity	3.21
Fineness	165 kg/m ²
Initial Setting Time	60 min
Final setting time	325 min

4.1.2 Aggregates

Fine aggregate available in the lab was used. The physical properties of fine aggregate like specific gravity and fineness modulus are tested in accordance with IS :2386 – 1963. Coarse aggregate available in the lab was used. The physical properties of fine aggregate like specific gravity and fineness modulus are tested in accordance with IS :2386. The results shown in Table 4.

Table 4: Properties of Aggregates

Properties	Fine aggregate	Coarse aggregate
Sp. Gravity	2.69	2.83
Angularity	-	5
Fineness Modulus	2.90	7.19

4.1.3. Water

This is the least expensive but most important ingredient of concrete. The water, which is used for making concrete, should be clean and free from harmful impurities such as oil, alkali, acid, etc., in general, the water, which is fit for drinking should be used for making concrete. The water added to the mix was calculated by taking into account the saturated surface dry condition of the aggregate. The water was mixed in the ratio 70% to 30%. 70% of the water was added during the initial dry mixing and the remaining 30% was added during the later stages along with calcium nitrite if added.

4.1.4. Calcium Nitrate

Calcium nitrate was obtained from a chemical dealer in Udipi. It is in tetra hydrate form. 4% by weight of cement was used in the RCC.

4.2 Mixing procedure

The materials were weigh batched correct to two decimal places. A pan mixer was used for mixing. The dry ingredients were dry mixed for 1 minute. After dry mixing water was added gradually to the mixer to attain the required workability. Calcium Nitrite was added uniformly to the mix in the last one minute of mixing.

4.3. Tests conducted

4.3.1. Fresh state of concrete

A. Slump test: Slump test was conducted based on the method specified by ASTM C-143 [4]. The slump cone was filled in three layers with each layer tamped 25 times using a tamping rod and the surface was levelled by rolling the tamping rod over the top surface of the cone. The cone was removed vertically and the drop in height of the concrete was measured and is shown in Figure 3.



Figure 3 Slump cone test

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4.3.2. Hardened state of Concrete

A. Accelerated Corrosion Testing In the accelerated corrosion test, corrosion is measured in an electrochemical way. The first set of samples was dried for a week before accelerated corrosion test was conducted on them. The samples after drying were placed immersed in a 5% NaCl solution in a tub. Two steel bars were kept in the tub which act as cathode. As shown in Figure 4 the positive terminal of a DC regulated power supply is connected to the rebars of the samples and the negative terminal is connected to the steel bars. A constant potential of 2.5 V is applied so as to produce a current of 10 mA flowing through the two samples. The concrete reinforcement bar acts as anode in the electric system and will attract the anions OH⁻, Cl⁻. The steel bar is the cathode of the system and O₂ reduces to OH⁻. The NaCl solution and the concrete cover realize the electrolytic environment. The electric potential forces the anions to the anode and the cations Na⁺, H⁺ to the cathode. By this means, the chemical reactions, which appear in the traditional corrosion process, are accelerated.



Figure 4 Set up of Accelerated Corrosion Test in the Physics Lab

The rate of corrosion is indicated by the amount of current passing for a given potential or the amount of potential required for a given current to pass through the sample. Measurements of potential required to produce 10 mA of current were made every 4 days to compare the rate of corrosion for the two samples. The test was conducted for 12 days post that the casts were subjected to split tensile strength and then rebars examined. For the second set, the samples were directly transferred from the curing tank to the tub, no time was given for the samples to dry. Instead of using NaCl solution, seawater from Malpe beach was used to realize the electrolytic environment. A constant potential of 2.5 V was applied. Measurements of potential required to produce 10 mA were made every day. Split tensile strength test was conducted post accelerated corrosion test. The experimental setup for the accelerated corrosion test is shown in Figure 4.

B. Split Tensile Strength: The split tensile strength test was performed on the samples after the accelerated corrosion test. Test was performed on a compression testing machine of a capacity of 3000 kN. Cylinder sizes of 150 mm diameter and 300 mm height were used for the test. The set up as shown in Figure 5.



Figure 5 Split Tensile strength on without Calcium Nitrate and with Calcium Nitrate

4.4. Mixing Sequence

Base mix design chosen for design was generic M₃₀ design mix, cement content and all other parameters were kept constant, lab temperature ranged from 26°C to 32°C. The pan mixer was used for mixing of concrete mixes meanwhile aggregates and cement were dry mixed for one minute. Water was added slowly while observing the workability of the mix finally 4% of Calcium nitrite were added in the last one minute of mixing.

V. RESULTS AND DISCUSSION

5.1. Accelerated Corrosion Test

Since the D.C Regulator used for the Accelerated Corrosion test had an accuracy of 0.01 A, this meant we could only get values of 10, 20, 30, 40, 50 mA and nothing in between. This gave a room for lot of error, hence instead of measuring current values for a particular voltage, the values of voltage for a given current (10 mA) were measured for both the samples individually to compare corrosion. A voltage of 2.5 V was impressed simultaneously on both the casts to produce a total current of 10 mA flowing through them. This was maintained throughout the period of 12 days. The minimum voltage required for a current of 10 mA to be produced during the 12th day had dropped to 1.4 V. Every 4th

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day the potential needed to produce 10 mA of current flowing through each sample was measured to compare the corrosion rate. The result obtained from test for the first set of samples is given below in the Table 5.

Day	1	4	8	12
Current (mA)	10	10	10	10
Voltage (with Ca(NO ₃) ₂)	5.2V	5V	4.9V	4.9V
Voltage (without Ca(NO ₃) ₂)	5.1V	2.2V	2V	1.9V

Table 5 Results of Accelerated Corrosion Test

The voltage of the sample divided by the current values gives the resistance provided by the sample to corrosion. The

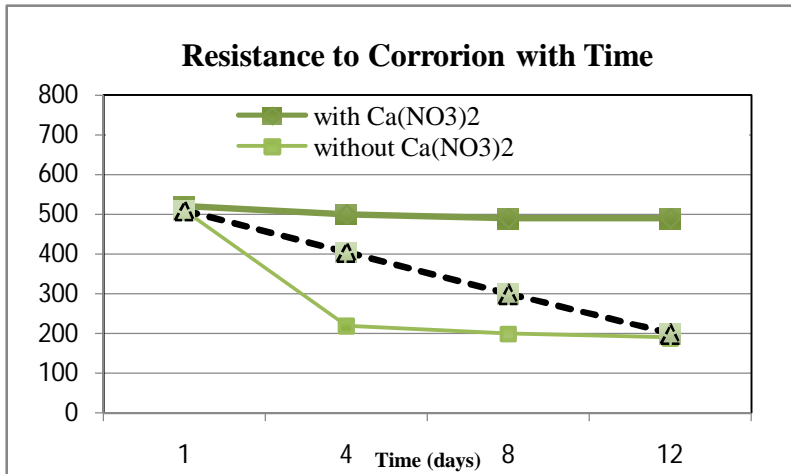


Figure 6 Graph of Resistance to Corrosion vs Time

Figure 6 below shows the resistance provided by the casts with time. The drop in resistance towards corrosion in the sample with calcium nitrate was 30 ohms and in the sample without calcium nitrate was 320 ohms. This implies the inhibitor successfully stopped 90% of the chloride-induced corrosion. We can also deduce from the Figure 6 that the chlorides have broken into the passive film in the first 3 days and hence the lower voltage required for the same amount of current to be produced. The chloride ion transport is driven by permeation through the pores since the samples were used after drying for a week and hence we see such a sudden change in the first 4 days. Visual examination (Figure 6) of both the samples clearly shows the corrosion

products have expanded thus breaking the bond between the concrete and the rebar in the sample without Calcium Nitrate and the sample with Calcium Nitrate has no visible corrosion product and bond between concrete and rebar is intact.

Table 6 – Results of accelerated corrosion of 2nd set

Day	1	2	3	4	5	6	7	8	9	10	11	12
I(mA)	10	10	10	10	10	10	10	10	10	10	10	10
With(V)	5.5	5.9	6.1	6.2	6.1	6.2	6.2	6.2	6.3	6.3	6.4	6.4
Without	5.7	5.9	6	5.9	5.5	4.7	4.5	3.7	3.4	3.1	2.7	2.5

In the first set of samples we observe that the resistance falls drastically in the first 3 days and then has a slow change in the later part of the experiment. But in the 2nd set of samples the change is gradual and only starts taking effect after day 3 observations have shown in Table 6. From the Figure 7 could be explained by the time taken by the chloride ions to reach the rebar surface. In the first set, the samples were let dry for a week before accelerated corrosion was carried out. The pores of the samples of the first set were thus empty and the chlorides rushed into the concrete with the water. But in the second set, the samples weren't allowed to dry and hence were saturated with water. The chloride ion transportation is taking place through diffusion and hence slow. We also notice that the resistance of the sample with

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Calcium Nitrate increases by 90 ohm that indicates that all of the chloride corrosion is inhibited by the Calcium Nitrate and the passive layer has improved during the period.

5.2. Split Tensile Strength

The split tensile strength of both the samples of the first set was checked after the accelerated corrosion test. The sample without Calcium Nitrate failed at 882 kN (90000kg) whereas the sample with Calcium Nitrate failed at 1275 kN (130000kg). The split tensile strength of concrete is given by $0.7 \times f_{ck} \dots \text{N/mm}^2 = 21 \text{ MPa}$

The formula for Split Tensile Strength is given by $T_{sp} = 2P/3.14DL = 18.71 \text{ MPa}$ (Without Calcium Nitrate) and 27.1 MPa (With Calcium Nitrate).

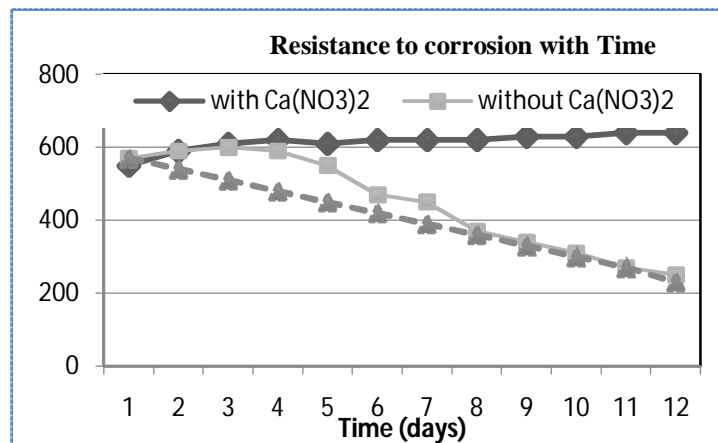


Figure 7 Graph of resistance to Corrosion 2nd set

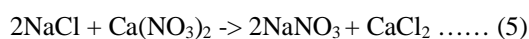
The failure pattern of the sample without Calcium Nitrate as seen in the Figure 5 is a single vertical crack starting at the top and going through the diameter at a considerably lower load suggesting that the concrete was already under strain from the expanding corrosion products and hence making that the weakest part of the cylinder. The failure pattern of sample with Calcium Nitrate shows cracks other than that which goes through the diameter thus suggesting that the bond between rebar and concrete was still strong and not a lot of internal stresses existed.

In the second set, the sample without Calcium Nitrate failed at a load of 892kN (91000kg) and the sample with Calcium Nitrate failed at a load of 1088kN (111000kg). This gives the split tensile strength of the second set of samples as 18.92 MPa (without calcium nitrate) and 23.1 MPa (with calcium nitrate). Although the failure pattern was similar for both the samples of set 2 (multiple cracks including one through the diameter) the strength reduction still suggests that the corrosion has caused significant strength loss.

The strength reduction is 30.9% in the first set and 18.1% in the second set, hence we can conclude that the un-bonding of concrete from the rebar and internal stresses developed contribute to lower strength of the corroded concrete.

5.3. Surface Rust

In the first set, the sample with Calcium Nitrate had developed surface rust. The cause for this could not be ascertained, but has to be directly or indirectly related to Calcium Nitrate, since the sample without Calcium Nitrate has no signs of any surface rust. Calcium Nitrate reacts with Sodium Chloride to form Sodium Nitrate and Calcium Chloride in the following way.



Both Sodium Nitrate and Calcium Chloride are white in color hence the surface rust cannot be both. The water used for the test was sweet water from the lab and

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Figure 8 Sample without Calcium Nitrate and with Calcium Nitrate post accelerated corrosion test.

must contain small quantity of impurities. Such impurities can combine with any one of the 3 substances (Calcium Nitrate, Sodium Nitrate, Calcium Chloride) to form the surface rust seems to be the likely probability.

5.4 Region of Maximum Damage

Examination of the rebars after the casts were cracked open revealed that most of the severe corrosion happened above the surface of the water. The expanding corrosion products which are seen in the Figure 8 only existed for the top 5 cm of the rebar in the concrete. A very obvious realization dawned on us, oxygen was one of the most necessary elements needed for corrosion and was abundantly available above the surface of water. Since the tub of the accelerated corrosion test was stationary, the available oxygen for corrosion under the surface of water was very limited. On the other hand, water from the tub had completely wet the samples and hence was available for corrosion for the part of the rebar above the water level too. Just as a rebar would not corrode if there was no moisture (i.e. water) in the air, it also doesn't corrode if there is no oxygen in the water. Hence the part of the rebar above the water level and in the concrete was an ideal situation for corrosion. Although chlorides were available in plenty below the surface of the water the rebars didn't corrode as much as the part of the rebar above water. This implies that chlorides don't actually cause corrosion but are a reason why it is accelerated by the destruction of the passive layer, and that is what all the theory about chloride corrosion also points to. This has huge implications for coastal structures. The parts of a concrete structure where water, chlorides and oxygen is present are the most susceptible to chloride induced corrosion.

VI. CONCLUSION

Limitations: The results obtained from the accelerated corrosion test are done in an excess chloride environment and hence they don't apply to concrete in general. Calcium nitrate works as a corrosion inhibitor for rebars in concrete, and provides effective protection against chloride induced corrosion specifically.

- From the results of the accelerated corrosion tests we can conclude that 90% of the Chloride Induced Corrosion was inhibited in the tub with 5% NaCl solution by 4% Calcium Nitrate by weight of cement. These samples were let dry for a week after curing, before the accelerated corrosion test was conducted.
- We can also conclude that 100% of the corrosion was inhibited when seawater was used instead of the NaCl solution in the previous case since sea water has 3.5% salt content. Accelerated corrosion test was carried out immediately on these samples after curing, without letting them dry for a week.
- There is an increase of resistance in the sample with calcium nitrate in the one where sea water was used which indicates the passive layer has improved leading to better resistance to corrosion with time, if not destroyed in some other way.
- The split tensile strength test confirms that the sample without calcium nitrate is weaker than the sample with calcium nitrate by 30.9% in the samples, which were corroded in NaCl solution and let dry for a week more after the curing period of 28 days.
- The sample without calcium nitrate is weaker by 18.1% in the samples, which were corroded in seawater and were not let dry for a week after the curing period.
- Further, the failure pattern of the samples without calcium nitrate indicates that the un-bonding and internal

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stresses developed due to the corrosion rust product is the cause of lower split tensile strength.

- This agrees with the theoretical view that chlorides destroy the passive layer and cause localized corrosion and is also confirmed by visual examination.

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