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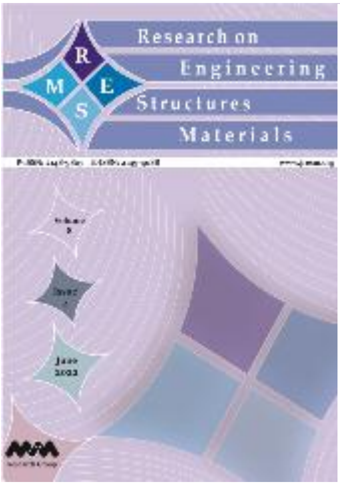

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Research Article

Investigation of the effect of boron inhibitor on reinforced concrete by using the accelerated corrosion technique

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Abstract

Reinforcement corrosion induced by chloride is one of the most common reasons accounting for the premature deterioration of reinforced concrete (RC) structures. In this study, the effects of the use of chemical additives and corrosion inhibitors on the durability properties of the concrete and the potential of reinforcement corrosion were experimentally investigated. In this context, the effects of commercially common inhibitors and green inhibitors due to preventing corrosion and the interactions of these additives with each other were revealed. The reinforced concrete samples were prepared with four different chemical additives and two different corrosion inhibitors. Utilized chemical additives are; set accelerator, set retarder, super-plasticizer, and hyper-plasticizer; also, two different corrosion inhibitors were commercially common calcium nitrite-based corrosion inhibitor, and boron inhibitor which is obtained from natural boron. According to the laboratory experiments, concrete properties were enhanced with the synergistic collaboration of chemical additives and corrosion inhibitors. These effects are more acceptable than if chemical additives are applied individually in the concrete mix. Among all series, the concrete sample containing boron and hyper-plasticizer provided significant corrosion protection by reducing the initial corrosion current by 146,2 %. Moreover, as a result of the accelerated corrosion tests, latest damage occurrence time was calculated at 264 hours for the samples containing boron inhibitors, which is 106 hours for the control sample.

1. Introduction

Corrosion is the chemical and electrochemical decomposition of metals and alloys as a result of various effects of the environment or damage as a result of physical dissolution. Due to the direct and indirect economic losses caused by corrosion, studies for corrosion protection are increasing day by day [1-3]. Metals tend to return to their natural state; therefore, it is impossible to completely eliminate corrosion but this process can be slowed down by taking the necessary precautions [4-9]. In order for reinforced concrete structures to continue their functions during their design life, some corrosion precautions should be taken. Research reveals that a reinforcement exposed to corrosion loses 50 percent of its bearing capacity at the end of 5 years, 90 percent at the end of 15 years, and completely at the end of 24 years [10]. Therefore, even without any external factors, a structure might collapse only with the effect of reinforcement corrosion. Today, corrosion of reinforcement is the primary reason of the destructive effect of earthquakes. A structure that is properly designed and protected from external influences (according to post-earthquake research) can stand statically for many years. The examples given below show that the insidious progress caused by corrosion, in fact, a structure that seems to be strong from the outside may have deteriorated and lost its mechanical strength in the depths.

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On-site images of the earthquake that occurred in Turkey on February 6, 2023 are given on Fig. 1. The damage caused by the corrosion effect on the reinforced concrete structures can be observed. Internal cracks formed as a result of volume expansion caused significantly high corrosion damage. When the site was examined, it was understood that many old buildings were under the influence of corrosion for many years, but this was not noticed by the residents. Since the formation of corrosion cannot be observed clearly from the surface of concrete, no previous corrosion progression applications have been made in the structures that were destroyed in the earthquake.

Fig. 1 On-site images of corrosion formation in earthquake site, Turkey

As a result of the research made on microstructures of concrete, especially in Hatay, some salt elements and sea shells were determined in the concrete mix. This is a shred of obvious evidence that sand was supplied from the sea. The biggest danger of sea sand is that it disrupts the structure of the concrete due to its high salt content. Therefore, concrete cannot preserve the design strength for the intended duration. It has been observed that sea sand causes corrosion of the rebar in the buildings, that is, its diameter decreases, and also causes it to rust and rot. As a result of on-site measurements, it was noticed that while the diameter of the reinforcement was 16 mm in the non-corroded parts of the structure, it decreased to 10 mm in the corroded areas.

There is a necessity for designers to produce high-quality and durable concrete in order to prevent the rapid deterioration of concrete in adverse environmental conditions. However, chlorine ions degrade the natural passive layer on the surface of the steel reinforcement and often cause corrosion of the reinforcement in concrete structures [11]. Various preventive methods such as producing impermeable concrete by increasing the consistency of concrete by using superplasticizers, covering the steel by using organic, inorganic, and metallic coatings, coating concrete with epoxy and resin-based waterproof chemicals, cathodic protection methods, and using corrosion inhibitors (addition to the concrete mix or application to surface) are applied to prevent the corrosion mechanism. In addition, during the production stage of concrete, the resistance of the reinforcement against corrosion could be also increased by using mineral additives that reduce the permeability of the concrete. One of the most commonly used methods to prevent or control corrosion by reducing the corrosion rate is the use of inhibitors [12]. In order for inhibitors to be effective in the concrete they are used in; it is important to choose the most suitable inhibitor and also the inhibitor amount for the building design.

In recent years, many commercial inhibitors, especially calcium nitrite, have been tried in concrete mixtures to protect the reinforcement from corrosion. The majority of researchers are of one mind that corrosion inhibitors used currently in the construction industry are known synthetic chemicals that are toxic to the earth [13,14,15]. For this reason, it has become a need for harmless inhibitors that might be considerably protectable to nature. These demands are significantly increased in recent years so researchers are focusing their studies in this field.

Eyu et al. [16] discussed the anticorrosive performance of Vernonia amygdalina extracts for carbon steel in concrete in 3.5M NaCI solution. Corrosion study was performed by using weight loss tests, corrosion potential calculations, half-cell calculations, concrete resistivity measurements as well as visual analysis. The outcomes acquired were also correlated with commercially available inhibitor like sodium nitrite or calcium nitrites. Phytochemical constituents of Vernonia amygdalina extract were described to contain alkaloids, saponins and tannins. Corrosion tests were illustrated by the plots against weight loss, inhibitor efficiency as well as corrosion rate. Outcomes achieved evidently discussed that Vernonia amygdalina extract showed analogous corrosion resistance when compared to commercially available inhibitors which are sodium and calcium nitrate.

Abdulrahman et al. [17] has broadly discussed and illustrated the anticorrosive behaviour of Bambusa arundinacea (BA) extract for reinforced steel corrosion in concrete. It was investigated to enhance the strength of concrete contaminate by chloride or sulphate environment. The obtained data was correlated with calcium nitrite and ethanolamine inhibitors. Effect of inclusion of $MgCl_2$ and $MgSO_4$ over concrete strength had been estimated and elucidated. Fundamental advantages of present analyses illustrate that inclusion of inhibitor shows negligible adverse effect on concrete durability. Bambusa arundinacea did not comprise and heavy metal that also eliminates the risk of production of dioxin on interacting with chloride contaminated water.

The above-mentioned studies were carried out to promote the use of natural (green) inhibitors rather than commercial inhibitors with known toxic effects such as calcium nitrite and sodium nitrite. In recent years, studies on boron have been increasing rapidly which can be utilized to obtain sustainable and ecological concrete additives. Boron inhibitor, which is obtained from boron and accepted as a green inhibitor, does not harmful to nature and human thanks to the borane complex it contains [18]. The novelty of this study is the investigation of the effects of boron inhibitor on concrete with advanced corrosion techniques. One of the main reasons for choosing a boron inhibitor is that it is derived from natural boron. Toxic compounds that are harmful to nature and living things, which are found in many concrete additives, are not contained by this inhibitor [19]. The acceleration of studies with boron in recent years, the number of components synthesized from boron is increasing [20]. This inhibitor is just one of many compounds synthesized from boron. Considering all these reasons, the effect of boron inhibitor on concrete should be examined.

Boron inhibitor is a colourless liquid containing triethylamine borane complex. This inhibitor is a kind of boronium cation compound. Due to their low reactivity and high stability, their usage areas are increasing [21]. After the synthesis process, it was diluted in water and turned into a concrete additive. In this study, the effectiveness of natural boron inhibitors against reinforcement corrosion was investigated by the application of the accelerated corrosion method. The chosen method while determining the efficiency of the boron inhibitor was making a comparison of green inhibitors with the commercial calcium nitrite inhibitors, which are widely used in the construction industry and have known harmful effects. Nowadays, concrete additives are used in almost every type of concrete. Thus, reactions of these chemicals with new-generation corrosion inhibitors are not clearly known and corrosion mechanism had to be investigated. The aim of this study, is to examine the interactions between different chemical admixtures and to develop a concrete mixture with high corrosion and durability properties.

2. Materials and Measurements

In this section, detailed information is given about the materials used in the production of standard concrete and the mechanical and durability measurements. The materials in the concrete mix are ensured to the standards and also the measuring devices have been calibrated.

2.1. Materials

The dosage of cement in the concrete mix was 350 kg/m^3 and the water-cement ratio was 0,5. The interaction of boron inhibitor and calcium nitrite inhibitor with chemical additives was investigated by determining two different mixture series. Four different chemical additives were mixed with each inhibitor and laboratory experiments were applied consecutively. These chemical additives are set accelerator, set retarder, super-plasticizer and hyper-plasticizer. Chemical additives were added to the mixtures at the rate recommended by the standards. Tap water is used in concrete mixes and the pH of the water is 7,85.

2.2. Sample Types and Curing Conditions

Concrete samples are cylinders of ϕ100x200 mm size. These cylinders have 14 mm diameter rebar at their centres. The samples were cured in the laboratory environment for one day and then they removed from the molds were kept in the curing condition until the relevant experiments were carried out. Reinforced concrete samples were kept in lime-saturated water pools with a temperature of 20 ± 2 ºC until the test time (standard curing).

The samples, whose curing period was completed, were taken out of the pool for accelerated corrosion test on the 28th and 90th days.

Each of the chemical additives used in this article can affect the slump value of fresh concrete. The slump test was carried out in accordance with the TS EN 12350-2 (2019) [22]. The fresh concrete mix was filled into the measuring cup by skewering 25 times in 3 layers. After the swelling of each layer, the air bubbles remaining in the concrete were removed by tapping the edges of the container 10-15 times. The excess part on the container was stripped off. After the cone is removed, the measurement is made between the cone and the top of the concrete mix.

2.3. Compressive Strength Test

The positive effect of chemical additives on the compressive strength of concrete has known. The compressive strength test was used on cubic samples with a dimension of 150 mm. Samples were loaded with a constant velocity of 0,3 MPa/s due to the TS EN 12390-3 (2019) standard [23]. With the help of a 2000 kN capacity concrete press, force was applied to the samples until the crack occurred on the sample.

2.4. Accelerated Corrosion Test

The accelerated corrosion laboratory test has different parts, these are; reinforced concrete, a plastic chamber, water with % 4 content of sodium chloride, a data logger device, and a direct current power source. The sample was produced by embedding 180 mm of 16 mm diameter reinforcement into a cylindrical sample ϕ100x200 mm size. The reinforcement bar (working electrode) is connected to the positive pole of the direct current source, which applies a constant 20 Volt to the system, and plates (counter electrode) are connected to the negative pole [24-29]. The time-dependent corrosion current graphs were drawn according to the damage formation times after the application of experiments was ended. Accelerated corrosion test was applied on the samples in curing conditions at 28^{th} and 90^{th} days [Figure 2].

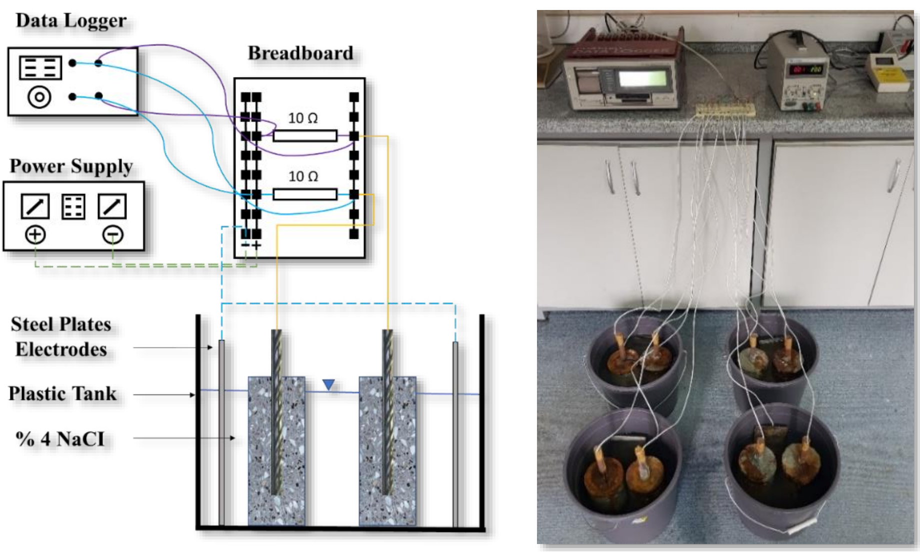

Fig. 2 Reinforced sample during accelerated corrosion; schematic illustration on the left and test setup on right

2.5. Micro Analysis on Concrete

Thanks to its natural compounds, the boron inhibitor creates a less porous microstructure in both early and late ages. Figure 3a shows ettringite formation in the microstructure of the control sample without inhibitor cured for 28 days. The boron inhibitor improved the hydration reactions occurring in the cement and caused the formation of non-pointed and denser ettringite. As evident from the SEM image on Figure 3b, the reactional activities which occurred by boron inhibitor and hyper plasticizer also contribute to the flowability of the concrete mix and improve the micro-filling ability.

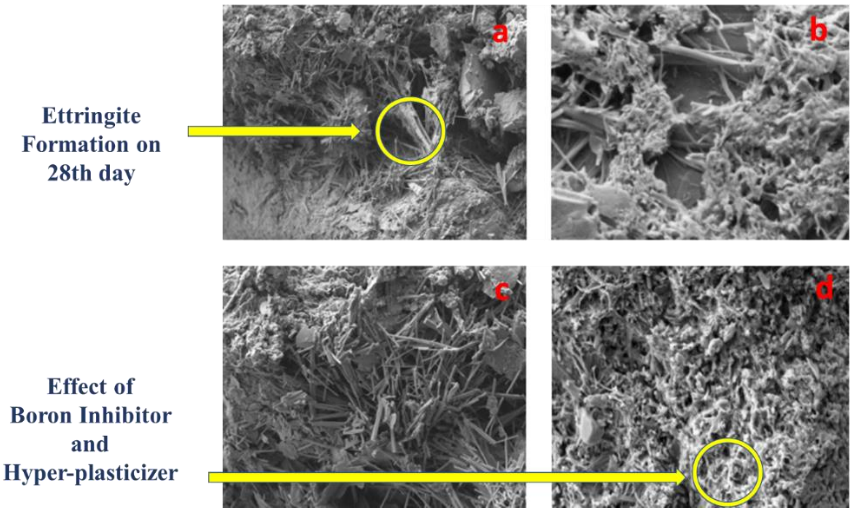

Fig. 3 a) Ettringite formation of the control sample on the 28th day b) Control sample on 90th day c) Effect of boron inhibitor and hyper-plasticizer on 28th day d) Sample with boron inhibitor and hyper-plasticizer on 90th day

The cement paste matrix demonstrated in Figure 3c is proof that more ettringites are developed according to 90 days of curing time. Figure 3d is the SEM images of the samples containing boron inhibitor and hyper-plasticizer after 90 days of curing. While the curing time increased, by the time porosity of the concrete samples containing the boron inhibitor started to decrease. By dint of all these, the porosity of the concrete was reduced, resulting in enhanced durability properties for concrete design including compressive strength and damage occurrence time of accelerated corrosion test.

3. Results and Discussion

3.1. Slump Values

In the series with chemical additives, the slump values increased in the series containing super and hyper-plasticizers. Considering the series with boron inhibitor and chemical additives, an increase in slump values was observed compared to the series in which only chemical additives were used. The slump values saw a slight rose when using the calcium nitrite-based corrosion inhibitor, although the amount of water was reduced and added to the mixture. Generally, with the addition of chemical admixtures to a concrete mix, slump values witnessed an increase. Especially, with the use of super and hyper-plasticizers in concrete series, the slump values rose significantly [Figure 4].

Since corrosion inhibitors are liquids with high water content, they affect the water/cement ratio of the concrete to a great extent, which might reduce the strength. The water/cement ratio was kept constant by reducing the amount of water with the added inhibitor value, however, the slump value of the BORIN series increased by 11% compared to the control concrete. The slump value of the FERRO series increased compared to the control series but remained lower than the BORIN series.

In the series with chemical additives, the set retarders generally did not have a considerable effect on the slump values. However, as a result of the plasticizing effect of the setting accelerator additives, an increase was experienced in the slump values. Utilizing plasticizers was by far the most noticeable result. The slump values increased by approximately 51.4 % in the series containing super and hyper-plasticizers. In the series with boron inhibitor and chemical additives, the slump value of each series increased by an average of 35.2% compared to the series in which only chemical additives were used. The calcium nitrite-based corrosion inhibitor also shows moderate change in slump values. The series used calcium nitrite and set retarder increased the slump value accounting for 17.6% compared to the series with contain only set retarder.

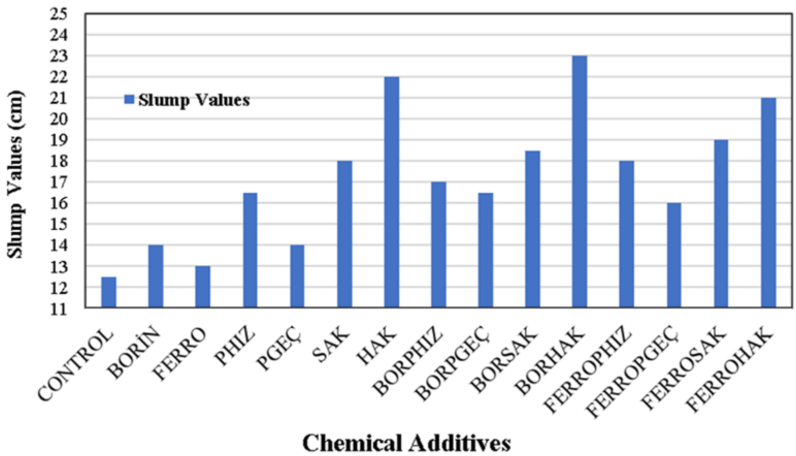

Fig. 4 Variation of slump values according to concrete series

3.2. Compressive Strength Values

In Figure 5, the variation of the compressive strength results of concrete with boron inhibitor and chemical additives over time is shown. When the figure was examined, it was seen that with the extension of curing times, compressive strength values also increased. Thus, hydration reactions occur better, new C-S-H gels are formed, and this affects positively the mechanical strength. As can be seen from Figure 5, it was concluded that the boron inhibitor did not degrade the chemical structure of the concrete (although it was used as a concrete additive for the first time) and it increased the compressive strength values more than the samples with calcium nitrite. The inhibitor obtained from boron did not cause any negative results when used with chemical additives. The use of the two chemicals together provided an increase in the compressive strength of concrete by creating a synergistic effect.

In the concrete samples where the setting accelerator and boron inhibitor were used together, an increase of 11.23% was observed in the concrete compressive strength compared to the concrete samples using only the set accelerator. Within the scope of this article, the highest compressive strength result is 50.4 MPa in the 90^{th} day of curing, which is obtained as a result of the use of hyper-plasticizer additives and boron inhibitor together. Hyper-plasticizer admixtures used together with boron inhibitor increased the concrete compressive strength by 27.59% compared to the control sample, and superplasticizer admixtures used together with boron inhibitor increased the concrete compressive strength by 17.42%. From this, it can be said that the green boron inhibitor increases the mechanical strength of the concrete and this will positively affect the corrosion resistance.

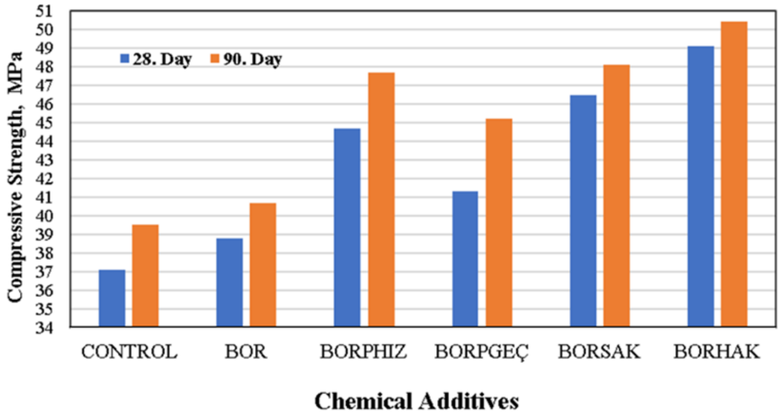

Fig. 5 Compressive strength of boron inhibitor and chemical additives

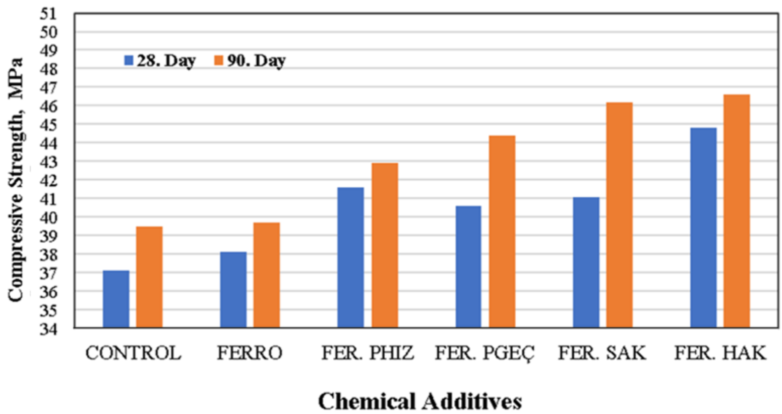

Fig. 6 Compressive strength of calcium nitrite and chemical additives

Figure 6 shows the variation of compressive strength results of concrete with calcium nitrite inhibitor and chemical additives over time. It was concluded that when calcium nitrite inhibitor and chemical additives are used together, they increase the compressive strength of concrete. The hyper-plasticizer admixtures used together with the calcium nitrite inhibitor increased the concrete compressive strength by 11.27%, and the superplasticizer admixtures used together with the calcium nitrite inhibitor increased the concrete compressive strength by 8.61% compared to the control sample. With the increase of curing time, an increase in compressive strength of approximately 6.11% was observed in all series. Although this increase is less than the boron inhibitor, it was seen that calcium nitrite inhibitor had a positive effect on the concrete compressive strength.

Boron inhibitor generally increased the compressive strength of concrete more than the calcium nitrite inhibitor, which is known as the market inhibitor. It was observed that the two inhibitors did not give harmful reactions with other chemicals during use in concrete and it was concluded that it increased the mechanical properties. When calcium nitrite is used in reinforced concrete with other chemical additives, the structure of the concrete mixture does not deteriorate. This is because nitrite components do not react with sulfonate and phosphate-based chemicals.

3.3. Accelerated Corrosion Test Results

After the accelerated corrosion test, the reinforcements inside the samples were corroded and damaged (Figure 7). Since the volume of corrosion products (rust) is 3-8 times larger than the volume of reinforcement involved in corrosion, rust products have created huge forces in the hardened concrete and it results in cracks and crumbles because of these increased internal stresses.

Fig. 7 Final stage of control and BORHAK sample after accelerated corrosion test

The variation of the accelerated corrosion test results of concretes with calcium nitrite inhibitor and chemical additives according to the concrete series is given in Figure 8. As seen in the figure, sudden current increases occurred in corrosion currents at the time of damage. The damage occurrence times of concrete samples were determined from these sudden current increases. It has been observed that the use of calcium nitrite in concrete samples generally reduces the initial corrosion current and prolongs the damage formation time. The FERROSAK90 sample increased the damage formation time by 37.9% compared to the control concrete with 163 hours. On the other hand, the FERROHAK90 sample gave better results, keeping the initial corrosion current at 94 mA and prolonging the damage formation time up to 192 hours by increasing 78.9%.

The rebar used in reinforced concrete structures corrodes rapidly in mediums containing Cl^- ions (especially if the permeability of the concrete is high). The passive film layer formed by the inhibitors generally has a protective effect around the reinforcement and prevents corrosion mechanism from occurring on the reinforcement surface. In the samples where these effects are less visible, the initial corrosion currents also rise. The use of inhibitors in concrete samples gave positive results for both inhibitor types. The nitrite and borane compounds in the inhibitors protected the passive film layer around the reinforcement and the passage of chloride ions was prevented. This result was more prominent in the natural boron inhibitor. As a result, the use of chemical additives and inhibitors improved the corrosion resistance of concretes and extended the damage formation period. The synergistic effect of these chemicals improved the durability properties of concrete and gave better results. In the graph, it was determined that the strength results differ according to the curing time. For instance, the use of super and hyper-plasticizers decreased the permeability of the concrete and extended the first crack formation time at 28th days compared to the 90th day strength of the control sample.

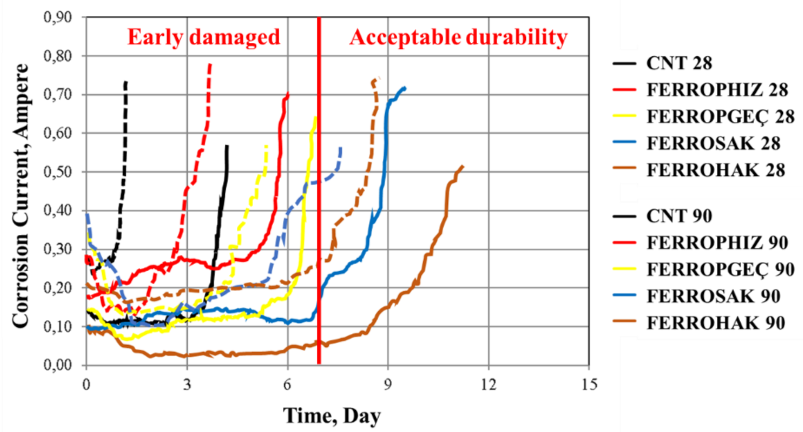

Fig. 8 Time-dependent corrosion current graph for concrete mix with calcium nitrite inhibitor

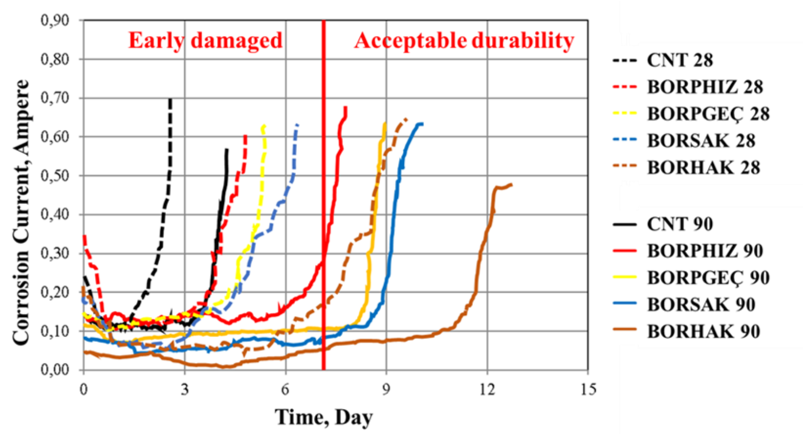

Fig. 9 Time-dependent corrosion current graph for concrete mix with natural boron inhibitor

In this study, the lowest initial corrosion current value and the longest damage formation time obtained from the samples using boron inhibitor (Figure 9). The BORHAK90 sample is the last damaged sample within the scope of the accelerated corrosion test performed with 264 hours, and it showed an increase of two times compared to the control sample. In addition, the BORHAK90 sample draws the lowest initial corrosion current with 47 mA. It can be understood from this that the boron inhibitor does not allow the formation of corrosion reactions in the sample by forming an impermeable compact structure. BORSAK28 sample increased the initial crack formation by 124,92%. The borane complex contained in the boron inhibitor protected the passive film layer around the reinforcement, and this synergistic effect between them, together with the reduction of the impermeability of the hyper plasticizer, gave the latest damage formation time (Table 1).

Table 1. Corrosion current and damage occurrence time of samples

Series	Corrosion Current (Ampere)		Damage Occurrence Time (Hour)	
	28th Day	90th Day	28th Day	90th Day
Control	289	127	68	106
FERROPHIZ	205	170	102	135
FERROPGEC	152	124	118	146
FERROSAK	131	102	142	163
FERROHAK	106	94	175	192
BORPHIZ	151	123	121	176
BORPGEC	135	112	167	199
BORSAK	109	92	196	209
BORHAK	83	47	227	264

4. Conclusions

In this study, the definition of corrosion mechanism and the importance of the application of newly produced concrete additives (green natural inhibitors) was emphasized rather than traditional inhibitors with known toxic effects on earth. One of the most important compounds obtained from boron in recent years is a boron inhibitor. In this research, the boron inhibitor, which was used to protect the base metal in different sectors, was used as a concrete additive to protect the reinforcement. Combinations of boron inhibitor with other concrete additives significantly reduced the corrosion potential of the reinforcement and extended the damage formation time. As a result of the laboratory tests, it has been seen that the curing time, the chemical additives, and the type of corrosion inhibitor have an important effect on mechanical and durability properties of concretes in various ways.

According to the analysis, the following results were obtained;

- The use of green boron inhibitor in concrete significantly improved the mechanical and durability properties of concrete. Increasing the curing times is absolutely necessary and important.
- It has been observed that the mechanical properties of the concrete are significantly improved when a calcium nitrite-based corrosion inhibitor is used as a chemical additive. The use of calcium nitrite and hyper-plasticizers filled the micro-voids in the concrete (with the help of enhanced consistency) and increased the compressive strength by 21,6% compared to the control sample. Among all results, the highest compressive strength result is 50,4 MPa, which is obtained as a result of the use of hyper plasticizer additives and boron inhibitor together. BORHAK concrete series increased the concrete compressive strength by 27,59% compared to the control sample.
- On the perspective of durability properties, it was concluded that the damage occurrence time obtained as a result of the accelerated corrosion test was prolonged. While the damage formation time was measured as 192 hours with the use of commercial calcium nitrite inhibitor and hyper-plasticizer in concrete mixtures, this time was found to be 264 hours with the use of boron inhibitor and

hyper-plasticizer. Both inhibitors increased the damage formation time compared to the control samples by 81,1% and 146,2%, respectively.

- BORHAK90 sample draws the lowest initial corrosion current with 47 mA. It is understood from this that the boron inhibitor does not allow the formation of corrosion reactions in the sample by forming an impermeable compact structure. BORSAK28 sample increased the initial crack formation by 124,92%. The borane complex contained in the boron inhibitor protected the passive film layer around the reinforcement.
- The efficiency of the boron-containing green inhibitor is way higher compared to the other inhibitors obtained from plant extracts according to the literature review in previous sections. Considering the corrosion protection potential and cost of all other green inhibitors, it is thought that these inhibitors will be more difficult to manufacture and hard to become commercially available. The fact that boron reserves are abundant and can be easily processed, especially in Turkey, makes boron inhibitors to be more utilizable and economical.

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