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REDUCTION OF CHLORIDE ION INGRESS INTO REINFORCED CONCRETE USING A HYDROPHOBIC ADDITIVE MATERIAL

Agus Maryoto[a]*, Buntara S. Gan[b], Han Aylie[c]

[a]Department of Civil Engineering, Jenderal Soedirman University, Jl. Mayjend. Sungkono KM 5, Purbalingga, Jawa Tengah, Indonesia
[b]Department of Architecture, College of Engineering, Nihon University, 1-Nakagawara, Koriyama, Fukushima, Japan
[c]Department of Civil Engineering, Diponegoro University, Jl. Prof. Soedarto, SH, Tembalang, Semarang, Jawa Tengah, Indonesia

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* Corresponding author
agus_maryoto1971@yahoo.co.id

Graphical abstract

Abstract

Reinforced concrete structures located in areas near the coast are exposed to potential corrosion attack. Corrosion attack can occur because of the infiltration of chloride ions into concrete. At the initiation of the corrosion process, the load-carrying capacity of a reinforced concrete member will be affected negatively as a function of time. Infiltration of chloride ions into the concrete could be avoided by improving the micro-concrete properties. Enhancement of these properties can be achieved by use of an additive in the concrete. Calcium stearate is a hydrophobic material that can improve the properties of concrete. To determine the optimum contribution in concrete, laboratory experiments were conducted. Calcium stearate was added in varying contents of 0, 0.2, 0.9, and 1.9% of the cement weight. Four types of tests were performed to analyse the contribution of calcium stearate, namely the compressive strength test, absorption test, chloride ion infiltration test, and corrosion test. The results show that an optimum value of the compressive strength of concrete is obtained through the addition of calcium stearate. Besides that, the absorption, chloride ion infiltration, and corrosion value decrease significantly when a higher amount of calcium stearate is added to the concrete.

Keywords: Corrosion attack, chloride ions, hydrophobic, infiltration, calcium stearate

1.0 INTRODUCTION

Many processes lead to degradation of reinforced concrete structures such as repeated loads, thermal loads, and corrosion of the reinforcement. Reinforced or pre-stressed concrete structures located in corrosive environments are in danger of corrosion attack. Since concrete is a porous material, sulphate, chloride, and other aggressive agents can infiltrate it through the capillary pores and react with oxygen to produce rust on the surface of the steel within the concrete structure. Infiltration of chloride ions is known as one of the most important factors that deteriorate the durability of reinforced concrete structures. Chemically, volume of the corrosive product is greater than the original steel when corrosion occurs on the steel. Therefore, corrosive product induces mechanical stress, causes cracking of the concrete, and finally encourages spalling of the concrete cover. For these reasons, many methods have been developed to protect steel reinforcements from corrosion, such as corrosion inhibitors, coating protection, integral waterproofing, and others.

Protection of reinforced concrete against corrosion by using an integral waterproofing system is a system that is superior when compared with coating systems and membrane systems. An advantage is that such a system is easy to use in the concrete. The waterproofing material is used by mixing it into fresh concrete. Another advantage of this system is that it has the same service life as the normal concrete.

Diffusion of chloride ions and oxygen through the capillaries is an important parameter that determines the corrosion of concrete reinforcement. These capillaries in the concrete are formed during the cement hydration process. Two types of capillaries are present, the first with a diameter of 10–20 nm and the second with a diameter of 1–2 nm [1]. Thomas and Jennings [2] describe the classification of pores in more detail. Pores are classified into two types, namely capillary pores and gel pores. Capillary pores are divided into two sizes, namely large ones of 10 μm to 50 nm and medium ones of 50–10 nm. Gel pores consist of small and micro pores. The diameter of small gel pores is 10–2.5 nm and that of micro gel pores is 2.5–0.5 nm. Once the chloride ions reach the surface of the reinforcement, a split passive protective layer corrosion process eventually begins [3]. Corrosion damage of the reinforcement occurs in not only reinforced but also prestressed concrete. Corrosion attacks the prestressing strand, and as well as reducing the bond with the concrete, also causes a loss of prestress. This does not occur in reinforced concrete structures. When reinforced concrete structure attacked by corrosion, only its bond capacity is reduced. The decline in the bond between the reinforcement and the concrete result cannot work together [4]. Corrosion of the prestressing strand is the main factor leading to the collapse of prestressed concrete structures [5]. It occurs more rapidly than the collapse of reinforced concrete during the corrosion attack.

Previous researchers have attempted to prevent corrosion attack due to the infiltration of chloride ions and nitrate ions in the concrete by using an additive that is mixed directly into the fresh concrete. Furthermore, green inhibitors have been investigated to reduce corrosion attack. These materials are renewable resources such as alkaloids, amino acids, and plants [6]. The use of combination of silica fume and metakaolin in mortar cement also helps to increase resistance to corrosion attack [7]. Benzimidazole compound is applied as an additive for integral waterproofing systems in concrete [8], [9]. The results show that the use of this material reduces corrosion attack on the reinforcement of concrete structures.

The most recent investigations of the effect of calcium stearate in concrete are reported by Maryoto [10]. The author examined accelerated corrosion in reinforced concrete with a calcium stearate dosage of up to 1 kg per cubic metre of concrete. Calcium stearate is a hydrophobic material. When it reacts with cement, the concrete will behave hydrophobically as well. Therefore, the parameters analysed consist of the compressive strength, permeability, and ingress of chloride ions. The results showed that the addition of calcium stearate to concrete increases the concrete's physical properties. Unfortunately, greater dosages have not yet been researched. As a result, the tendencies of the effect of calcium stearate in concrete cannot be defined accurately. Besides, the extent of corrosion of steel reinforcement by the infiltration of chloride ions also cannot be described clearly.

The purpose of this study is to find the contribution of calcium stearate in the concrete to the reduction of the infiltration of chloride ions. Hence, four contents of calcium stearate were investigated: 0, 0.2, 0.9, and 1.9% of the weight of cement. In order to verify the effect of calcium stearate, four tests were carried out in the laboratory, namely compressive strength, chloride ion infiltration, water absorption, and accelerated corrosion tests. The total number of specimens analysed in the study was 48 pieces.

2.0 METHODOLOGY

2.1 Calcium Stearate

Calcium stearate is a hydrophobic material. The most interesting property of calcium stearate is that it tends to repel water without being dissolved previously in the other solution media. Another advantage is that it does not contain chloride or sulfate ions. The characteristics of the calcium stearate are as shown in Table 1.

Table 1 Physical properties of calcium stearate I

No.	Properties	Analysis
1	Chemical formula	$Ca(C_{18}H_{35}O_2)_2$
2	Appearance	White fine powder
3	Melting point	120^{0}C
4	Free fatty acid	Maximum 0.5%
5	Loss on drying	Maximum 2%
6	Metal content	0.65 ppm
7	Specific gravity	0.25

2.2 Mix Proportion of Concrete

Normally, the compressive strength of concrete used for girders, piers, abutments, and other structures exposed to corrosive environments is around 30 MPa on average. The mix proportion of concrete in this study is then considered to have an average compressive strength of 30 MPa. The Indonesian

Standard SK-SNI.T 15-1990-03 [11] is referred to in order to design the mix proportions of the concrete. To obtain a reliable mix design of concrete, the physical properties of aggregate of sand and crushed stone are found. Table 2 shows the mix proportions of the concrete in full. The amount of calcium stearate can also be viewed in the table. There are four mix proportions of concrete, namely the mix with the addition of calcium stearate contents of 0, 0.2, 0.9, and 1.9% of the weight of cement.

Table 2 Mix proportions of 30 MPa concrete

No.	Material	Unit weight (kg/m³)
1	G_{max} (mm)	20
2	W/C (%)	44
3	s/a (%)	30
4	Cement	530
5	Sand	495
6	Crushed stone	1085
7	Water	235
8	Calcium stearate	0, 0.2, 0.9, 1.9%

In the table, G_{max} is the maximum size of aggregate, W/C is the ratio of water to cement by weight, s/a is the ratio of sand to total aggregate by volume, and the cement used is ordinary Portland cement type I.

2.3 Specimens

Table 3 shows the total number of specimens. In the table, N is the concrete without calcium stearate, and Cal is the concrete with calcium stearate. The number following "Cal" is the dosage of calcium stearate, which is the ratio of calcium stearate to cement by weight. CS is the compressive strength, WA is water absorption, AC is accelerated corrosion, and FC is filtration of chloride ions. Cyl-1 is a cylinder 100 mm in diameter and 200 mm in height. Cyl-2 is a cylinder 75 mm in diameter and 150 mm in height. Cube is a cube with sides of 150 mm. Beam is a beam 100 mm in width, 100 mm in height, and 200 mm in length.

Four types of tests were conducted in the laboratory, namely compressive strength, water absorption, accelerated corrosion, and chloride ion infiltration tests. In the compressive strength test, the test specimen is a cylinder with a diameter of 100 mm and a height of 200 mm (Cyl-1), whereas in the water absorption test, the test specimen is a cylinder with a diameter of 75 mm and a height of 150 mm (Cyl-2). Furthermore, a beam whose dimensions are a width of 100 mm, a height of 100 mm, and a length of 200 mm is utilized for the accelerated corrosion test. A plain steel bar 12 mm in diameter and 150 mm in length is embedded inside the concrete beam. Finally, the test specimen used for the chloride ion infiltration test is a cube with sides of 150 mm.

Table 3 Number of specimens

No.	Code	Type of test	Number of specimens	Type of specimen
1	N	CS	3	Cyl-1
2	Cal-0.2	CS	3	Cyl-1
3	Cal-0.9	CS	3	Cyl-1
4	Cal-1.9	CS	3	Cyl-1
5	N	WA	3	Cyl-2
6	Cal-0.2	WA	3	Cyl-2
7	Cal-0.9	WA	3	Cyl-2
8	Cal-1.9	WA	3	Cyl-2
9	N	AC	3	Cube
10	Cal-0.2	AC	3	Cube
11	Cal-0.9	AC	3	Cube
12	Cal-1.9	AC	3	Cube
13	N	FC	3	Beam
14	Cal-0.2	FC	3	Beam
15	Cal-0.9	FC	3	Beam
16	Cal-1.9	FC	3	Beam

2.4 Procedures for Compressive Strength Test

When the specimens for the compressive strength test are one day old, they are removed from the mould. The next step is to cure the specimens by soaking them in fresh water at a temperature of around 25 °C. On the 27th day, the specimens are removed from the water and put in ambient air for one day. Finally, the compressive strength test is performed after the surface of the specimen has been capped by using sulphur to flatten it. A Universal Testing Machine (UTM) is utilized to find the compression load. A curve showing the relation between extension and compression load can be observed on the computer connected to the UTM. The compressive strength is calculated by using Equation 1. Figure 1 shows those processes. Figure 1a shows the casting of the concrete in the mould. Figure 1b describes the compressive strength test. (The curing process was carried out by submersion.)

$$\sigma = \frac{P}{A} MPa \tag{1}$$

where σ is the compressive strength (MPa), P is the maximum load (N), and A is the surface area of the cylinder (mm²).

a. Casting of concrete b. Compression test

Figure 1 Steps of compressive strength test

2.5 Procedures for Water Absorption Test

Indonesia Standard SKSNI S-36-1990-03 [12] is referred to in order to analyse the water absorption of the concrete specimen. According to the Indonesian code, the water absorption test is conducted when the specimens are 28 days old, that is, after they have been cured in fresh water for 28 days. Then, they are dried for three days in an oven at a temperature of 100 ± 5 °C. They are then placed in the ambient air for one day. Next, the specimens are weighed in the dry condition (a). After that, they are soaked in fresh water for 30 minutes and then removed from the water and cleaned to remove the remaining fresh water from their surfaces. The last step is the weighting of the specimen in the saturated surface dry (SSD) condition (b). The level of water absorption is computed by using the equation below. Figure 2 illustrates the procedures followed in the water absorption test.

$$Water\ absorption\,(\%) = \frac{b-a}{a} \times 100\% \qquad (2)$$

a. Drying in the oven

b. Weighing dry specimen

c. Soaking in fresh water

d. Cleaning the specimen until the SSD condition is obtained

Figure 2 Sequence of water absorption test

2.6 Procedures for Accelerated Corrosion Test

The corrosion test in the study adopts electrolytic corrosion to accelerate the process of corrosion. The specimens used for the accelerated corrosion test are prepared as follows:

a. A plain steel bar, 12 mm in diameter, is connected with a cable by using a weld connection (Figure 3a)
b. The connection is protected using duct tape to avoid direct contact with the concrete (Figure 3b)
c. A plain steel bar, 12 mm in diameter, is installed in the beam mould. To locate the steel bar in the centre of the beam, the steel bar is hung up using a yarn (Figure 3c).
d. The fresh concrete is placed in the beam moulding (Figure 3d).
e. After one day, the beam specimen is released from the mould. Then the specimen is cured by covering it with a wet mattress for 28 days (Figure 3e).

a. Connection between the steel bar and cable

b. Protection of the connection

c. Installation of the steel bar

d. Placement of the concrete

e. Curing of the corrosion specimen

f. Connecting the cable and power supply

Figure 3 Sequence of electrolytic corrosion test

f. The cable in the specimen is connected to the direct current power supply (Figure 3f)
g. The cable on the copper plate is connected to the direct current power supply. The specimen is put in the bucket and the copper plate is placed under the specimen. The scheme of the accelerated corrosion test is shown in Figure 4 [10]. One direct current power supply generates accelerated corrosion for four specimens. The voltage is set at around 12 V. The process of accelerated corrosion is operated for 14 days.
h. At the end of the period of electrolytic corrosion, the steel bar is taken out by breaking the specimen.
i. The rust on the steel bar is cleaned using a wire bar and then the specimen is soaked in a solution of 10% ammonium citrate to remove all of the rust.
j. Finally, the percentage corrosion can be calculated and analysed.

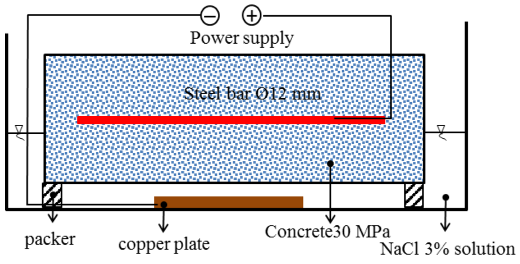


Figure 4 Scheme of accelerated corrosion [10]

The percentage corrosion is calculated according to Equation (3) below.

$$Corrosion\,(\%) = \frac{w_0 - w_1}{w_0} \times 100\% \qquad (3)$$

where w_0 is the weight of the original steel bar before the corrosion and w_1 is the weight of the steel bar after corrosion.

2.7 Procedures for the Chloride Ion Infiltration Test

The test specimens, which have dimensions of about 150 mm × 150 mm × 150 mm, are removed from the curing pond after a period of 28 days. The test specimens are placed in the open air and aerated for one day in order to dry them. All five sides of the test specimen surface are protected by coating it with a coating material such as Aqua Proof. This treatment is conducted in order to make five surfaces of the specimen impermeable to water in the short term, as shown in Figure 5.

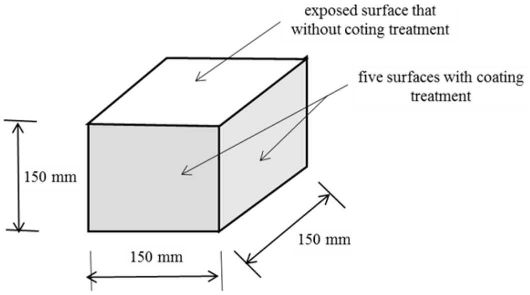


Figure 5 Coating treatment on the specimen surface

The specimen is soaked in water containing 3% NaCl for 90 days with the unprotected surface in the upper position. Then the test specimen is lifted up and its protected coating is drilled at distances of 1, 2, 4, 6, and 8 cm from the exposed side to 3% NaCl, as shown in Figure 6.

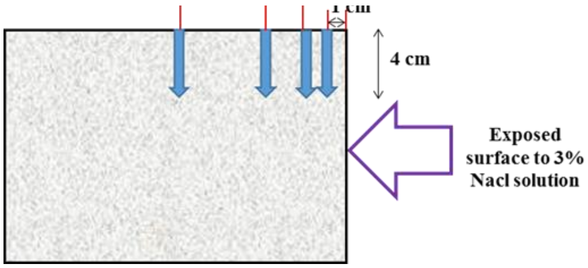


Figure 6 Drilling positions on the surface of specimen

Analysis of the concrete powder produced by drilling is done by observing the chloride content, which can be determined by conducting titration analysis of the powder. The complete process of titration testing is shown in Figure 7 [13].

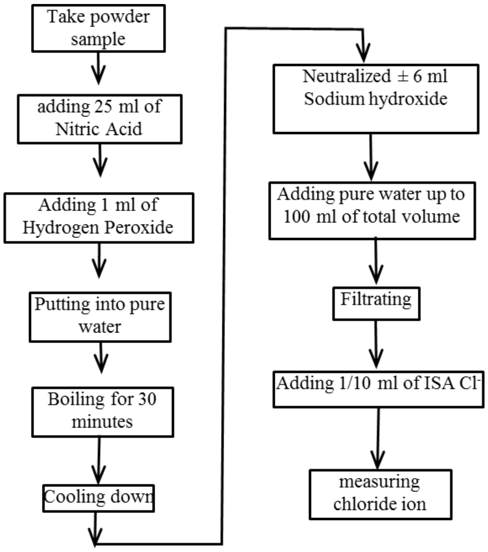


Figure 7 Procedure for filtration of chloride ions

The process of drilling the specimens can be illustrated in Figure 8a and the powder is shown in Figure 8b. The powders are packed in plastic bags and are subsequently analysed using the chloride ion infiltration procedure, as shown in Figure 7.

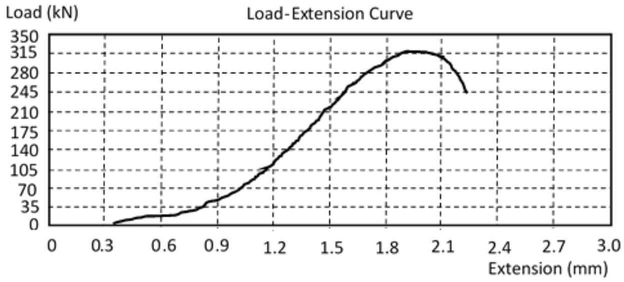

a. Drilling of specimen b. Concrete powder

Figure 8 Processes of filtration test

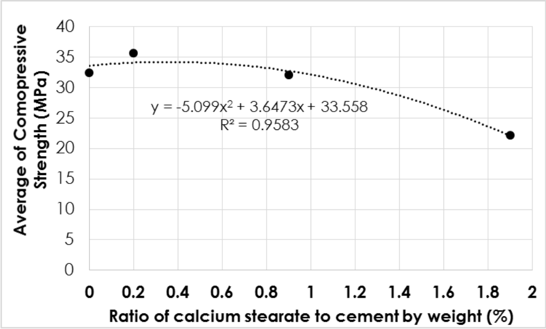
3.0 RESULTS AND DISCUSSION

3.1 Results of Compressive Strength Test

An output example of the compressive strength test performed using the UTM is presented in Figure 9a. The computer automatically records this output when the UTM is running for the compression test. The graph shows the relationship between extension and load. These data are the result of the compressive strength of the specimen with 0.2% calcium stearate. According to Figure 9b, it can be confirmed that the compressive strength is 34 MPa.



a. Relationship between extension and load



b. Relationship between calcium stearate content and compressive strength

Figure 9 Average result of compressive strength

Figure 9b expresses the compressive strength as a function of calcium stearate content. Based on the graph, it can be observed that the compressive strength of concrete without calcium stearate is a little lower than the compressive strength of concrete with 0.2% calcium stearate. The enhancement of the compressive strength may occur because the water–cementitious ratio is a little higher. Another reason is that calcium stearate reacts completely with cement to form a wax-like constituent, which coats the inner surface of the capillary during the hardening process of the concrete. It can also be seen that the addition of calcium stearate increases the compressive strength. According to the trend line in Figure 8b, when the content of calcium stearate is higher than 0.4%, the compressive strength tends to decrease, falling even lower than the compressive strength of concrete without calcium stearate. When a higher content of calcium stearate is used in concrete, a greater amount of wax-like constituent is produced. This material coats the inner capillary pores with a greater thickness. As a result, the compressive strength of the concrete declines. This can be explained by the fact that the physical properties of wax are softer than calcium silicate hydrate (tobermorite). Calcium silicate hydrate is produced when cement reacts with water. It is matched to Cellat et al. study [15]. This work investigated addition of butyl stearate in concrete. The dosages of butyl stearate used were 0%, 1%, 2%, 3%, 5% and 10%. The result show that the compressive strengths of concrete decreased due to increasing of butyl stearate dosages.

Further discussion of water absorption, accelerated corrosion, and infiltration of chloride ions will focus on the influence of 0.4% calcium stearate content in the concrete.

3.2 Results of Water Absorption Test

Based on a previous investigation [10], Maryoto stated that the values of absorption of concrete without and with 0.2% calcium stearate are 2.00% and 0.94%, respectively. The percent reduction of water absorption was decreased about 100%. This result agrees with a previous study [14]. The author stated clearly that the addition of calcium stearate in the concrete led to a slight decrease in the water–cementitious ratio of concrete. During the hardening of concrete, water evaporates and the capillaries formed have a smaller diameter due to the reaction of calcium stearate and cement. The product is a hydrophobic material that has the physical property of being wax-like. Because of this property, the concrete has a higher contact angle with water. More energy is required to force the water to penetrate the concrete. A smaller diameter of the capillaries means that the concrete is more solid. Another effect of the reduction in the diameter of the capillaries in concrete is an increase in the compressive strength. The tendency is valid when the maximum content of calcium stearate in the concrete is around 0.4%.

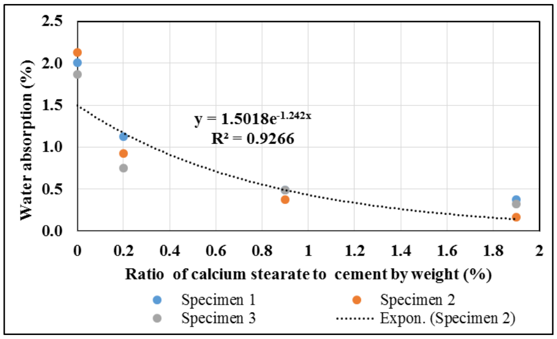


Figure 10 Water absorption of concrete

Figure 10 presents the water absorption of concrete without and with calcium stearate in various contents. It indicates that the addition of calcium stearate to the concrete significantly reduces the water absorption. The greater the addition of calcium stearate, the lower the water absorption. According to the trend line of specimen 2 on the graph, when the content of calcium stearate is around 0.4%, the water absorption is close to 0.8%. The water absorption decreases by 53% compared to concrete without calcium stearate.

3.3 Results of Accelerated Corrosion Test

The water absorption of concrete discussed in the previous section can be used to detect how much water is absorbed by the concrete. Even though the corrosive ions infiltrate the concrete together with water, the real corrosion due to the chloride ions cannot be analysed by using a water absorption test.

Nature, the process of corrosion by aggressive ions takes place over a long time. To cut the time taken for the process of corrosion, accelerated corrosion is carried out by the electrolytic corrosion test. Figure 11 demonstrates the effect of calcium stearate usage in concrete. The graph shows the correlation between calcium stearate content and percentage corrosion. The percentage of corrosion decreases significantly due to the increasing of calcium stearate content. This result has a similar tendency to the water absorption. Based on the trend line in Figure 10, when the content of calcium stearate is 0.4%, the percentage corrosion is around 5%. The corrosion is reduced by 28.5% compared to the corrosion of steel bars in concrete without calcium stearate.

Another evidence that calcium stearate in concrete reduces the level of corrosion is the appearance of crack propagation on the surface of the specimen. It is well known that the volume of corrosion product is higher than the volume of the original steel. The coefficient of the corrosion product is around 2.5. This physical property of corrosion product expansion means that more space is needed to hold its volume. As a result, the cover of the reinforced concrete is pressed out.

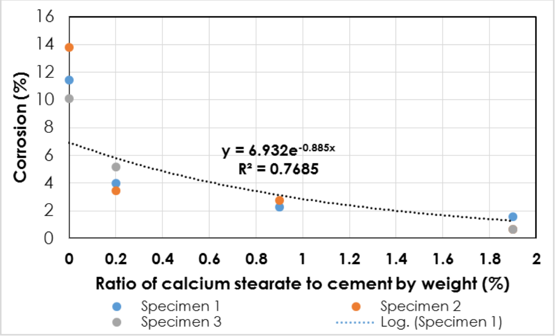


Figure 11 Accelerated corrosion of steel bar in concrete

Finally, cracks occur in the surface of the concrete. More corrosion product pushes out the cover, and the cracks generated become wider. Figure 12 shows the cracking of the concrete specimens with 0, 0.2, 0.9, and 1.9% calcium stearate. The widest crack can be observed in the specimen without calcium stearate. As the calcium stearate content increases, the cracking declines noticeably. This result agree with Geetha and Perumal investigation [16]. The study tried stearate based admixture in concrete with the dose 0, 0.18, 0.20 and 0.22 litre per 50 kg of cement. By the addition of stearate based admixture, corrosion attack decreased 16.06%, 22.12% and 26.97% consecutively.

a. Specimen "N" b. Specimen "Cal-0.2"

c. Specimen "Cal-0.9" d. Specimen "Cal-1.9"

Figure 12 Corrosion cracks on the surface of concrete

3.4 Results of Infiltration of Chloride Ions

The process of corrosion takes place when aggressive ions such as chloride ions reach the surface of the steel bar in concrete. The presence of chloride ions in concrete can indicate the service life of the reinforced concrete. The percentage of chloride ions in concrete can be confirmed by using Figure 13.

The horizontal axes present the depth of penetration of chloride ions and the vertical axes represent the content of chloride ions as a percentage. The figure shows that the percentage of chloride ions in the concrete decreases when the location of drilling is at a greater depth from the exposed surface.

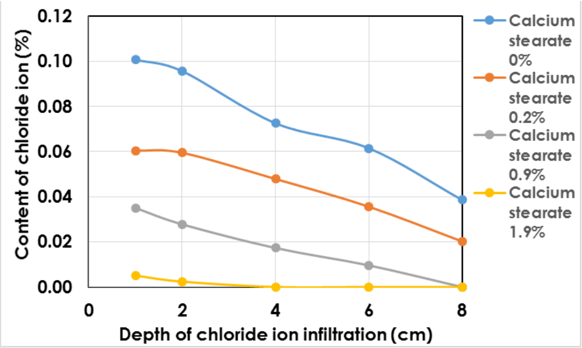


Figure 13 Infiltration of chloride ions to various depths

A similar tendency appears in concrete with a higher content of calcium stearate, where the infiltration of chloride ions is decreased. This is because the reaction between calcium stearate and cement coats the inner surface of the capillary and the constituent has a hydrophobic property.

4.0 CONCLUSION

According to the discussion in the previous sections, some conclusions can be drawn as follows:

- The research showed that the addition of calcium stearate reduced the absorption rate significantly. With a calcium stearate content of 0.4%, the water absorption of concrete decreases by approximately 53%.
- The optimum calcium stearate content used in the concrete as an additive is 0.4%. According to the trend line, the content of 0.4% in concrete produces a compressive strength as high as that of concrete without calcium stearate. Thus the calcium stearate does not affect the load-carrying capacity.
- The addition of 0.4% calcium stearate in concrete reduces corrosion attack due to chloride ions, which is reduced by 28.5%.
- The infiltration of chloride ions is also diminished by around 15% due to the addition of 0.4% calcium stearate in concrete.

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