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The effect of wetting-drying cycles on the properties of organic corrosion inhibitor concrete

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Abstract

This paper presents the results of an experimental investigation into the effects of exposure to wet and dry cycles on compressive strength and water permeability resistance in organic corrosion inhibitor concrete. Two types of organic corrosion inhibitors were investigated. One is added to the concrete mix (SKG.1), and the other is applied to the concrete surface (SKG.3). Wet-dry cycles were used, with two days wet and five days dry. Under the effect of three chloride concentrations at 3.5, 5, and 7% of NaCl solution, after 7, 28, 84, and 200 days of exposure, compressive strength and water permeability were determined. The results indicated that the concentration of NaCl solution had affected the compressive strength of concrete (C) under exposure to wet-dry cycles. SKG.1 has reduced the effect of chloride solution on both the compressive strength and water permeability of concrete exposed to wet and dry conditions. The influence of the NaCl solution on compressive strength appear to be minimal after exposure to wet and dry cycles in the sample (OS) where SKG.3 was applied on the concrete surface.

Keywords: Organic corrosion inhibitors, concrete, wet-dry cycles, compressive strength permeability

1. Introduction

Reinforcement corrosion due to chloride attack on structural member increase the cost of construction and later maintenance and eventually the less of the service life of the structure. With the technological development of additive material make it possible to reduce or eliminate the effect of such attack. The most important materials used to protect from chloride attack are Corrosion Inhibitor Materials (CIMs). These are effective and low cost materials. These materials should be examined under ocean and sea concentrations with different rates of exposure. Using sub-arrangements to transform wave energy into electrical energy, aspirations for creating more sustainable electric energy with less environmental impact, such as wave motion, can be realized. These ambitions, present new challenges in terms of protecting structures from aggressive environments. Climate change will add challenges for engineers to protect transport road and building exposed to the marine environment, to reduce and limit the development of chloride attacks effect. It is the responsibility of engineers to ensure environmentally friendly products like CIMs are offered, and this is where the possibilities and challenges are for today's marine civil engineering community. Therefore, increasing the resistance of installations to aggressive marine conditions while minimizing their environmental impact is critical.

Degradation of buildings exposed to seawater due to poor design, poor maintenance, corrosion, breakage, freezing, thawing, thermal damage, insufficient communication between contractor and designer, unsatisfactory construction work under supervision. A better understanding of the possible causes of failure can help designers and owners make appropriate decisions during design. It is necessary to study the behavior of concrete and reinforced concrete under different conditions of exposure to seawater and its effect on marine structures ^[1]. The increase of offshore structures, including civil and military terminals, offshore airports, offshore wind power stations, radar stations, island reefs, and fortifications with different natures of exposure, makes the protection of reinforcement steel from corrosion more difficult and dispersed for researchers. Concrete exposed to the marine environment is vulnerable to a variety of physical and chemical degradation procedures. The average content of dissolved salt in seawater is about 35 g/l ^[2]. The concentration of the salt changes with the geographical location.

Chloride ions present in seawater attack the structural elements with a so-called chemical attack on all exposed concrete.

The increase in the population with the increase in dependence on the ports makes the need for more durable and flexible infrastructure, and increasing demand for land space. Rising temperatures increase the chloride ion concentration, which affects coastal infrastructure. The presence of buildings near, or under a source of chloride put concrete under aggressive conditions in terms of chloride attack, especially for bridges and marine platforms.

Buildings are exposed to environmental factors with a different concentration of chloride from 3.5 to 7% of solution. Previous studies allowed a concentration of 0.2% by weight of cement, and stated that 0.6% by weight of cement is a critical concentration. It also stated that 1% by the weight of cement, the structure is highly exposed to the chloride environment. Table 1 shows chloride concentrations and their effects on the structure. In the marine environment, the tidal zone is the most affected area by chloride. The area submerged in seawater is affected, but not as much as the tidal zone due to the presence of sufficient oxygen in the water. That areas prone to groundwater such as tunnels and foundations exposed to wetting, and drying cycle [3].

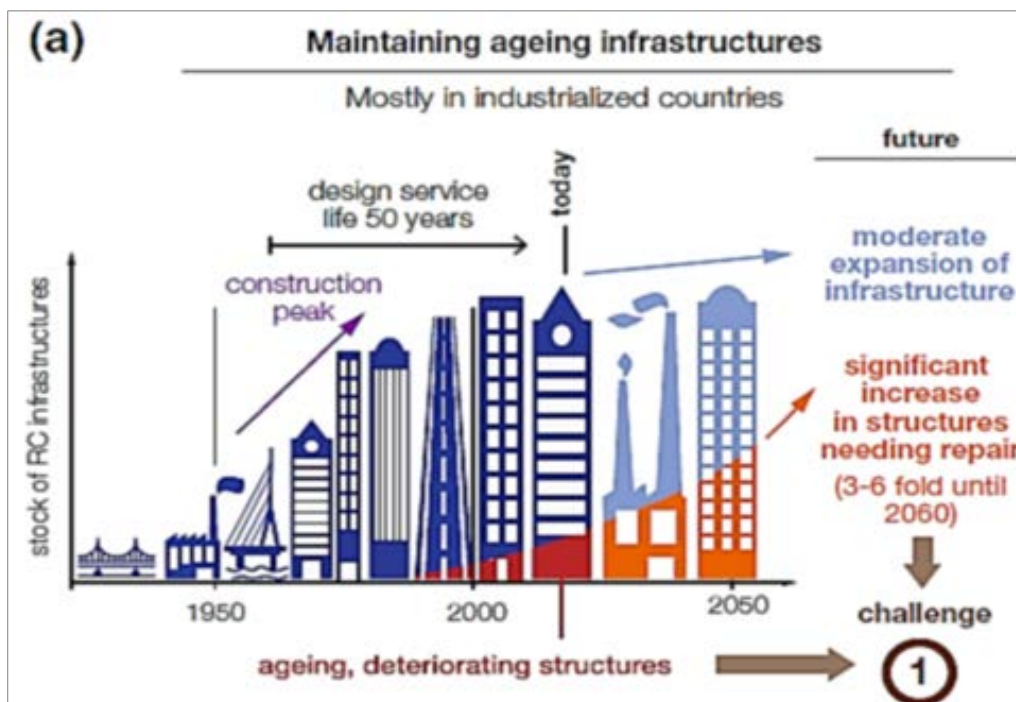
Table 1: Effect of chloride content on concrete structure

Chloride content	Everett and Treadaway (1980)	Browne (1980)
<0.4	Low risk	Negligible risk
0.4 - 1	Medium risk	Possible risk
>1	High risk	-----
1 - 2	-----	Probable risk
>2	-----	Certain risk

Many techniques have failed during experimental tests,

notwithstanding the researchers' best efforts to comprehend steel corrosion in concrete. By analyzing the most recent discoveries, it may be inferred that there are some gaps in understanding of the corrosion initiation and propagation mechanisms since many issues remain unanswered. Poor durability of the reinforcement concrete is the most prevalent cause of degradation in RC models. This was obvious in several investigations of actual situations of early RC structure deterioration, with the conclusion that corrosion was the main degradation process in 70 to 90 percent of the evaluated cases [4]. Forecasting the long-term durability performance of RC structures in their real surroundings is critical for both maintenance planning of existing structures and developing durable and sustainable new structures as explained in Figure 1. Although these two difficulties exist in all countries, however, it was not sufficiently cared for. Industrialized countries often have the difficulty of dealing with existing, aging infrastructures that were predominantly created in the second part of the past century, while expanding existing infrastructure accounts for a modest portion of these countries' building activity. Given the predicted growth in essential repair work throughout the next few decades, there is unquestionably an urgent need to abandon the existing rather conservative and consequently costly approach. This can only be accomplished by developing new and cost-effective technologies, as well as gaining a thorough understanding of corrosion degrading mechanisms [5].

Ammonia is a colourless gas with a distinct characteristic of a pungent smell. A compound of both nitrogen and hydrogen so that it formulates NH₃. The chemical structure of Amines consists of a basic nitrogen atom with an unshared electron pair (:N). Amines and Alkanolamines were described as (organic basic nitrogen compounds). It consists of salts with organic or inorganic acids to be used as a corrosion inhibitor. In the case of the Amine and Alkanolamines synthesis, one or more atom of the element hydrogen is replaced [6].



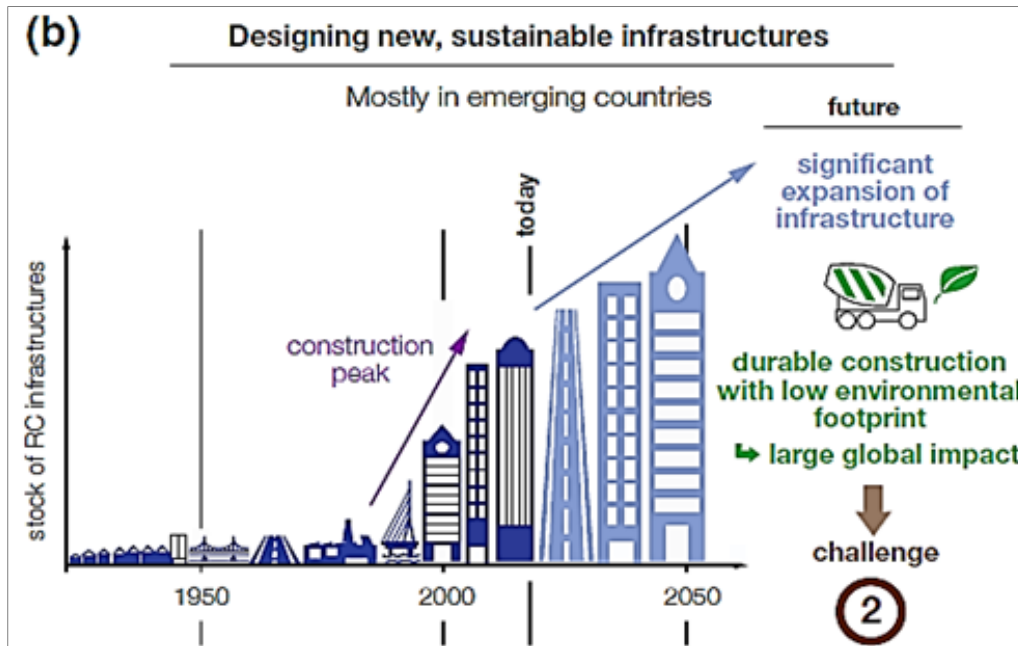


Fig 1: The technological challenges associated with the corrosion of steel in concrete in the world [5]

Mixed inhibitors consist of organic compounds as Amines and alkenolamines that are adsorbed on the surface of the metal by forming a film and preventing the anodic and cathodic reactions of iron and oxygen. As shown in Figure

2. the mechanism of organic corrosion inhibitors is through adsorption (physisorption and/or chemisorption) on the metal surface [7].

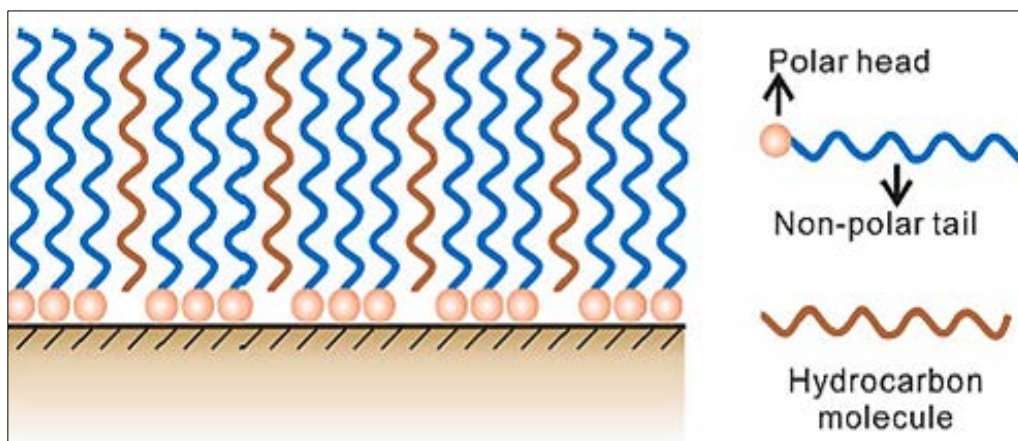


Fig 2: Schematic mechanism of mixed inhibitors [7]

Determining the efficiency of inhibitors type Amines and Alkanolamines (organic basic nitrogen compounds) is difficult. Most commercial Inhibitors are made from a mixture of chemicals whose composition is unknown to the researcher and is not disclosed by the manufacturer. Therefore, commercial inhibitors are called (Amino-Alcohol Based Mixed Corrosion Inhibitor) [8, 9].

Studies have demonstrated the efficacy of Amino Alcohols (AMAs) from the use of S KG.3 and their interaction with iron. Penetration depends on porosity of concrete and moisture, and does not affected by water through sprinkling and puddles on the concrete structure [10]. The effectiveness of AMAs was verified in terms of their interaction with rebar and formation of a layer on the iron surface so that a simulation of concrete conditions was used. The mechanism

of action of SKG.1 is primarily anodic protection. It must be added in the appropriate amount so that corrosion acceleration does not occur [11]. The effectiveness of the inhibitors was verified during and after exposure, and the study proved that the efficacy of the inhibitors is good after exposure to the chloride environment. This enables us to use Migrating Corrosion Inhibitors (MCIs) for the purpose of treating concrete exposed to the harsh marine environment [12].

The components are rarely identified from the factory, Material Safety Data Sheet (MSDS), but some of them were examined due to the safety and security requirements of some countries, where the great difference in the composition of the same product for different factories is shown in Table 2 [13].

Table 2: Composition of the same product for different factories material safety data sheet

Country	Product	Method of use	Type of ingredient	Chemical ingredients
				MSDS
Norway	SKG.1	Mixed into concrete	2-dimethylaminoethanol (DMEA)	5 - 10
			Organic/ inorganic nitrogen compounds	Not given
Canadian			Salt of alkanolamine	15 - 40
Norway	SKG.3	Applied on concrete	2-aminoethanol	5 - 10
			Alkanolamine	10 - 30
Canadian			Ethyl alcohol	1 - A 5

The effects of incorporation of organic corrosion inhibitors on the properties of concrete are incompletely understood where the straightforward and clear explanation of this effect still needed.

The main objective of this study is to provide more insight about the effect of organic corrosion inhibitor on the compressive strength and permeability of concrete containing different concentrations of chloride solution exposed to wetting-drying cycles.

2. Experimental methodology and design

2.1 Materials

The materials used in this study were organic corrosion inhibitors (SKG.1, SKG.3), Ordinary Portland Cement (OPC), local Sand, and coarse aggregate. The raw materials for the experimental program have been provided and processed to conform to ASTM standards. Certain measures were taken, such as washing both local sand and coarse aggregate to meet the standard requirement. One organic corrosion inhibitor (OCI) is added to concrete and another one is applied to the surface to reduce the chloride effect. The use of organic inhibitors (amines) is environmentally friendly and non-toxic. However, the percentage recommended by the manufacturer was used.

2.1.1 Cement

Commercially available ordinary Portland cement (OPC)

which CEM I 42.5 N according to European BSEN 197-1^[14] was used. Table 3 show the cement chemical compositions tested by X-ray fluorescence analysis.

Table 3: Cement chemical compositions tested by X-ray fluorescence analysis

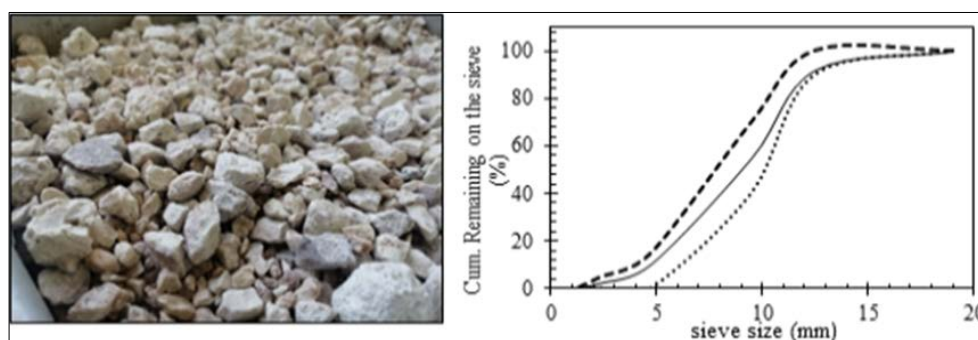
Chemical composition%	CEMENT (OPC)
Silicon dioxide (SiO ₂)	20.90
Aluminum oxide (Al ₂ O ₃)	6.06
Iron oxide (Fe ₂ O ₃)	3.68
Calcium oxide (CaO)	64.56
Sodium oxide (Na ₂ O)	0.24
Potassium oxide (K ₂ O)	0.98
Chloride (Cl)	0.007
Sulfur trioxide (SO ₃)	2.53

2.1.2 Coarse aggregates (CA)

Coarse aggregates conforming to American standers ASTM C33^[15] was used. The maximum size of coarse aggregates was 12.5 mm. Particle size distribution and physical properties of the coarse aggregates is shown in Figure 3 and Table 4 respectively.

2.1.3 Fine aggregates (FA).

Local sand with a specific gravity of 2.56 is used. The sand was oven-dried at 100°C and cooled for at least 24 hours before use. The physical properties of sand are given in Table 5.

**Fig 3:** Sieve analysis of the coarse aggregates**Table 4:** Properties of coarse aggregates

Properties of coarse aggregates	Test result	ASTM stander	
		Number	Range
Aggregate impact value.	18.66%	C33	<30%
Abrasion resistance of aggregate in Los Angeles machine.	28.53%	C131	<40%
Percentage absorption for aggregate.	2.5%	C127	-
Specific weight of coarse aggregate	2.60	C33	(2.5-2.75)
Moisture in the coarse aggregate	0.24%	-	-

Table 5: Properties of fine aggregate

Properties of fine aggregates	Test result	ASTM stander	
		Number	Range
Percentage absorption for fine aggregates	2.28%	C128	<2%
Specific weight of fine aggregates	2.56%	C33	(2.5-2.75)
Moisture in the fine aggregates	0.31%	C70	-

2.1.4 Corrosion inhibiting concreting mixture (SKG.1)

It is a liquid concrete mixture based on Sika ferrogard® technology for use in reinforced concrete and mortars. It acts as an inhibitor to protect steel reinforcement from corrosion. Designed for reinforced concrete especially the building exposed to chloride environment to provide protection against corrosion caused by chloride. It is often used in structures that have a long service life, such as concrete roads, bridges, tunnels and, retaining walls. Mechanism of SKG.1 mixes both anodic and cathodic reactions of the electrochemical corrosion process to reduce reinforcement corrosion. The product forms a film on the steel surface which delays the damage of corrosion and also reduces the degree of corrosion. SKG.1 was mixed with water. The quantity of SKG.1 in the mix design was taken into consideration when determining the quantity of water for a specific W/C ratio. Table 6 show the chemical composition of SKG.1 according to the manufactory description.

Table 6: chemical composition of SKG.1

Chemical name	Concentration (% w/w)
2-dimethylaminoethanol	10%
2,2'-(methylimino) diethanol	10%

2.1.5 Corrosion inhibiting concrete surface applied (SKG.3)

SKG.3 is a surface coating that works by impregnating through hard concrete with a liquid corrosion inhibitor. It is designed to first penetrate the surface and then diffuse

through concrete in the form of vapour or liquid to steel reinforcement bars embedded in the concrete, SKG.3 forms a protective film on the steel surface which delays the corrosion process caused by the presence of chlorides as well as the carbonation of concrete. SKG.3 consists of a mix of amino alcohols and organic and inorganic inhibitors that protect both anodic and cathodic parts of the corrosion cell. It increases the service life of buildings by delaying the onset of corrosion and reducing the overall corrosion activity. SKG.3 protects steel reinforcement by forming a protective layer on the surface of steel reinforced. Table 7 shows the chemical composition of SKG.3 according to the manufactory description.

Table 7: chemical composition of SKG.3

Chemical name	Concentration (% w/w)
2-aminoethanol	20%
Tributyl phosphate	1%

2.1.6 Mix design

The mixed proportions of concrete are summarized in Table 8 The amount of Ordinary Portland Cement type I (OPC) of 360 kg/m³ was used in all mixes. All concrete mixtures had a water-to-cement ratio of 0.6 and an estimated slump of fresh concrete between 150 and 200 mm. Control specimen (C) with no admixture or surface coating. SKG.1 specimen (OA) and SKG.3 specimen (OS) were used as corrosion inhibitors. SKG.1 was added to the concrete at a ratio of 12.5 kg / m³. SKG.3 was used as an application on the surface at a rate of 0.4 kg / m².

Table 8: Mix proportion of concrete mixes

MIX	Binder	% per cement weight					SKG.1	Slump
		Cement	SKG.3 (kg/m ²)	CA	FA	water	Kg/m ³	cm
C	OPC	360	0	989	679.3	216	-	14
O.A	OPC+SKG.1	360	0	971	667.4	216	12.5	23.5
O.S	OPC+SKG.3	360	0.4	989	679.3	216	-	14.2

2.3 Test methods

2.3.1 Compressive strength of concrete under wet-dry cycle

The mixing process followed the procedure described in ASTM C192/C192M^[16] using the pan mixer (model UTC-0750-T). The concrete specimens were cast using cubes (100 X100 X 100 mm) from steel molds in accordance with ASTM C31/C31M^[17]. The moulds were covered with a non-absorptive, non-reactive sheet of tough, durable, impervious plastic for 24 hours. Demoulding take place after that and thereafter placed in a curing tank for up to 28 days. After curing, the samples are subjected to a wet and dry cycle in basins with a concentration of 3.5, 5, and 7% sodium chloride solution. So one cycle is two days wet and left to dry for five days. Specimens were tested for compressive strength at the ages of 7, 28, 84, and 200 days

following the listed procedures of the test method BS EN 12390-3^[18]. An average of three samples were taken for each test. The OS concrete specimen was coated with the SKG.3 materials several times and left for an hour before painting each coat. Then it was left for a day before being exposed to the wet and dry cycle.

2.3.2 Permeability penetration under wet-dry cycle

A water penetration depth test was performed according to BS EN 12390-8^[19], where the sample was not pre-saturated. The Concrete specimens (C, OA, and OS) were cubes (150×150×150) mm and used to obtain the water penetration depth. After curing specimens in the curing tank for about 28 days, the samples are subjected to a wet and dry cycle in basins with a concentration of 3.5, 5 and, 7% sodium chloride solution. So that one cycle is two days

wet and left to dry for five days. During testing, the water pressure of 500 ± 50 KPa was applied to the bottom face of each cubic. Water pressure was applied to the sample for 72 ± 2 hours. Samples were removed from the apparatus and, then each sample was split into two halves to measure the depth of water penetration. The result of water penetration depth was taken as the maximum depth of water penetration for the three cubic samples poured from the same batch of concrete and tested at the same time. The readings were taken after 7, 28, 84 and, 200 days.

3. Results

3.1 Effect of wet and dry cycle on compressive strength of concrete: Generally, it can be seen that the compressive strength for concrete C, OA, and OS under exposure to a

3.5% chloride ion concentration were increased as the age of exposure to the wet and dry cycle extended (Figure 4a). Specifically, the compressive strength increased rapidly during the first 28 days of experiencing a wet and dry cycle whereas increased slightly between 28 and 84 days, with OA samples outperforming both C and OS at this time. After 84 days of exposure, the compressive strength of C, OA, and OS have almost stabilised.

Figures 4b – 4c illustrates the change in pressure resistance of C, OA, and OS samples under exposure to both chloride ion concentrations of 5, and 7%. The results show that the compressive strength of C and OA samples is unaffected by the rise in chloride concentration, with the exception of OS, which showed a modest drop in compressive strength with an increase in chloride concentrations.

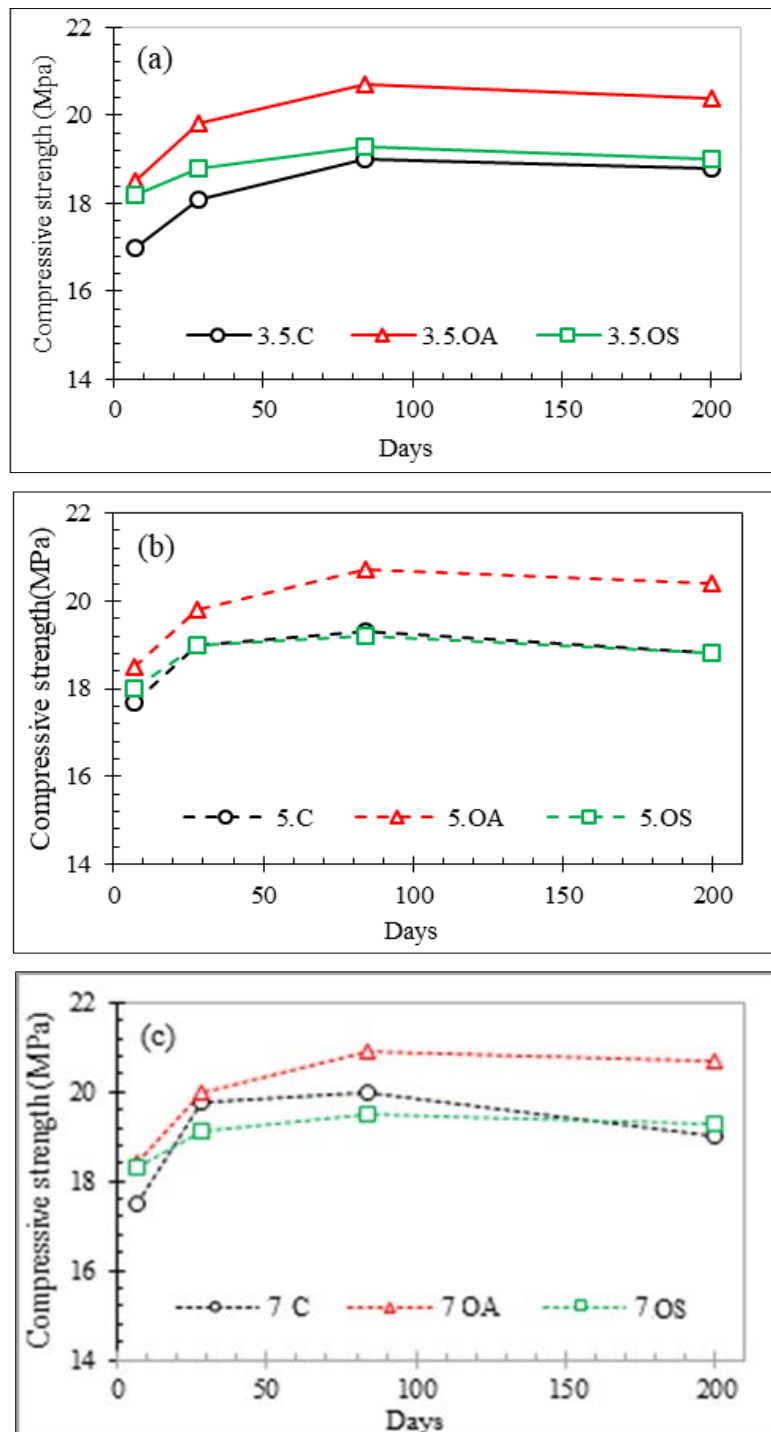


Fig 4: Influence of chloride concentration on the compressive strength of C, OA and OS samples under wetting-drying cycles

From Figure 5a -5c, it can be observed that the compressive strength increased at both the ages of 28 and 84 days of exposure to the cycles of wetness and drying, while it decreased gradually after 84 days. In addition, the increase in compressive strength of concrete (C) exposed to wet and dry cycles depends on the solution concentration. The increases in compressive strength at 28 days of exposure could be explained by the clogging of pores by salt crystals at 28 days of age, which gave additional compressive strength to the sample. As the wet and dry cycles continue, the size of the crystals shrinks and the microstructure of the sample is affected by repeated wetness and drying, causing a slight decrease in compressive strength. This result is in agreement with several previous studies [20, 21]. For the

sample (OA), the compressive strength increased slightly at the age of 28 and 84 days, in contrast, it decreased at the age of 200 days from exposure to wetness and drying. In addition, the compressive strength of (OA) was unrelated to increasing chloride solution concentration. This may be due to the existence of SKG.1 inside the pores reduced the effect of chloride solution on the microstructure. For the sample (OS), the effect by exposure to wet and dry cycles is negligible. This may be due to the existence of SKG.3 on the surface of the sample, which resisted the ingress of chloride solution into the microstructure, and thus the effect was limited. These results are consistent with the predictions of several previous studies [10].

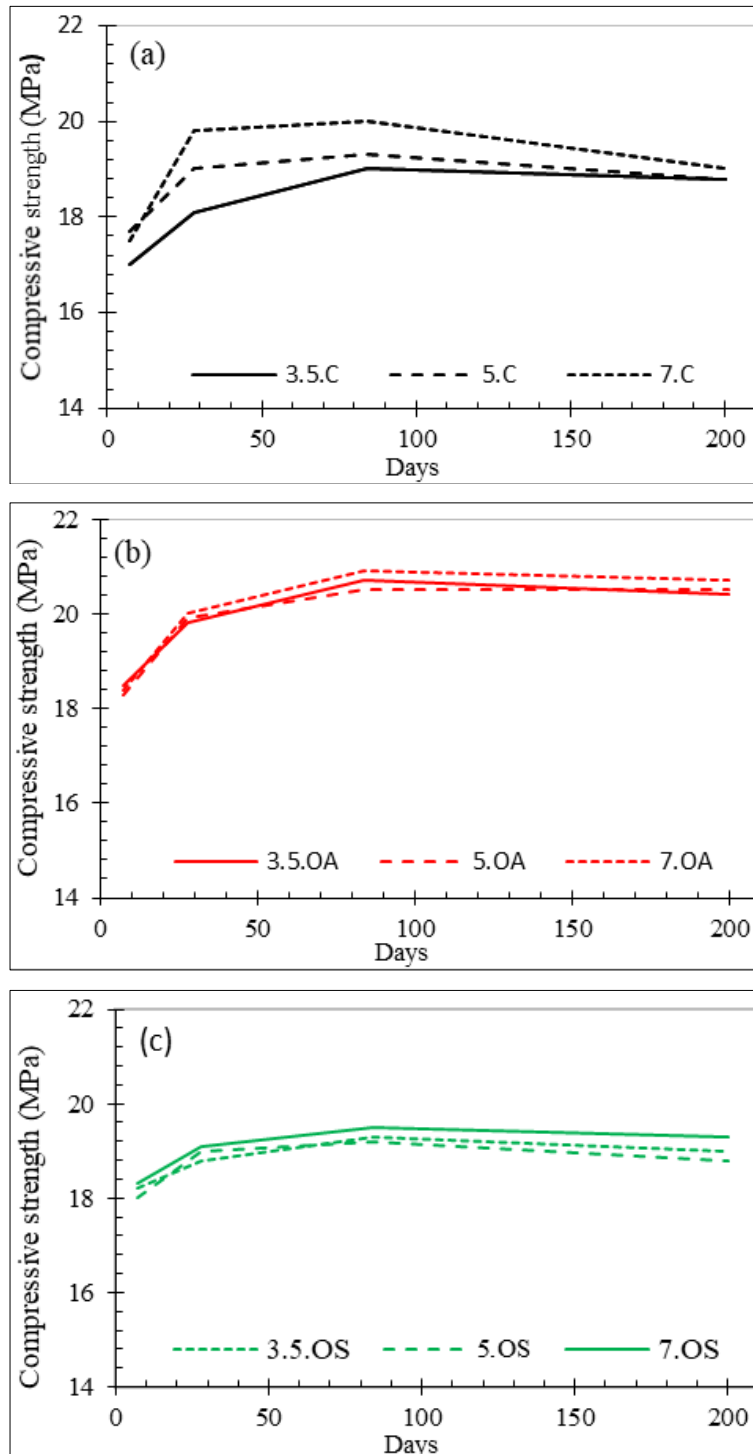


Fig 5: Influence of wetting-drying cycles on compressive strength of OCI concrete containing chloride solution

3.2 Effect of wet and dry cycle on permeability of concrete:

It can be seen that the permeability of all the C, OA, and OS concrete decreases with the increasing duration of exposure. Permeability decreased in the period between 7 and 28 days of exposure, and these decreases continued in the period after 28 days (Figure 6a – 6c).

The concentration of the solution has an effect on the increase in permeability of concrete (C) exposed to wet and dry cycles (Figure 6d). It can be explained that salt crystals are the cause of pore blockage [20, 21]. Wet and dry cycles continue, and repeated wetting and drying alter the material's microstructure.

Furthermore, the rising concentration of chloride solution had also an effect on permeability of concrete (OA) which increased with increased chloride solution (Figure 6e). As was the case in the compressive strength test, this might be due to the presence of SKG.1 within the pores, which reduced the influence of the chloride solution on the microstructure. For the concrete (OS), the results were the closest to the concrete (C) by exposure to wet and dry cycles (Figure 6f). This may be due to the existence of SKG.3 on the surface of the sample, which do not resist the water pressure from this test, and thus the effect was clear.

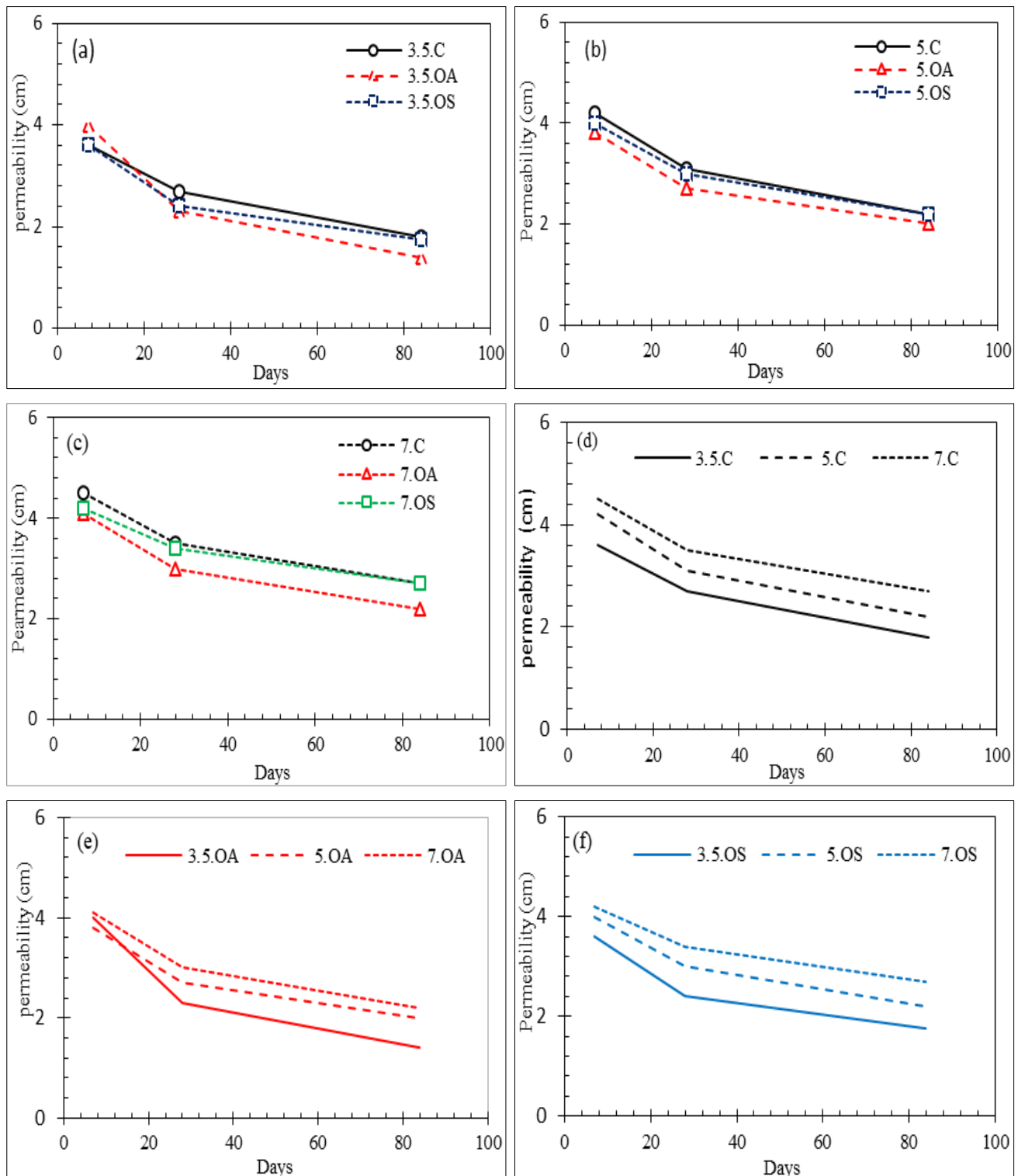


Fig 6: Influence of wetting-drying cycles on permeability of OCI concrete containing chloride solution

4. Conclusion

The growing concerns regarding the reinforcement corrosion due to chloride attack on structural member, increased the interest in the use of additives that reduce or eliminate the effect of such attack.

The effects of two types of organic corrosion inhibitors (SKG.1; SKG.3) on the mechanical and transport properties of concrete containing different concentration of chloride solution and exposed to wet – dry cycles were investigated.

The results revealed that the concentration of NaCl solution affects the compressive strength of concrete (C) under exposure to wet-dry cycles however this effect was less and negligible on concrete (OA) and concrete (OS) respectively. Additionally, it was concluded that the increase in permeability of concrete (C) and concrete (OS) exposed to wet and dry cycles are influenced by the concentration of chloride solution whereas, the permeability of concrete (OA) at early ages was decreased slightly.

5. Recommendations

Based on this research result, the following recommendations were obtained

1. The influence of temperature on inhibitor efficacy was not considered in this study. Therefore, it could be recommended to investigate the effect of temperature on inhibitor effectiveness.
2. Wetting and drying cycles were used as natural corrosion accelerators in this investigation. As a result, investigating the effect of submergence on the inhibitor's efficiency is recommend
3. Steel-reinforced corrosion was accomplished in this study when a water-to-cement ratio of 0.6 was used to accelerate steel-reinforced corrosion. As a result, it is critical to recommend that the effect of 0.4 and 0.5 water-to-cement ratios on the inhibitor's efficacy be investigated.

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