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Effect of moisture variation on oxygen consumption rate of corroding steel in chloride contaminated concrete

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ABSTRACT

This paper is based on experimental investigations carried out on the effect of varying relative humidity on oxygen consumption rate of corroding reinforcement steel in concrete under impressed potential macro-cell cathodic reaction for which the previous research data is limited. The objective of this research is to investigate the qualitative as well as the quantitative relation between the oxygen consumption rate of corroding reinforcement carbon steel in chloride contaminated concrete and ambient relative humidity under continuous as well as cyclic exposure conditions. For this purpose, corrosion cells with locally separated anode and cathode steel reinforcement bars under four different environmental conditions (namely air dry, submerged, 95% relative humidity and alternate wetting–drying) have been tested under strict laboratory control. By analyzing the results of these tests, it was possible to understand the behavior of oxygen consumption rate of corroding steel reinforcement in chloride contaminated concrete under varying moisture environmental conditions.

1. Introduction

Steel reinforcement within concrete corrodes under the effect of various aggressive agents and severe environments such as chloride, carbonation, high temperature, and acids. However, these aggressive agents and severe environments can cause corrosion only if enough amounts of oxygen and moisture are available in the vicinity of corroding reinforcement bar in concrete. Therefore, it is necessary to understand the coupled effect of oxygen and moisture on corrosion of RC structures. In this research qualitative as well as quantitative deep experimental investigations have been carried out in order to clarify the involved mechanisms of corrosion under variable oxygen consumption rate and moisture conditions for which the previous research data is limited, especially for the coupled effects of oxygen consumption rate and moisture diffusion through the concrete cover pores under impressed potential macro-cell cathodic reaction. This specific condition is very practical but has a limited research in the past. Also, a direct quantitative relation between cathodic current and oxygen consumption rate for moisture controlled corrosion reaction has not been documented in the past but such a physical phenomenon is very important to understand.

In the past, Maruya et al. [1] has worked on the numerical modeling of steel corrosion in concrete structures due to chloride ion, oxygen and water movement. Some other researchers also worked on the effect of concrete pore solution and moisture on the quality of oxide films on black steel reinforcement [2] and actors influencing chloride transport coupled with oxygen and moisture movement in concrete structures exposed to severe corrosive environments such as sea water [3]. Dangla et al. [4] explored the rebar corrosion in concrete exposed to variable humidity conditions and Conciatori et al. [5] studied chloride ion and water ingress into concrete considering for real corrosive climate. In the past, research has been conducted on the effect of Cl^-, moisture and O_2 on corrosion of steel in concrete [6], service life of corroding reinforced concrete structures and the parameters effecting it under nonlinear relationships of moisture, oxygen and chloride concentration [7,8]. Ample literature in available on the corrosion rate evolution in concrete structures exposed to different atmospheric conditions [9], general steel corrosion in concrete structures and durability assessment of reinforced concrete structures under coupled deterioration processes due to chloride attack and its dependence on varying moisture and oxygen [10–14]. However, the previous reported above did not include the vital relation between oxygen consumption rate and moisture variation under macro-cell cathodic state of reaction which needs to be further investigated. No doubt research has been carried out for macro-cell corrosion of steel in concrete [15,16]. But, no one has dealt with it with the implication narrated above which is the novelty of this paper.

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Investigations of galvanostatic transient method used to monitor the corrosion rate of steel in concrete have been conducted in the past [17], protection corrosion current distribution in reinforced concrete cathodic protection systems have been explored [18] and corrosion equilibrium potentials of membrane electrodes have been studied in the past [19]. But, this electrical circuit setup has not been investigated under impressed potential condition which is the originality of this research. Corrosion of steel in concrete is an electrochemical reaction and is one of the most intensely researched phenomena from the beginning within the study of durability of reinforced concrete [20–22]. The influence of oxygen on corrosion of steel in concrete has been explored by the previous researchers [23,24], the diffusion of dissolved oxygen through concrete is available in the literature since long ago [25], the oxygen permeability of concrete is also well known [26], but limited work has been done to quantitatively differentiate the influence of various moisture conditions under constant as well as cyclic behavior on the oxygen consumption rate of steel embedded in concrete under enforced high corrosion rates and separated anode–cathode formation which is very imported to be understood and has been carried out in this paper.

Thus, the objective of this research is experimental determination and clarification of the influence of oxygen consumption rate controlled by moisture diffusivity on the corrosion process of reinforcement in concrete structures under various defined laboratory conditions. The cathodic reaction is associated with the consumption of the electrons generated in the anodic process and formation of hydroxyl ions. In order to complete the corrosion cell, the hydroxyl ions move to the location of anode from the cathode through concrete. The speed of movement depends on the concrete resistance. It is true that once the passivity of steel bars in concrete is destroyed, it is the cathodic process, oxygen consumption rate, relative humidity and the electrical resistivity of concrete which control the rate of corrosion. The current investigation focuses on four types of environmental exposure conditions (air dry, submerged, 95% relative humidity and alternate wetting–drying) consecutively, which is also the originality and novelty of this research. The exact quantity of limiting corrosion current under water has also been a question mark which is also solved in this paper.

2. Experimentation

2.1. Materials

Ordinary Portland cement (OPC) as per JIS R5210 specifications was used throughout the experiment in this research. Natural river sand passed through JIS A1102 sieve No. 4 (4.75-mm openings), was used as fine aggregate for all concrete mixes. Its density and water absorption were 2.65 g/cm^3 and 2.21%, respectively. Crushed sandstone with a maximum size of 20 mm was used as coarse aggregate with density of 2.70 g/cm^3 and water absorption 0.59%. It was retained on the sieve No. 4 (4.75-mm-openings) and cleaned before being used. Ordinary tap water supplied at Concrete laboratory, Department of Civil Engineering, The University of Tokyo was used for all concrete mixes. Deformed round carbon steel bars 13 mm in diameter were used as reinforcing steel. The surface of steel bar was polished by sand paper No. 200. Finally, steel bar was degreased by acetone just prior to being placed in the mould.

2.2. Mix proportions

Trial concrete mixing was firstly conducted to achieve 05–08 cm of slump value and 3.5 ± 1% of air content. The concrete composition is as mentioned in Table 1.

Table 1
Concrete composition.

Sr. # (–)	*W*/*C* (%)	Binder (kg/m^3)	Water (kg/m^3)	Fine agg. (kg/m^3)	Coarse agg. (kg/m^3)	SP (kg/m^3)
1	30	555	167	654	980	6.1
2	45	371	167	756	1031	2.2

SP: super plasticizer SP 8SBS Rheobuild BASF (poly carboxylic acid ether).

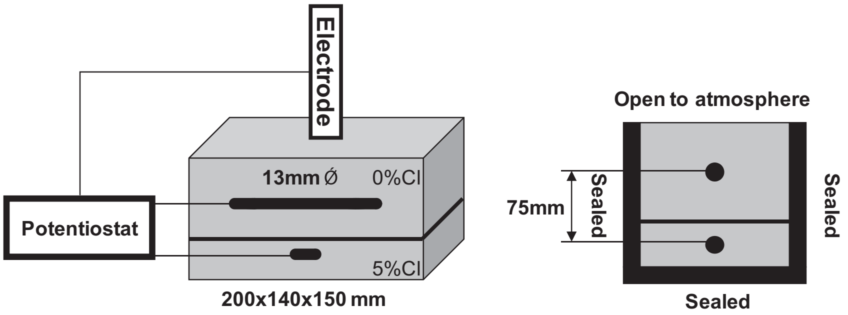
2.3. Storage conditions

Unless otherwise noted, specimens were left at the casting place to set for 24 h and then given sealed curing for 28 days at 20 °C and stored in air-conditioned large environment control room at 60% relative humidity condition.

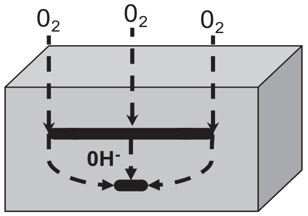
2.4. Experiment specimen

The structure of the test specimens is shown in Fig. 1. Five centimeter thick layer of concrete, to which chloride content (5% total chloride by mass of binder) has been added, was first filled into an air and watertight plastic container of size 200 mm × 140 mm × 150 mm (length × width × height). After setting of the chloride-containing concrete, a second chloride-free concrete layer was added onto the first one. Reinforcing steel bars were embedded in both concrete layers and connected via external cables, allowing measurement of the macro-cell current between the two electrodes. After concrete pouring the specimens were epoxy coated on the top face edges of plastic containers in order to avoid any possible oxygen penetration through side walls and given the prescribed environment exposure. Tests were performed with a cathode/anode steel area ratio of 400–20 cm^2 (cathode surface area = 20 times anode surface area; length of cathode steel = 200 mm, length of anode steel = 10 mm) and an axial distance of 75 mm between the steel bars to keep the nature of cathodic reaction oxygen diffusion controlled. The electrode material was mild steel deformed reinforcing round bars with a diameter of 13 mm. The concrete cover of the cathode was kept 50 mm and the concrete cover of the anode to the container bottom was 25 mm. This was done to create a distance of 75 mm between anode and cathode necessary to make the reaction oxygen diffusion controlled and not hydroxyl ion controlled. The methodology is based on the previous research [23,24] with necessary modifications as required by the research objectives of this study and to cover up the deficiencies in the previous research. For example in the previous research, relative humidity and temperature could not be maintained at the desired levels. So, in this research very accurate automatic environment control chambers were used to keep the humidity and temperature on the required constant levels all the time. This adds to the reliability and repeatability of this research results. Furthermore, plain steel bars were used in the previous research but in this research deformed steel bars are used because the aim of this experimentation is not only the laboratory specimens but also the real structures and practical applicable material specifications. Surface texture and properties influence a lot on the corrosion rate of steel in concrete [13]. Then, four different types of environmental conditions were tested under impressed potential technique which is also unique to this research. The exact quantity of limiting corrosion current under water has also been a question mark which is solved in this paper.

The ideology follows that oxygen penetrates from the top surface and reacts with the cathodic steel producing hydroxyl ions. Then the hydroxyl ions move towards the anodic steel embedded in the chloride contaminated concrete and produce products of corrosion at the anode site. The steel embedded in



(a) Specimens Configuration and Chloride Contents



(b) Movement of Ions within the Concrete Test Specimen

Fig. 1. Experiment scheme: (a) typical experiment specimen and (b) movement of ions within in the concrete test specimen.

the chloride-free concrete is protected against corrosion by the alkalinity of the concrete. Throughout the experiment its action is exclusively cathodic.

Final examination after completion of the tests shows no corrosion phenomena on the steel surface due to either chloride diffusion from the lower concrete layer or due to carbonation from the concrete surface. This produces cathodic macro-cell current which can be measured using an ampere meter. Furthermore, a potentiostat was used to impress external potential difference and measure the change in cathodic current to find out the oxygen consumption rate variations under different environment conditions.

3. Environment exposure conditions

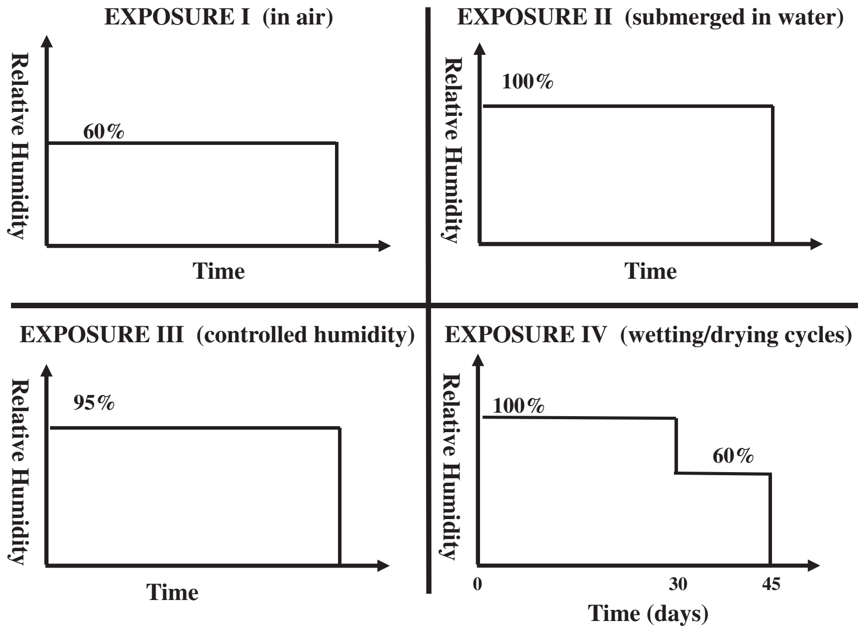
Environmental exposure conditions are as shown in Fig. 2a–c. Exposure I represent the normal benchmark condition of dry air at 60% R.H maintained in the humidity camber, while exposures II and III are intended to clarify the role played by oxygen under high moisture state in constant stable environments, while the exposure IV is intended to clarify the behavior of oxygen consumption rate under long wetting–drying cycle with enough duration for cathodic reaction to consume all the oxygen and make the corroding steel deficient in oxygen. Details are discussed in the following sections of this research paper. The exposure II specimens were kept all the time under water in order to find out the minimum corrosion rates which can still occur under water.

The exposure III had a background of difference of opinion between various researchers for the oxygen diffusion under high relative humidity conditions. Some researchers were of the opinion that oxygen diffusion is not limited under high relative humidity until the concrete is submerged under water. While, on the other hand, some researchers said that high relative humidity does inhibit the oxygen penetration under certain conditions in a given specified time interval [1,6,23–26].

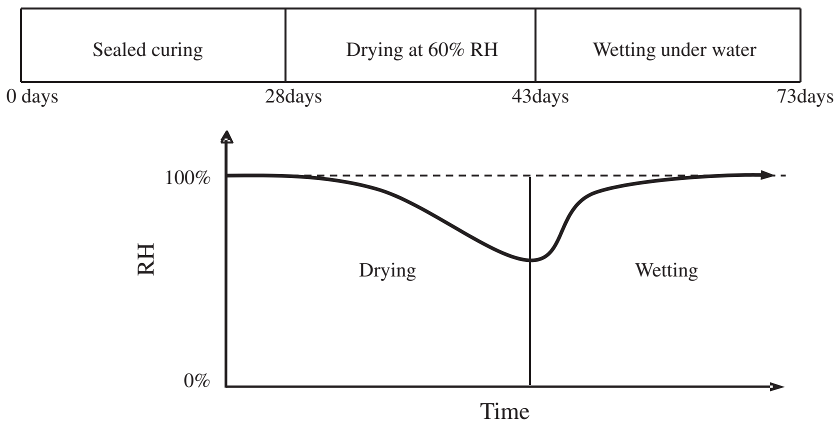
In order to investigate this effect the specimens were put in an environment control chamber set at a high relative humidity of 95% and 20 °C temperature. The machine used for this purpose is capable of producing and monitoring relative humidity and temperature with utmost accuracy. The reason for using 95% R.H in exposure III was to see if such a high relative humidity can limit the amount of oxygen by blocking the pores of concrete with moisture and reducing the connectivity required for transfer of gaseous oxygen from the environment to the steel bar surface. Exposure IV represents the changing cyclic environmental conditions. The wetting cycle was kept 1 month based on the calculations obtained from the past research results [1,6,23,24] that all the oxygen present in the specimen will be consumed by the corroding reinforcement bar in 1 month for the given experiment conditions and afterwards there will be not enough oxygen gas left for corrosion reaction. It was intended to observe the behavior of corroding rebar by measuring the oxygen consumption rate and cathodic current from full supply of oxygen until the oxygen gas is completely consumed. Similar intention was involved in keeping the drying cycle 15 days so that the concrete can fully recover from oxygen deficiency and its moisture content comes back to the air dry condition. This cycle repeats after every 45 days. Please note that in Fig. 2b the ordinate value in the upper portion of the figure does not represent any unit measurement but is merely a separation/dividing line for different conditions. The heights of plots/rectangles for the sealed, drying, and wetting conditions have been varied in order to easily differentiate between the three conditions for the easy understanding of the reader.

4. Experiment test setup and measurement

The specimens used for the potentiostatic tests were corrosion cells with an age of 28 days. They were placed under four different kinds of environmental humidity conditions as explained earlier. The reference electrode, in relation to which the potentiostat controlled the potential of the cathode, was a copper–copper sulfate ($Cu–CuSO_4$) electrode calibrated with manganese dioxide (MnO_2) electrode according to the previous research [23,24] connected to the concrete surface using a cotton swab containing soapy water for better contact of electrode and concrete surface [27]. A potentiostat (HAB-151, Hokuto Denkyo Co. Ltd.) was used to reduce the potential of the upper electrode (cathode) to a constant value. After this cathodic polarization, the oxygen reduction rate



(a) Four Exposure Conditions



(b) Alternate drying-wetting Cycle

Fig. 2. Environment exposure conditions of this experimentation.

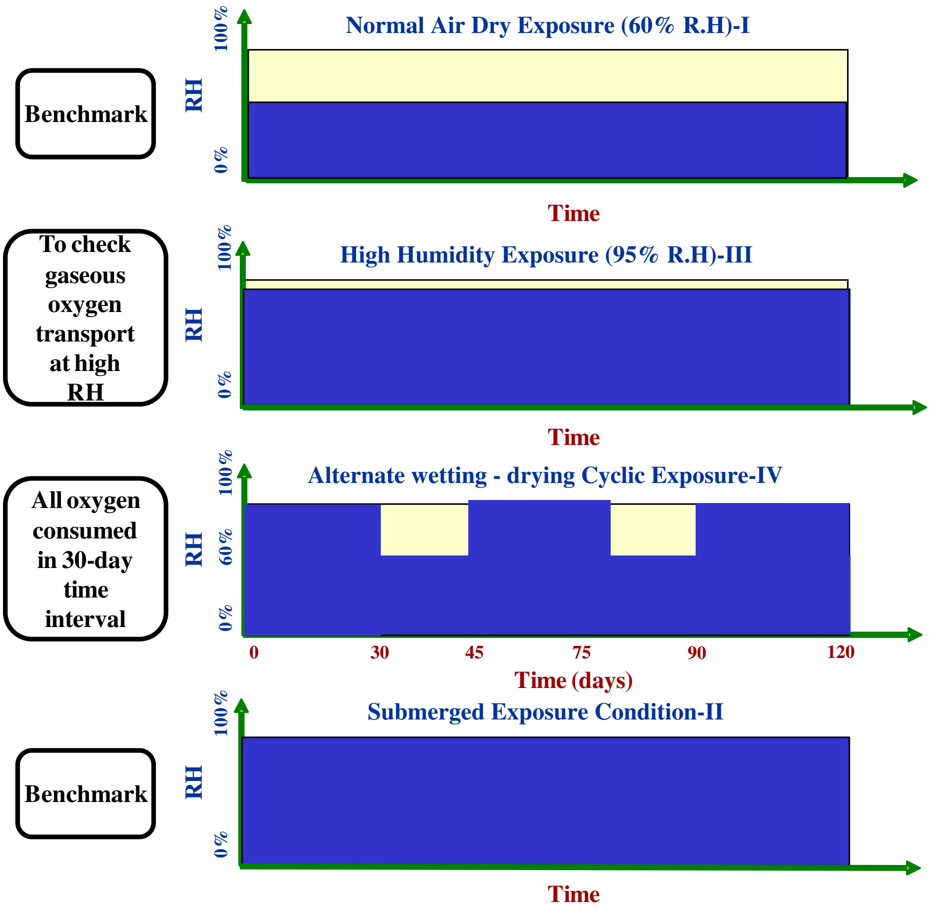
was determined by measuring the current flowing between the cathode and the lower electrode (anode). The current flowing with the potential adjusted to the reduced level was measured and evaluated at the current output of the potentiostat, using the standard current measuring system. Fig. 3 below diagrams the above setup and process used in the experiment scheme carried out in this research. It was reported in the previous research [23,24] that as long as potentials set at the cathode do not exceed the limiting potential for hydrogen production, current may be converted directly into the rate of oxygen consumption at the cathode (1A = 597 mg O_2/h).

5. Experiment results and discussions

5.1. Normal air dry condition

Experiment results are presented in the following pages. Figs. 4 and 5 show the experimental cathodic current and oxygen consumption rate measurements for normal air dry humidity conditions 'exposure condition I' which is already explained in the earlier sections. It must be noted that the first observation was taken after finishing a sealed curing time of 28 days. The first reading was very low as the specimen was deprived of oxygen for 28 days while under sealed curing. The oxygen consumption rate and cathodic current increase and reach the maximum value after 1 day and then decrease to a nominal gradual value which remained constant afterwards throughout the experiment. Different water to cement ratios were used, 0.45% for case shown in Fig. 4 and 0.3% for case shown in Fig. 5. But, no appreciable difference can be seen in the oxygen consumption rate. This suggests that *W*/*C* ratio does not limit the oxygen diffusion if the concrete is in normal air dry condition.

To confirm the fact observed in the above figure that oxygen diffusion in not limited under normal air dry conditions, another test was performed (Fig. 6) in which external potential of −500 mV was applied to the corroding steel. This was done to artificially increase the corrosion rate and observe the variation in the oxygen consumption rate. This is similar to what can be called as reverse of cathodic protection. Corrosion occurs by the flow of electrons or electric current and electrons need a potential difference to travel from one point to the other of the applied potential. The same



(c) Purpose of each Laboratory Exposure

Fig. 2 (*continued*)

Fig. 3. Test setup of experiment measurement.

technique has been applied here in this experimentation. Again it was observed that the oxygen consumption rate did not show any prominent variation with time and remained almost constant. This shows that the oxygen diffusion into concrete was not limited even by increasing the corrosion rate artificially.

Finally, the corrosion rate was further increased by apply even higher external potential on the negative side equal to −700 mV MnO_2 by using a potentiostat as explained earlier. But Fig. 7 shows no decrease in the oxygen consumption rate with time even at such a high corrosion rate reaching hydrogen discharge. But, it must be noted here that the area of anode in this experiment is very small. If the area of anode was large, it may lead to diffusion control at such a high external potential.

5.2. Submerged condition

In another test series, concrete corrosion cells were immersed under water (exposure II, already explained in the previous sections). As shown in Fig. 8 the oxygen consumption rate and cathodic current fall with time and reached a constant limiting value of 0.04 $\mu A/cm^2$. To confirm this limiting value, external potential of −500 mV was imposed on the corroding circuit. It was observed that the cathodic current increased for a couple days and then came back to its original limiting value. This means that some oxygen was still present in the vicinity of the corroding reinforcement steel bar which may be adsorbed to the surface of steel and was consumed after applying high polarization. After that the current remained constant.

As a final check the external potential was increased again up to −700 mV towards the negative side. It was observed that the current increased again for a couple of days and then again came back to its limiting value showing that some oxygen was still adsorbed to the surface of steel even after 3 months of underwater immersion. Finally it is concluded that the corrosion reaction does not completely stop under water but continues at a very low value (0.04 $\mu A/cm^2$) which is actually of not much practical importance and cannot corrode the reinforced concrete structure easily. This

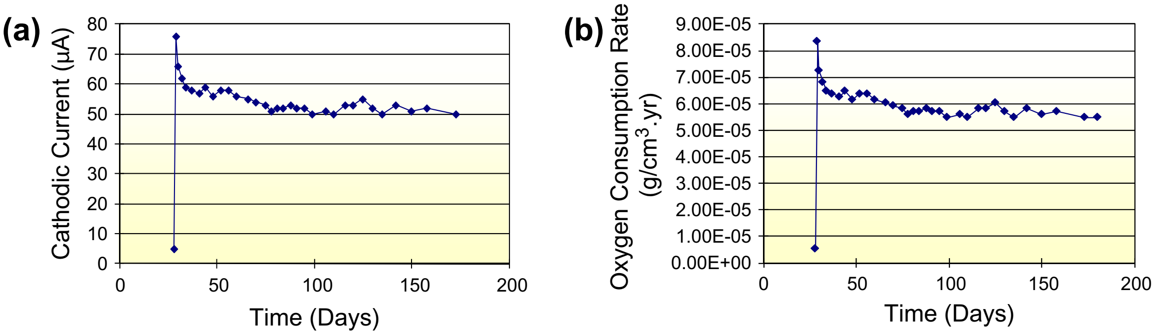


Fig. 4. (a) and (b) Current and oxygen consumption rate for air dry humidity condition (*W*/*C*: 0.45, 0 mV).

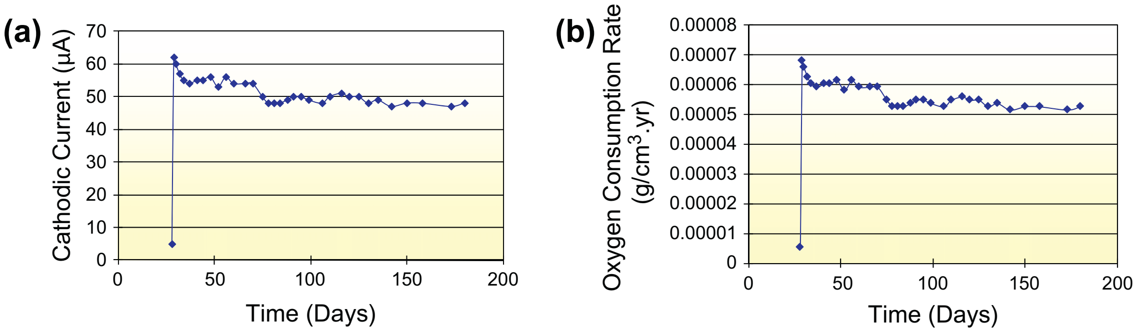


Fig. 5. (a) and (b) Current and oxygen consumption rate for air dry condition (*W*/*C*: 0.3, 0 mV).

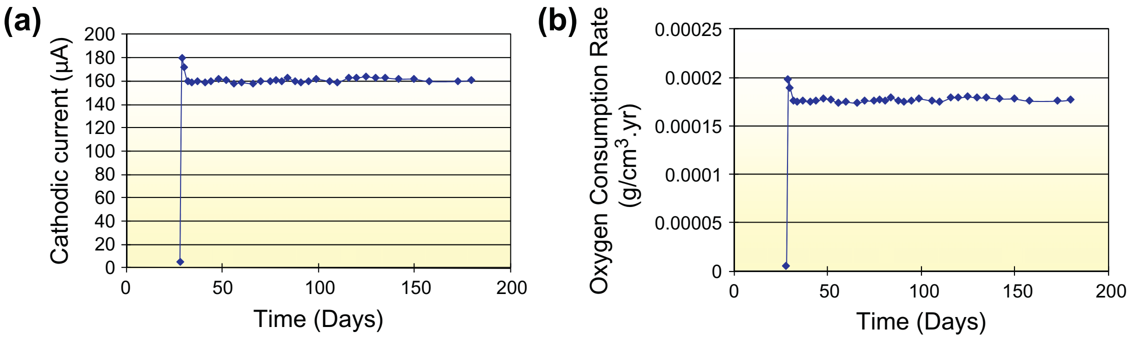


Fig. 6. (a) and (b) Current and oxygen consumption rate for air dry condition (*W*/*C*: 0.45, −500 mV).

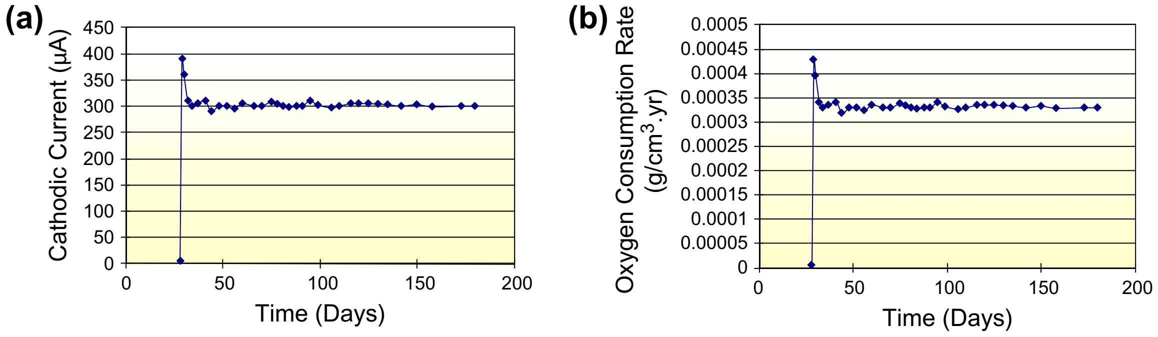


Fig. 7. (a) and (b) Current and oxygen consumption rate for air dry condition (*W*/*C*: 0.45, −700 mV).

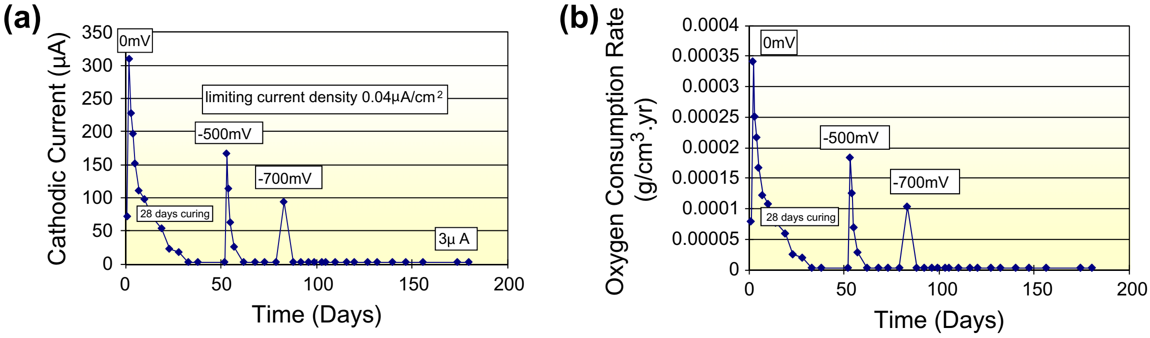


Fig. 8. (a) and (b) Current and oxygen consumption rate (exposure II and *W*/*C*: 0.45).

explains the reason that why reinforced concrete structures which are submerged under very saline water in the sea does not corrode. Although the sea water has a lot of chlorides but due to being constantly under water corrode only under the limiting current density as shown by the experiments in this research.

5.3. High relative humidity condition

In case of specimen under exposure III right after putting the specimen in 95% R.H condition in the environment control chamber, the cathodic current increased up to a value around 300 μA from the normal air dry first value of 70 μA. Afterwards, the current fall gradually reaching a constant value ranging around 40 μA. This is a little bit lower than the average value at normal air dry condition in the range of 60 μA as already discussed. It means that oxygen consumption rate does get reduced by limited oxygen diffusivity into the concrete at such a high relative humidity of 95%. It can be concluded that high relative humidity effects the oxygen diffusion by blocking its way up to the corroding steel rebar but the blockage offered is much lower as compared to submerged condition in which the relative humidity is 100% as the cathodic current although less than that in normal air dry condition is still much higher than the limiting current of 0.04 μA obtained in under water current measurements.

After observing no further fall of cathodic current and attaining a constant oxygen consumption rate, the corroding macro-cell was exposed to an external potential of −500 mV MnO_2. The cathodic current rise to a certain value but then again attained a constant oxygen consumption rate showing no fall in the current with the passage of time. It means that even at an elevated corrosion rate, the demand of oxygen was met completely as there was not fall in the oxygen consumption rate. The high relative humidity although effects the oxygen diffusion to some extent but could not block the oxygen diffusion even at this high rate of corrosion. A similar trend was seen at a further negative potential increment of −700 mV. Neither limiting current value was achieved nor was any decrease in the cathodic current observed as shown in Fig. 9 below. The current and resulting oxygen consumption rate remained at a more or less constant value until the end of 6 months of experiment duration. But, it must be noted here again that the area of anode in this experiment is small. If the area of anode was large, it may lead to diffusion control at such a high corrosion rate even in air condition.

In a nutshell, it can be said that no doubt the effect of high relative humidity such as 95% is there, and cannot be neglected. But, the effect is not as much as in case of submerged condition. Although the oxygen consumption rate is checked at high relative humidity if the concrete cover is thick and *W*/*C* is low or some admixture is used to reduce the porosity of concrete, but it is not possible to bring the corrosion rate under diffusion control, unless the relative humidity is 100%. In all cases of relative humidity less than one hundred percent, the diffusivity of concrete is never less enough to make the system oxygen diffusion controlled.

5.4. Alternate wetting/drying condition

Fig. 10 below shows the experiment results for specimen under exposure IV. Similar methodology and treatment was given as discussed for the other three cases in the above sections of this research. The cathodic current was measured first at zero external potential (only under normal chloride induced natural potential). The experiment values of current and oxygen consumption rate obtained in the specific wetting and drying cycles correspond to what was obtained in the previous experiments for each of the submerged and normal air dry exposure conditions. First two cycles are under no external potential while the third cycle results are under the influence of impressed potential of −500 mV MnO_2. It can been seen that the cathodic current increase with

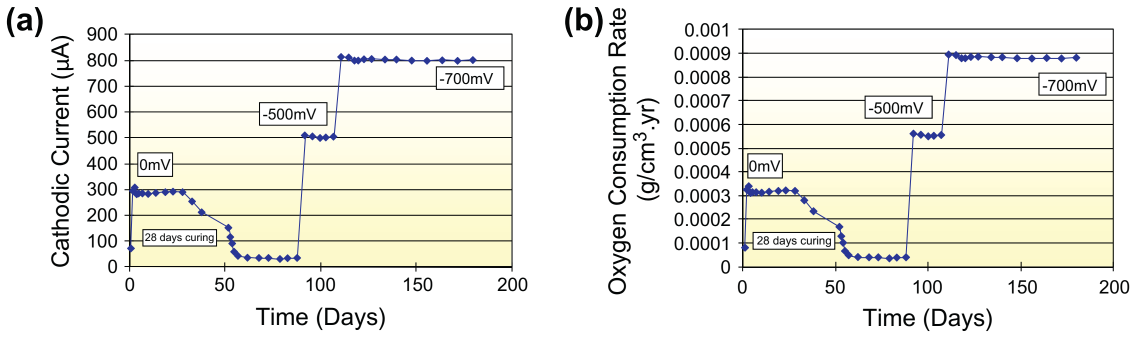


Fig. 9. (a) and (b) Current and oxygen consumption rate (exposure III and *W*/*C*: 0.45).

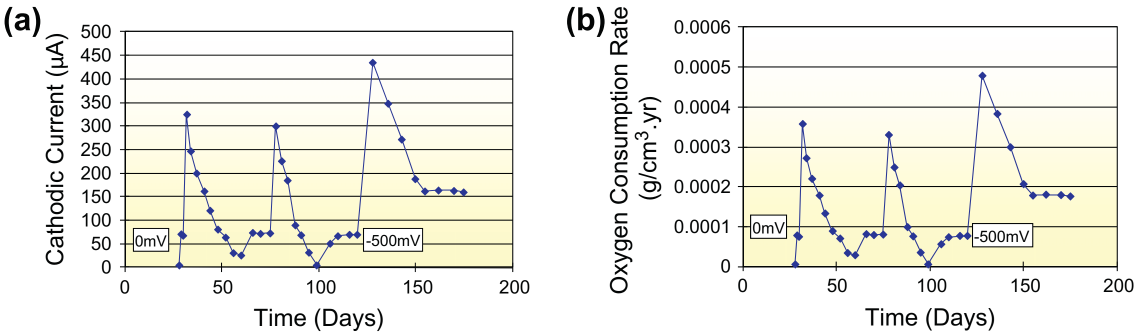


Fig. 10. (a) and (b) Current and oxygen consumption rate (exposure IV and *W*/*C*: 0.45).

the start of immersion cycle and then gradually reduces to a value below the air dry normal potential and then comes back to air dry normal potential at the start of every drying cycle.

6. Conclusions

The oxygen consumption rate was calculated based on an assumed relation with the cathodic current due to chloride induced corrosion under variable humidity environment through extensive laboratory experimentation involving impressed potential macro-cell corrosion reaction for which the previous research data is limited. Salient conclusions of this research are as follows:

(a) The exact quantity of limiting corrosion current under water has been a question mark in the past which is solved in this research. For the test conditions of this research, the limiting corrosion current under water has been experimentally determined as 0.04 μA/cm^2.

(b) It can be concluded from the experiment results that the diffusion of oxygen is a vital limiting factor for corrosion reaction only when the concrete is either submerged or is in high relative humidity environment (95% in this research) with thick concrete cover and low *W*/*C* ratio (0.3 in this research).

(c) The laboratory tests show that the diffusion of oxygen through the concrete cover is only a significant limiting factor for the corrosion rate when the concrete around the reinforcement is water-saturated and the oxygen within the concrete near the reinforcement surface is consumed by the cathodic reaction of the corrosion process. However, it must be noted here that the ratio of anode area to the total volume of concrete is small in this experiment. If the area of anodic steel was large, there is a possibility that it may lead to oxygen diffusion control even in normal air dry condition at very high corrosion rate.

(d) It was seen from the alternate wetting–drying cyclic exposure tests that the cathodic current increase with the start of immersion cycle and then gradually reduces to a value below the air dry normal potential and then comes back to air dry normal potential at the start of every drying cycle.

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