*- Technical Paper -*

# RECOVERY IN MIX POTENTIAL AND POLARIZATION RESISTANCE OF STEEL BAR IN CEMENT HARDENED MATRIX DURING EARLY AGE OF 6 MONTHS -SEA-WATER MIXED MORTAR AND CRACKED CONCRETE-

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ABSTRACT

Sea-water contains high chloride ion concentration which can promote corrosion of steel bar. However, it has a potential to be used as mixing water. On the other hand, cracks should be avoided in reinforced concrete for durability reason. Previous research showed thicker cover depth gave positive effect on potential and passivity film condition of steel bar. This paper focuses on the electrochemical property of steel bar embedded in cement hardened matrix during very early age until 6month after casting, under continuing hydration activity. In addition, electrochemical performance showed positive trend by increment in time as well as utilization of mineral admixture.

Keywords: electrochemical method, corrosion potential, sea-water mixing, cracked concrete

## 1. INTRODUCTION

In general, sea-water as mixing water and cracks are supposed to be avoided in reinforced concrete for durability reason. The high chloride ion concentration, leads to the higher corrosion rate in the presence of oxygen and water. Therefore, sea-water it is not recommended for reinforced concrete as mixing water [1]. N. Otsuki reported, partial replacement of cement by BFS showed good performance when sea-water is used as mixing water in view point of durability [2]. On the other hand, in the presence of cracks, the corrosion rate will be larger. Cracks reduce the service life of the structure by permitting access of moisture, chloride ions and oxygen to reach the surface reinforcing bar, thus, accelerating the onset of corrosion. In contrary, it is expected that with any size of cracks, the corrosion rate of steel bars in concrete will depend much more on the surrounding concrete (i.e., concrete resistance, oxygen permeability, and chloride ion content) rather than crack widths [3]. In order to investigate the effect of sea-water as mixing water, and crack in associated with exposure condition as well as utilization of mineral admixture ($BFS_{4000}$) on corrosion potential of steel bar in the early 6months, the experimental laboratory study was carried out. Electrochemical methods such as half-cell potential and anodic polarization curve have been used for evaluating electrochemical performance of steel bar in sea-water mixed mortar and cracked concrete.

## 2. EXPERIMENTAL DETAILS

### 2.1 Materials

The specimens were made with Ordinary Portland Cement (OPC) and Blast Furnace Slag ($BFS_{4000}$). The physical properties are shown in Table 1. Further, the physical characteristics and chemical components are presented in Table 2.

Table 1 Physical properties

| Material | Density, g/cm$^3$ | Fineness, cm$^2$/g | Water Absorption % |
|---|---|---|---|
| OPC | 3.16 | 3300 | |
| $BFS_{4000}$ | 2.91 | 4060 | |
| Lime Stone | 2.70 | - | 0.25 |
| Sea-water | 1.03 | | |
| Washed Sea Sand | 2.53 | - | 2.08 |

Table 2 Physical characteristics and chemical component

| Items | OPC | BFS4000 |
|---|---|---|
| MgO, % | 1.2 | 5.32 |
| SO3, % | 2.23 | - |
| LOI, % | 2.15 | 0.33 |
| Total alkali, % | 0.51 | - |
| Chloride, % | 0.019 | 0.006 |

Table 3 Mix proportions of mortar -Sea-water mixing series-

| Material | N-TW | N-SW | B-SW |
|---|---|---|---|
| W/B, % | 50 | 50 | 50 |
| s/a, % | 45 | 45 | 45 |
| Tap water, kg/m$^3$ | 255 | - | - |
| Sea-water, kg/m$^3$ | - | 255 | 255 |
| Cement, kg/m$^3$ | 510 | 510 | 255 |
| BFS, kg/m$^3$ | - | - | 255 |
| Sand, kg/m$^3$ | 1508 | 1525 | 1515 |

### 2.2 Mix Proportions

The mix proportions of mortar are summarized in Table 3. The water to binder ratio (W/B) of mortar was 50%. There is three type of mix, OPC with tap water mixing for control, OPC with sea-water mixing and BFS with sea-water mixing. The chloride content of

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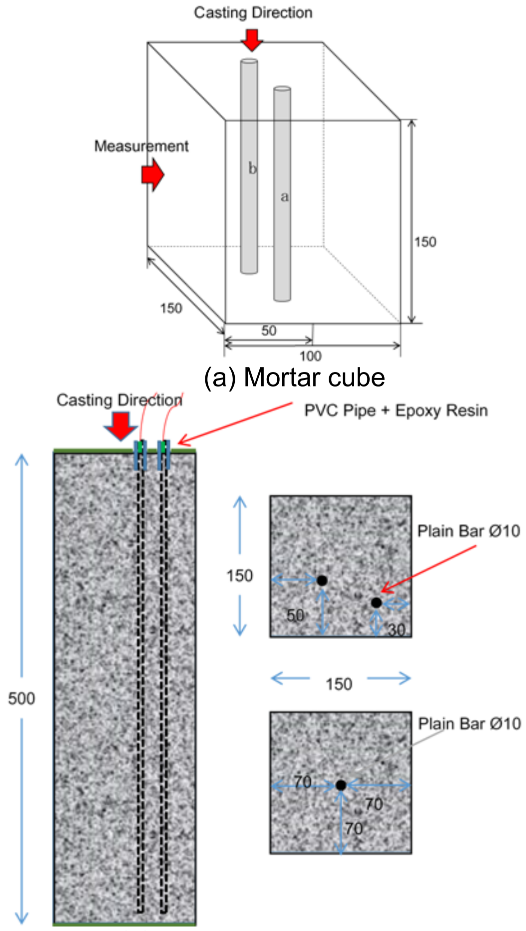
mortar mixed with natural sea-water is 3.35 kg/m³, much larger than 1.2 kg/m³ as chloride threshold for corrosion initiation, according to the JSCE standard [4]. In addition, the mix proportions of reinforced concrete prisms are summarized in Table 4. The water to binder ratio (W/B) of concrete was 50%. Lime stone with 20 mm maximum size was used as coarse aggregate and washed sea sand was used as fine aggregate. The ratio of fine aggregate to total aggregate volume (s/a) was 45%. "N" is for OPC mixed concrete, "B" is for BFS mixed concrete.

Table 4 Mix proportions of concrete
-Cracked concrete series-

| Material | N | B |
|---|---|---|
| W/B, % | 50 | 50 |
| s/a, % | 45 | 45 |
| Tap water, kg/m³ | 160 | 160 |
| Cement, kg/m³ | 320 | 160 |
| BFS, kg/m³ | - | 160 |
| Sand, kg/m³ | 805 | 800 |
| Gravel, kg/m³ | 1030 | 1023 |
| Water-reducing, kg/m³ | 1.3 | 1.3 |
| Air-entraining agent, mL/m³ | 2000 | 2000 |
| Slump, cm | 5.5 | 6.5 |
| Air content, % | 4.0 | 5.0 |



(a) Mortar cube

(b) Reinforced concrete prism

Fig.1 Detail of specimen (in mm)

## 2.3 Specimen Design

Mortar cube specimen with dimension of 150x 100x150 mm³ is prepared for sea-water mix series. Round steel bar with diameter ϕ13 mm, and with cover thickness 50 mm were embedded. Further, the dimension of the reinforced concrete prism specimen is 150x150x500 mm³. Plain bar with diameter ϕ10 mm, and with various cover depth were embedded. Detail of mortar cube and reinforced concrete prism specimens design is shown in Fig.1. Cylindrical mortar and concrete, with 50 mm of diameter and 100 mm of height, and 100 mm of diameter and 200 mm of high, were prepared to check mechanical properties, respectively. The specimen was demolded at one day after casting, then immediately cured in the tap water until 28 days. After that, five side surface for mortar cube and both ends surface for reinforced concrete prism were coated by epoxy resin before subjected to various exposure condition. Furthermore, specimens were set into various exposure condition after 28 days with full immersion condition. For dry-wet cycle, one cycle was 7 days, consisted of 5 days drying (air dry) and 2 days wetting (immersed in tap water and sea-water). Then, for continuous immersed, specimen put into 3% NaCl solution. Specimen code for mortar cube, for example "N50T-T" means "cement type (N) – W/B (50) – mixing water (T) – exposure water (T)". Variation of specimen and exposure type of mortar cube were presented in Table 5 and Table 6, respectively. In addition, variation of reinforced concrete prisms were presented in Table 7.

Table 5 Variation of mortar specimen
-Sea-water mix series-

| Cement Type | Specimen Name | Type of Exposure I | II | III | IV |
|---|---|---|---|---|---|
| OPC | N50 | ○ | ○ | ○ | ○ |
| $BFS_{4000}$ | B50 | | ○ | | ○ |

Table 6 Type of exposure
-Sea-water mix series-

| Type | Mix | Curing/Dry-wet Cycle (5/2) |
|---|---|---|
| I | T | T |
| II | S | T |
| III | T | S |
| IV | S | S |

Notation: T = tap water; S = sea-water

Table 7 Variation of concrete specimen
-Cracked concrete series-

<table>
<tr><td rowspan="7">Type</td><td colspan="7">Cover Depth, mm</td></tr>
<tr><td colspan="2">30</td><td colspan="2">50</td><td>70</td><td>30</td><td>50</td></tr>
<tr><td colspan="7">Exposure Condition</td></tr>
<tr><td colspan="5">Dry-Wet Cycle</td><td colspan="2">Continuous Immersed</td></tr>
<tr><td>S.W</td><td colspan="4">T.W</td><td colspan="2">NaCl (3%)</td></tr>
<tr><td colspan="7">Crack Width, mm</td></tr>
<tr><td>0.3</td><td>0.3</td><td>0.3</td><td>0.5</td><td>0.3</td><td>0.3</td><td>0.5</td></tr>
<tr><td>OPC</td><td>A</td><td>B</td><td>B</td><td>C</td><td>D</td><td>E</td><td>F</td></tr>
<tr><td>$BFS_{4000}$</td><td>G</td><td>H</td><td>H</td><td>-</td><td></td><td>J</td><td>-</td></tr>
</table>

After 28 days curing, all reinforced concrete prism specimens were cracked under two point flexural loading. Before loading, the π-gauge was mounted on the bottom side to measure the crack width. Photo 1 shows loading condition for pre-cracking and crack width measurement by crack scale. The continuous load was applied slowly by manual for visual inspection of crack on the bottom side near mid span. The load was increased until the crack width reached the target width. Then, the beams were completely unloaded. In addition, at ten points, residual crack width was measured just after loading release in order to determine average crack width at bottom and two lateral sides surface by using scale crack. The crack width for each specimen are summarized in Table 8. The specimen code for cracked concrete, for example "N-30-0.3-SW" means "cement type (N) – cover depth (30) – crack width (0.3)– exposure condition (SW)".

Table 8 Crack width

| Code | Specimen | Crack (mm) Target | Max. D.L | Average S.C |
|---|---|---|---|---|
| A | N-30-0.3-SW | 0.30 | 0.30 | 0.25 |
| B | N-30-0.3-TW | 0.30 | 0.34 | 0.15 |
| B | N-50-0.3-TW | | | |
| C | N-50-0.5-TW | 0.50 | 0.65 | 0.50 |
| D | N-70-0.3-TW | 0.30 | 0.30 | 0.20 |
| E | N-30-0.3-NaCl | 0.30 | 0.48 | 0.30 |
| F | N-50-0.5-NaCl | 0.50 | 0.45 | 0.40 |
| G | B-30-0.3-SW | 0.30 | 0.34 | 0.15 |
| H | B-30-0.3-TW | 0.30 | 0.34 | 0.15 |
| H | B-50-0.3-TW | | | |
| J | B-30-0.3-NaCl | 0.30 | 0.32 | 0.20 |

Notation; D.L: data logger; S.C: scale crack

(a) (b)

Photo 1 (a) Pre-cracking loading; (b) Crack width measurement

## 2.4 Experimental Methods

The corrosion potential of steel bar in concrete was evaluated by half-cell potential, $E_{corr}$ (half-cell rebar/concrete) that is, potential difference (in voltage) toward a reference electrode [5]. The half-cell potential was measured after one hour from the end of wet cycle by using the silver/silver chloride reference electrode (SCE) at crack location (Fig.2). Then, potential value is converted to the value against copper/copper sulfate reference electrode (CSE). According to the ASTM C876-09 [6], more positive than -200 mV indicates 90% probability of no corrosion, between -350 mV and -200 mV indicates uncertainty of corrosion and more negative than -350 mV indicates 90% probability of corrosion.

Anodic polarization curve was measured in order to evaluate the condition of passivity film on the steel surface embedded in concrete as presented in Table 9. When the current density becomes larger, generally it means the grade of passivity film of steel bars becomes worse [7]. The fact that high current density implies the polarization resistance of concrete is lower as well. In the measurement of polarization curve, the potential of the steel bar ($E_{corr}$) was shifted to +700 mV from the natural potential with a sweep rate of 50 mV/min. The current was recorded continuously. The maximum current density obtained from anodic polarization curve was then used to judge passivity grade.

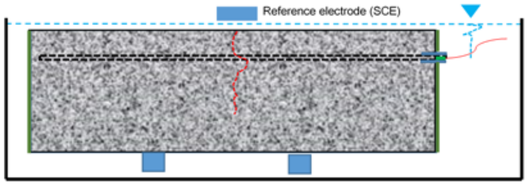



Fig.2 Measurement area of specimen

Table 9 Grade of passivity film associated anodic polarization curve

| Grade | Polarization curve | Condition |
|---|---|---|
| Grade 0 | potential 0.2-0.6V, current density is over 100 μA/cm² at least one time | no passivity exist |
| Grade 1 | potential 0.2-0.6V, current density is 10-100 μA/cm² | certain degree of passivity exist |
| Grade 2 | potential 0.2-0.6V, current density is over 10 μA/cm² at least once but not to qualified to Grade 1 and 3 | |
| Grade 3 | potential 0.2-0.6V, current density is 1-10 μA/cm² | |
| Grade 4 | potential 0.2-0.6V, current density is over 1 μA/cm² at least once but not qualified to Grade 1, 2 and 3. | |
| Grade 5 | potential 0.2-0.6V, current density is less than 1 μA/cm² | excellent passivity exist |

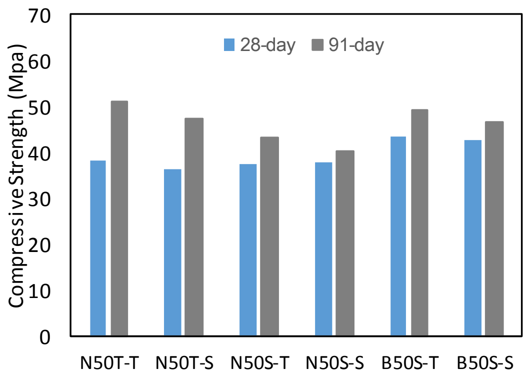



Fig.3 Compressive strength of mortar

# 3. RESULT AND DISCUSSION

## 3.1 Mechanical Properties

Fig.3 shows the compressive strength of sea-water mixed mortar. The control specimen was made of Ordinary Portland Cement and mixed by tap water (N50T-T). Specimen mixed with sea-water with OPC showed decreasing in compressive strength (N50S-T). This implies that the compressive strength is reduced due to sea-water as mixing water. Also, sea-water cured mortar (N50T-S) shows smaller strength than tap water cured mortar (N50T-T). In addition, compressive strength of all specimens shows increment by time, that is, the strength at the 91 day is larger than the strength at 28day. Moreover, partial replacement of cement by Blast Furnace Slag for 50% by mass of cement showed positive effect in compressive strength. This is shown in the specimens with sea-water as mixing and curing water.

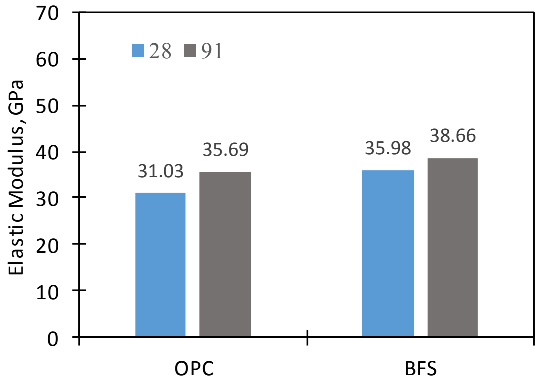



Fig.4 Compressive strength of concrete

Durability of reinforced concrete depend on quality of concrete itself. Therefore, mechanical property is one parameter for assessment of durability of reinforced concrete. Fig.4 represents the strength development of OPC concrete and BFS concrete with a curing period 28 and 91 days. Both for OPC and BFS, cylindrical specimen for compressive strength test was casted with tap water for mixing and curing water. It was observed that compressive strength was increased up to 91day from 28day. BFS concrete showed slightly larger strength and increment compared with OPC. It means that partial replacement of cement by BFS gives positive effect in mechanical properties, such as compressive strength.

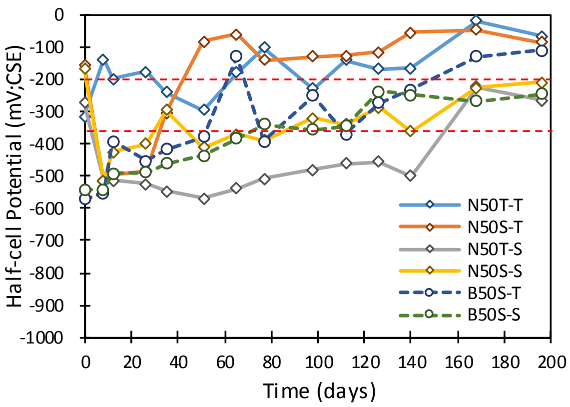



Fig.5 Half-cell potential (sea-water mix)

### 3.2 Half-cell Potential of Steel Bar in Sea-water Mixed Mortar

Half-cell potential of sea-water mixed mortar until 6 month is shown in Fig.5. The good performance of N50T-T is a proof that tap water as mixing and curing water is desirable for reinforced concrete. For N50S-T, in the early 14 days the potential fallen down lower -350 mV, however, due to the use of tap water as curing water, the potential recovered to more positive than -200 mV after 60 days exposure. The positive trend is shown in the BFS specimen B50S-T and B50S-S, in the initial measurement, the potential was lower than -500 mV, and however, it was recovered by time, to more positive, around -200 mV. As shown in this figure, until 6month, the same phenomenon was found on both BFS and OPC specimens. This is considered that the effect of sea-water as mixing water emerged in very early age until 20 to 50 days, however, as hydration proceed, the potential is gradually improved until about -200 mV during 6 month. In addition, after 6 month of exposure, tap water curing specimen categorize as 90% probability of no corrosion while sea-water curing specimen categorize as uncertainty of corrosion.

### 3.3 Half-cell Potential of Cracked Concrete

Fig.6 shows half-cell potential of OPC specimen with different cover depth (30 mm, 50 mm and 70 mm) and crack width. The effect of cover depth until 6month of exposure cannot be observed. The potential value is categorized as “uncertainty” or “90% probability of no corrosion”. After 140 days, the potential of N-50-0.5-TW extremely fallen down to more negative than -350 mV. This may be due to the larger crack width, which affected to the result of potential value. As seen in the potential trend of N-50-0.5-NaCl, the exposure condition largely affected the potential than crack width. The potential of N-50-0.5-NaCl fallen down to -700 mV at the 40 days, however, it recovered to about -350 mV at the 6 month.

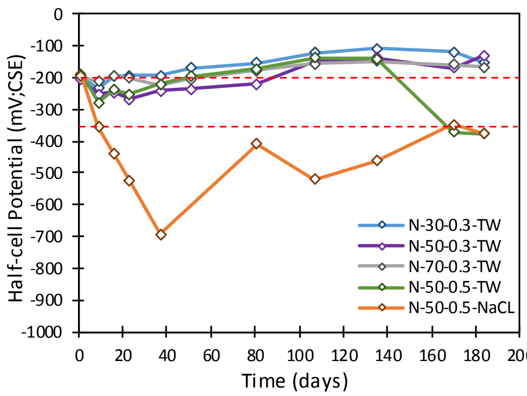



Fig.6 Half-cell potential (effect of cover depth and crack width)

Half-cell potential of specimens under various exposure condition until 6 month as presented in Fig.7. The effect of exposure condition is observed on half-cell potential with same cover depth and same crack width. Dry-wet cyclic in the tap water showed potential more positive value than -200mV, which indicates the 90% probability of no corrosion. Then, dry-wet cyclic in sea-water showed potential more negative than -350mV from 0 day to 80 days, however, after 80 days, the potential became between -350mV and -200mV which indicates the uncertainty of corrosion. The worse condition is found in the continuous immersion in 3% NaCl solution, where the potential fallen down to more negative than -350mV which indicates the 90% probability of corrosion. The potential level is different for these three cases, however, slight improvement in potential can be seen for all cases, until 6 month.

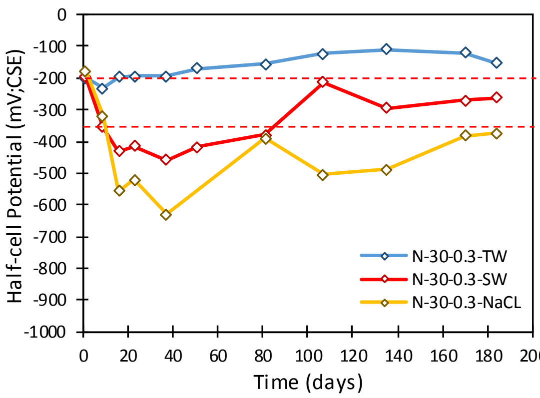



Fig.7 Half-cell potential (effect of exposure condition)

From various information, previous study and literature, corrosion behavior of steel bar embedded in concrete appears to be very much dependent on cement type. Half-cell potential value of specimen with additional mineral admixture (BFS) is shown in Fig. 8. The worse condition is shown in the immersion in 3% NaCl solution, for the same cover depth and crack width, and followed by the dry-wet cycle exposure in sea-water and tap water. Comparing OPC mix "N" and BFS mix "B", it is clear that the potential of "B" is lower than that of "N". The very negative half-cell potential for BFS concrete was also observed by Jarrah [8] and may be attributed to the reducing effects of Sulphur substances like $S^{2-}$ and not to a steel corrosion effect. Manganese and other substances such as sulfides plentiful in blast furnace slag where is like a glassy by-product of making iron, that substance provide a reducing effect and lead to very negative potential values. In addition, after around 50 days, 180 days potential of all cases show improvement or stability until 6 month.

### 3.4 Anodic Polarization Curve

Condition of passivity film of steel bar until 6 months exposure is summarized in Table 10 for sea-water mixed mortar. Measurement was conducted two times, 0 month (before exposure) and after 6 month exposure. In contrary with the result of half-cell potential, BFS specimen showed degradation in passivity film from 0 month to 6 month. On the other hand, OPC specimen showed clear recovery of passivity film condition from 0 month to 6 month. One reason for the recovery in OPC specimen, may be due to the hydration of cement, followed by increase in the pH of concrete. The passive film remains stable when alkalinity of the concrete exceeds around pH 12, and the steel bar could be protected from further corrosion. Meanwhile, BFS specimen is rather dense and impermeable compared to OPC, and also it has the chloride binding ability.

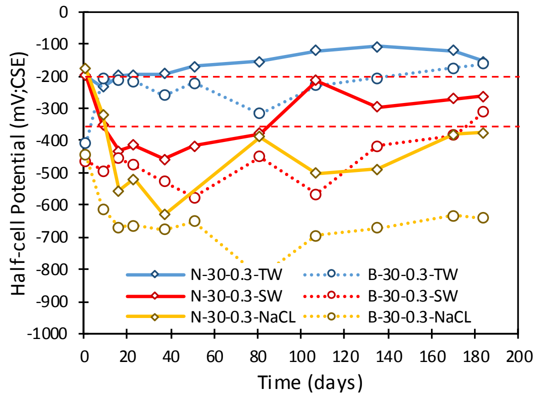



Fig.8 Half-cell potential (effect of additional admixture)

Table 10 Summary of current and passivity condition sea-water mixed

| Specimen | Current μA/cm² | | Passive Grade | | Ratio | |
|---|---|---|---|---|---|---|
| | 0M | 6M | 0M | 6M | 6M/0M μA/cm² | 6M-0M Grade |
| N50T-T | 7.63 | 0.98 | III | V | 0.1 | +1 |
| N50S-T | 5.18 | 0.97 | III | V | 0.2 | +1 |
| N50T-S | 14.07 | 3.03 | II | IV | 0.2 | +2 |
| N50S-S | 7.60 | 2.63 | III | IV | 0.3 | +1 |
| B50S-T | 1.55 | 12.98 | IV | II | 8.4 | -2 |
| B50S-S | 4.48 | 15.42 | IV | II | 3.4 | -2 |

The effect of cover depth on anodic polarization curve can be observed in the specimen, N-30-0.3-TW, N-50-0.3-TW and N-70-0.3-TW in the Fig.9. It shows that the passivity film is better as increasing the thickness of cover depth. Even in the cracked concrete, eventually the rate of corrosion could be reduced by increasing cover depth [9].

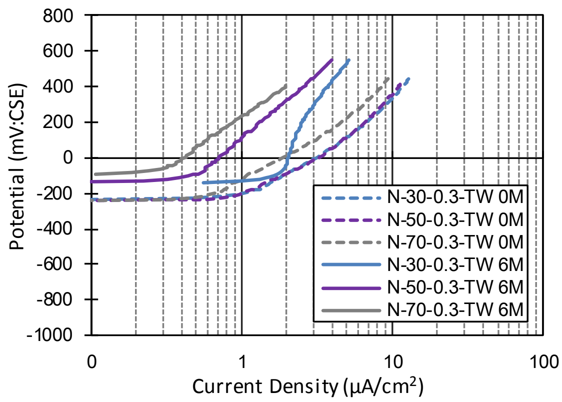



Fig. 9 Anodic polarization curve for N-30-0.3-TW, N-50-0.3-TW and N-70-0.3-TW (0 and 6 month)

Exposure condition effect is also derived. The worst passivity film condition for same cover depth and same crack width is for N-30-0.3-NaCl (15.12 μA/cm²) followed by N-30-0.3-TW (13.01μA/cm²) and N-30-0.3-SW (8.47μA/cm²). However, anodic polarization curve of the specimen exposed in various condition showed good recovering until 6 months exposure.

The specimen with BFS shows larger current density than that of OPC specimen, for the same cover depth and same crack width, as B-30-0.3-NaCl is 21.17μA/cm², B-30-0.3-TW is 17.71μA/cm², B-30-

0.3-SW is 18.82μA/cm$^2$, N-30-0.3-NaCl is 15.12μA/cm$^2$, N-30-0.3-TW is 13.01μA/cm$^2$, and N-30-0.3-SW is 8.47μA/cm$^2$ at 0 day measurement. Furthermore, after 6 month exposure, BFS specimen showed good recovery on passivity film than OPC specimen. It is shown in Table 11 that BFS specimen obtained smaller current density than OPC specimen, except specimen exposed into continuous immersed in 3% NaCl obtained same current density. It was reported that internal binding capacity of cement pastes or mortars increase greatly when OPC is replaced by BFS, up to 70% of replacement ratio [10].

In addition, it can be seen that in all specimen, passivity film condition is recovered or even stable (N-50-0.5-TW) from 0 day to 6 month. This is due to the hydration progress of cement, therefore, the interface between steel and cement hardened matrix is much improved during very early age until 6 months.

Table 11 Summary of current and passivity condition cracked concrete

| Specimen | Current μA/cm$^2$ | | Passive Grade | | Ratio 6M/0M | 6M-0M |
|---|---|---|---|---|---|---|
| | 0M | 6M | 0M | 6M | μA/cm$^2$ | Grade |
| N-30-0.3-SW | 8.47 | 4.32 | III | IV | 0.5 | +1 |
| N-30-0.3-TW | 13.01 | 5.21 | II | III | 0.4 | +1 |
| N-50-0.3-TW | 11.78 | 4.07 | II | IV | 0.3 | +2 |
| N-50-0.5-TW | 3.96 | 3.88 | IV | IV | 1.0 | - |
| N-70-0.3-TW | 9.28 | 1.96 | III | IV | 0.2 | +1 |
| N-30-0.3-NaCl | 15.12 | 8.19 | II | III | 0.5 | +1 |
| N-50-0.5-NaCl | 11.62 | 3.97 | II | IV | 0.3 | +2 |
| B-30-0.3-SW | 18.82 | 4.29 | II | IV | 0.2 | +2 |
| B-30-0.3-TW | 17.71 | 2.98 | II | IV | 0.2 | +2 |
| B-50-0.3-TW | 13.75 | 2.73 | II | IV | 0.2 | +2 |
| B-30-0.3-NaCl | 21.17 | 8.39 | II | III | 0.4 | +1 |

## 4. CONCLUSIONS

Sea-water used as mixing and curing water affected to the corrosion potential and passivity condition of steel bar. As well, cover depth and exposure condition along with additional mineral admixture. Based on the experimental study focusing on the very early age until 6 month of exposure, following conclusions are drawn:

1. Sea-water decreased half-cell potential more negative in very early age just after casting. However, time-dependently it changed to positive until the age of 6 month.
2. Partial replacement of cement by $BFS_{4000}$ with sea-water as mixing and curing water showed positive effectiveness. The potential trend showed recovery as increasing in the exposure time.
3. The same recovery or improvement was found in the passivity film condition judged by anodic polarization curve even in the sea-water mixed concrete.
4. Until 6 month exposure, the effect of cover depth was not clearly seen on corrosion potential. However, on anodic polarization curve, it was shown that the thicker the cover depth is, better the passivity film is.
5. The exposure condition gave more influence rather than crack width on potential and also passivity film condition.
6. The severest exposure conditions on corrosion potential was the continuous immersion in 3% NaCl solution, followed by the dry-wet cyclic in sea-water and then in tap water.
7. In cracked concrete, improvement in potential and passivity film was found in very early age cement hardened matrix, both for OPC mix and BFS mix.

## ACKNOWLEDGEMENT

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