



# Evaluation of Permanent Embeddable Reference Electrodes in Solution and Concrete Specimen

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#### Summary

Monitoring concrete structures is crucial for assessing the corrosion risk of steel reinforcement and optimizing maintenance strategies. This study evaluated the MnO<sub>2</sub>/NaOH, Ag/AgCl, and Ti/MMO permanent, embeddable reference electrodes, which are rarely known or used in Brazil for corrosion monitoring. The electrodes underwent assessment tests, immersion tests in solutions simulating concrete pore water, and a test in a reinforced concrete specimen. Results showed that the relative potentials of the electrodes in each immersion test did not always align with those in the assessment test, emphasizing the influence of the medium's characteristics on electrode selection and performance evaluation. However, the potentials remained stable and reproducible among units of each type in all tests. Long-term field tests in various conditions are relevant to validate their performance, considering environmental factors and Brazilian construction standards.

### 1 INTRODUCTION

Corrosion risk in atmospherically exposed reinforced concrete structures is typically assessed through inspections and field or laboratory tests, focusing on key parameters such as the reinforcement's open circuit potential (OCP), visual examination, concrete cover thickness, electrical resistivity, pH levels, and aggressive agents like chlorides [1], [2]. Permanent monitoring systems can complement or even replace certain corrosion inspections. These systems, widely used abroad, incorporate embedded reference electrodes in concrete to monitor reinforcing steel or predict corrosion initiation.

True reference electrodes are essential in electrochemical analysis, as they typically provide a stable and reversible redox reaction, maintain a constant potential relative to the standard hydrogen reference electrode (SHE) regardless of the medium's conditions, and quickly recover their initial potential after accidental polarization [3], [4]. In concrete, true reference electrodes [5], [6] have to maintain a stable and reproducible potential while being suitable for long-term embedding in the concrete's aqueous medium. Although embeddable pseudo-reference electrodes lack some properties of true reference electrodes [7], they are widely used due to their durability, adaptability, and suitability for continuous rebar monitoring despite inherent precision limitations [8]. Recent advancements in electrode development aim to overcome these limitations [9].

Embedded electrodes should be planned during the conceptual design phase, considering the structural characteristics, environmental conditions, and corrosion risks. Consulting a corrosion specialist may be necessary, as no standardized procedure defines the minimum number of electrodes, their distribution, or the monitoring system layout. This also applies to measurement frequency and long-term analysis. For example, embedded electrodes can be installed in selected piles and blocks in pier and bridge structures exposed to marine environments, considering tidal variations, splash zones, and salt spray exposure. In slabs and beams, electrodes should be placed on the underside, typically covering sections closest to seawater.

Each electrode is typically paired with a companion rebar lead [7], preferably above the outermost reinforcement in representative structural elements. In existing structures, this is generally achieved by drilling through the concrete cover to insert the electrode and sealing the opening with a cementitious material. Once installed, monitoring can be conducted manually or through advanced systems with electronic data storage and remote transmission, enabling real-time data access and analysis.

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In the project's conceptual design phase, a network of electrode cables will be embedded in the concrete, with terminals directed to specific panels for manual OCP readings or automatic recording of its values. The locations of these panels are determined based on various parameters, mainly ease of access and proximity to a more significant number of elements to be monitored simultaneously. In existing structures, the electrodes are connected to the panels via a network fixed to the concrete surface. The remote monitoring system is necessary for hard-to-reach locations. In this system, each panel includes a voltmeter for periodic OCP readings of the reinforcement, which is unnecessary for manual measurements. In the latter case, the panel only contains connection terminals between the electrodes, reinforcement, and the voltmeter cables.

Despite the widespread use of embedded electrodes for early corrosion detection in concrete, their application in Brazil remains limited due to high import costs and limited knowledge. This highlights the importance of national research efforts, such as this study, which evaluates two imported electrode types—MnO<sub>2</sub>/NaOH and Ag/AgCl—alongside a locally produced metal-metal oxide (Ti/MMO) electrode as a potential alternative. This Ti/MMO electrode, made from inert anode mesh wire, has proven effective as a pseudo-reference electrode for monitoring corrosion and was recently introduced commercially [9].

#### 2 METHODS

For this study, three types of embedded reference electrodes (here referred to as internal electrodes) were selected. Two of these are imported models:

- MnO<sub>2</sub>/NaOH Electrode (EM): made of manganese dioxide (MnO<sub>2</sub>) in a 0.5 mol/L NaOH solution;

- Ag/AgCl Electrode (EPCP): made of silver-silver chloride (Ag/AgCl) in a 0.5 mol/L KCl solution.

The third internal electrode is a locally produced solid model called EMMO, prepared in the laboratory using a 3 mm titanium wire activated with mixed-metal oxide. The electrical connection was established at one end of the wire, while the opposite end was coated with a paint layer. The wire Ti/MMO was analyzed during the selection process, and the composition of the mixed-metal oxide layer was evaluated using EDS/SEM analysis. The results revealed a continuous oxide film with an average thickness of 6  $\mu$ m, primarily composed of iridium (53 %) and tantalum (27 %).

An initial assessment test was conducted based on the ABNT NBR 11105 [10] standard at a controlled temperature (23 °C  $\pm$  2 °C) to verify the relative potential of the selected electrodes with respect to the SHE. Four units of EM and EMMO electrodes and five units of the EPCP electrodes were used. The procedure consisted of:

- immersing the electrodes for 24 h in:

- saturated Ca(OH)2 solution for the EM and EMMO electrodes;
- 0.5 mol/L NaCl solution for the EPCP electrode.
- take three consecutive readings at 5-minute intervals using a high-impedance multimeter (> 10 MΩ).

In addition to the assessment test, cyclic potentiodynamic curves were obtained for one additional unit of each internal electrode type to assess their response to a small amplitude current. Traditional true reference electrodes (external electrodes) are minimally affected by small currents [4]. The curves were generated after 24 h of immersion in the same solutions mentioned, applying an over-voltage of  $\pm$  20 mV around the OCP, with a scan rate of 0.167 mV/s. The process started cathodically from the OCP, followed by anodic scanning after one minute. An external Ag|AgCl|KCl 3 mol/L reference electrode was used (210 mV vs SHE).

To assess the sensitivity of the electrodes to the concrete high alkalinity, pH reduction, and chloride presence, immersion tests were conducted at a room temperature of 23 °C  $\pm$  2 °C. The procedure consisted of the following steps:

- immersion in Ca(OH)<sub>2</sub> solution (pH 12.5): this test (160 days) simulates uncontaminated concrete;
- deaeration and aeration cycles in the same solution (as shown in Fig. 1, left): this experiment (25 days) involved continuous 24 h cycles of nitrogen purging (deaeration) and synthetic air purging (aeration) to evaluate electrode stability under different dissolved oxygen levels. These conditions mimic changes that can occur at the electrode-concrete interface due to varying oxygen availability;
- immersion in a new Ca(OH)<sub>2</sub> solution with pH adjusted to 9.5 using Na<sub>2</sub>CO<sub>3</sub>: this test (100 days) simulates carbonated concrete, a phenomenon that accelerates corrosion in reinforced concrete structures, particularly in large urban centers in Brazil;

- immersion in Ca(OH)<sub>2</sub> solution with 0.4 % NaCl: this test (90 days) simulates concrete contaminated with chlorides, a common condition for concrete exposed to marine environments. Such contamination leads to significant degradation of structures due to rebar corrosion.

A concrete specimen (CS) was prepared to monitor the electrochemical response of steel using an internal electrode and an external reference electrode (Ag|AgCl|KCl 3 mol/L). Four EM and EPCP units were installed beneath two duplicate steel bars, while four EMMO units were attached to the formwork. This study focuses on the results obtained from these bars, which had cover depths of 2 cm (B1/B1.1) and 3 cm (B3/B3.1), as illustrated in Fig. 1 (right). A reservoir was positioned over the CS to facilitate exposure, initially to a Ca(OH)<sub>2</sub> solution (up to Cycle 3) and subsequently to a 3 % NaCl solution. The wetting and drying cycles were conducted over 14 months, as specified by ASTM G109 [11].



Fig. 1 Ongoing test for some units of the EM and EPCP electrodes, with O<sub>2</sub> and N<sub>2</sub> purging stage (left), and the EM, EPCP, and EMMO (not visible in the figure) electrodes fixed in the rebar in the CS, before concrete casting (right).

# 3 RESULTS AND DISCUSSION

## 3.1 Electrodes' assessment tests

Table 1 displays the electrochemical potential differences of the internal reference electrodes, measured relative to the SHE and converted to the silver chloride external reference electrode (Ag|AgCl|KCl 3 mol/L). The table provides individual unit average values (AV) and overall average values (OAV) for each type of internal electrode.

Data analysis revealed that the standard deviation (SD) for the internal EM and EPCP electrodes was very low, consistently remaining within the acceptable range of  $\pm 5 \text{ mV}$  [10], which applies to true reference electrodes. In contrast, the EMMO electrode displayed a higher standard deviation of 17 mV. Although greater, this value is still acceptable for a pseudo-reference electrode, particularly in applications where high precision is not critical. The increased variability is attributed to the inherent challenges in maintaining a precise reference potential for pseudo-reference electrodes, which are inherently less stable than true reference electrodes [3], [7].

Electrode	EM (Ca(OH) <sub>2</sub> sat. solution)				EPCP (0.5 mol/L NaCl solution)				<b>EMMO</b> (Ca(OH) <sub>2</sub> sat. solution)				
Unit	12	13	14	15	1	3	7	8	9	1	2	3	4
Values (vs. SHE)	417	424	426	429	253	259	259	253	255	295	291	308	332
	418	428	427	431	254	260	260	255	258	296	291	310	333
	417	425	427	430	254	259	260	254	256	295	291	310	332
AV (vs. SHE)	417	426	426	430	254	260	256	254	256	295	291	309	333
OAV	425 (SHE)				257 (SHE)				<b>307</b> (SHE)				
	215 (Ag AgCl KCl 3 mol/L)				47 (Ag AgCl KCl 3 mol/L)				97 (Ag AgCl KCl 3 mol/L)				
SD	5				2				17				

Table 1 Reference potential (mV) of internal electrodes relative to SHE in the assessment test

AV = Average Value; OAV = Overall Average value; SD = Standard Deviation

# 3.2 Cyclic Potentiodynamic Curves

Fig. 2 presents the overpotential versus current curves obtained by applying  $\pm 20$  mV around the opencircuit potential (OCP). The results show distinct behaviors among the EM, EPCP, and EMMO electrodes. The EM and EPCP electrodes exhibited linear curves with minimal noise, demonstrating their ability to maintain stable and consistent performance. The potential shift and recovery were almost instantaneous, in line with the expected behavior of true reference electrodes, as described in the manufacturers' technical specifications.

On the other hand, the EMMO electrode exhibited hysteresis and significant noise in its overpotential versus current curve. This behavior indicates that this type of electrode does not meet the essential criteria for a true reference electrode, particularly its ability to withstand small currents with minimal polarization and to return to its original potential after the applied charge is removed [3], [7].



#### 3.3 Immersion Tests

Fig. 3 and 4 illustrate the internal electrodes' potential (OCP) monitoring during the immersion test. Table 2 presents the average values (AV) for each electrode unit and the overall average value (OAV) for each electrode type, calculated over the same period for all three electrode types. The table also includes the assessment test data from Table 1 for easy comparison. All potential values are referenced to the external Ag|AgCl|KCl 3 mol/L electrode.







Fig. 4 Potential evolution with time of the internal electrode (mV, vs. Ag|AgCl|KCl 3 mol/L) in the Ca(OH)<sub>2</sub> solution at pH adjusted to 9.5 (left) and with the addition of 0.4 % NaCl (right). The approximate period for calculating the average values (AV and OAV) is indicated.

Electro	Ca(OH) <sub>2</sub>		$\frac{Ca(OH)_2}{Ca(OH)_2} - \frac{N_2}{O_2}$		Ca(OH)2 - 0,4 % NaCl		Ca(OH)2- pH 9,5		Table 1	
Е	Unit	AV	OAV (SD)	AV	OAV (SD)	AV	OAV (SD)	AV	OAV (SD)	OAV (SD)
EM	1	215	214	218	217	230	229	337	340	215
	2	214	(5)	217	(3)	229	(5)	343	(3)	(5)
EPCP	2	25	21	14	10	21	26	78	56	47
	4	18	(5)	6	(6)	32	(14)	34	(23)	(2)
ЕММО	5	77		97		82		110		
	6	72	90	82	100	81	89	124	118	97
	7	104	(18)	113	(14)	90	(16)	117	(11)	(17)
	8	107		107		102		121	1	

Table 2Potential values (mV, Ag|AgCl|KCl 3 mol/L) for all immersion tests and the assessment<br/>test (Table 1)

In Fig. 3 (left), the two units of the EM and EPCP electrodes exhibited similar behavior from the start of the test. The EMMO electrodes initially showed more pronounced variation between units but stabilized after approximately 60 days of immersion. Each electrode type's average values (AV and OAV) potentials were calculated (Table 2). In the subsequent phase of the test (Fig. 3, right), the EM and EMMO electrodes maintained their previously observed behavior, with OAV values of 217 mV (compared to 214 mV in the previous period) and 100 mV (compared to 90 mV in the last period), respectively. For the EPCP electrode, the OAV decreased significantly to 10 mV (from 21 mV in the previous period).

However, considering the OAV of 10 mV alone, this variation is relatively minor in practical terms. It can be attributed to natural fluctuations in the system and environmental conditions during field OCP measurements. These results indicate that all three electrode types suit rebar monitoring in uncontaminated concrete, even under low dissolved oxygen availability in the pore water near the rebars.

When comparing these results (Fig. 3, right) with the assessment test (Table 1), the OAV for the EM (215 mV) and EMMO (97 mV) electrodes align closely with the immersion test values, particularly for EM, confirming their reliability for OCP monitoring in uncontaminated reinforced concrete. In contrast, the OAV for EPCP in the assessment test (47 mV) was more than double that observed in the immersion test, likely due to differences in the solutions used. It is believed that the NaCl solution in the immersion test more accurately represents the traditional application of this electrode type abroad, particularly in concrete cathodic protection systems [8], which are widely used in marine structures.

Fig. 4 presents the immersion test results for the internal electrodes after exposure to a  $Ca(OH)_2$  solution with pH adjusted to 9.5 (left) and a solution with pH 12.5 and 0,4 % NaCl (right). In both solutions, initial exposure caused variations in the OCP of the electrodes, particularly for the EMMO

AV = Average Value; OAV = Overall Average value; SD = Standard Deviation.

electrode. However, the potentials stabilized after approximately 60 days, and from this point onward, the average values (AV and OA) were calculated, as detailed in Table 2.

Analyzing the results for pH 9.5 (Fig. 4, left), as shown in Table 2, the average electrode values increased at this pH, exceeding those obtained in other solutions and the assessment test. The EMMO units exhibited the most consistent average values across different tests, with an OAV of 118 mV, closely matching the assessment test result (97 mV) and the Ca(OH)<sub>2</sub> solution at pH 12.5 (N<sub>2</sub>/O<sub>2</sub>).

For the EM and EPCP electrodes, a noticeable increase in values was observed, particularly for the EM electrode, which reached an OAV of 340 mV, compared to 215 mV in the assessment test and 214 mV in the pH 12.5 solution (Fig. 3). The EPCP electrode, in turn, recorded an OAV of 56 mV at pH 9.5, significantly higher than the 10 mV observed in the pH 12.5 solution with  $N_2/O_2$  (Fig. 3, right).

These results confirm that the potential of both the EM and EPCP electrodes is highly pH-dependent. Specifically, for the EM electrode, the potential shift was approximately 40 mV per pH unit, slightly deviating from the 30 mV per pH unit shift described in NACE Publication 11100 [7].

When analyzing the solution with pH 12.5 and 0.4% NaCl (Fig. 4, right), as shown in Table 2, the average electrode values increased slightly compared to other immersion tests. The lowest OAV values recorded were EM at 229 mV (compared to 214 mV in the Ca(OH)<sub>2</sub> pH 12.5 test), EPCP at 26 mV (compared to 10 mV in the Ca(OH)<sub>2</sub> pH 12.5 - N<sub>2</sub>/O<sub>2</sub> test), and EMMO at 89 mV (compared to 90 mV in the Ca(OH)<sub>2</sub> pH 12.5 test). Only the EPCP electrode exhibited a significantly different response than the assessment test (47 mV). Notably, the EPCP electrode's OAV closely approached the assessment test value only when immersed in the pH 9.5 (340 mV) deviating significantly from the assessment test value (215 mV).

Based on these findings, it is evident that the EM and EPCP electrodes require consideration of the specific environmental conditions in which they are immersed rather than relying solely on their assessment test values. This highlights the importance of verifying their reference potential against an external electrode when embedded in concrete for field applications. In contrast, the EMMO electrode exhibited stable potential values that remained broadly consistent with the assessment test results across all immersion tests. This behavior aligns with findings from reference [8], which reported that EMMO electrodes demonstrate stability in aqueous solutions with pH values ranging from 7.4 to 13.5. However, since the EMMO electrode functions as a pseudo-reference, it is still essential to validate its potential under field conditions, as it may experience variations over time due to changes in concrete properties, particularly in areas with high chloride contamination.

#### 3.4 Concrete specimens test

Fig. 5 presents the OCP (mV) monitoring results for selected rebars (B1/B1.1 and B3/B3.1, Fig. 1, right), with the embedded reference electrodes (EM, EPCP, EMMO) shown as dashed lines and the external electrode (Ag|AgCl|KCl 3 mol/L) represented by solid black lines. All OCP values from the internal electrodes were converted to the external electrode scale using two different overall average values (OAV) to facilitate comparison. One OAV, derived from the assessment test (Fig. 5, left, Table 1), and the other from the immersion test in a 0.4 % NaCl solution (Fig. 5, right, Table 2), which more accurately represents the concrete specimen's high pH and chloride exposure.

As shown in Fig. 5 (left), during the first five months of testing (up to Cycle 5), most electrodes — except for the internal EM electrode — exhibited PCA values close to or greater than -100 mV. After Cycle 7, the electrode values began to stabilize. Notably, the external electrode (Ag|AgCl|KCl 3 mol/L) showed a significant drop, aligning with values recorded by the EM electrode. This drop was attributed to a reduction in the electrical resistivity of the cover concrete, caused by prolonged exposure to an aqueous Ca(OH)<sub>2</sub> solution (up to Cycle 3) followed by exposure to a saline solution (3 % NaCl). The gradual increase in OCP over time remains unexplained regarding the EM electrode. However, this behavior reinforces the potential importance of allowing a stabilization period for the electrodes in the immersion medium.

Between Cycles 11 and 14, the general OCP values of the bars exhibited slight fluctuations for each type of electrode. By the end of Cycle 14, the range of PCA values for the bars, considering all electrodes, was approximately -50 mV to -130 mV. The external electrode (B3.1, -120 mV) and the internal EM electrode (EM15 - B3.1, -127 mV) recorded the most electronegative values, indicating that the bars were in an uncertain state, according to ASTM C897 criteria [13]. The values indicated a passive

state for the EPCP and EMMO electrodes, with the most negative value recorded for an EMMO electrode unit (-65 mV). Visual inspection of the bars at the end of the test confirmed that all bars were in a passive state.





Fig. 5 (right) shows the OCP values of the bars, with the conversion of the OAV obtained in a  $Ca(OH)_2$  solution containing 0.4% NaCl (Table 2). It can be observed that the range of values decreased to approximately -50 mV to -100 mV, bringing the OCP values closer when using different electrodes. This suggests that converting OCP measurements using the average immersion test values helps interpret the results more accurately.

As shown in the graphs (Fig. 5, left and right), all units of each type of internal electrode exhibited consistent behavior during the tests on the concrete specimen, aligning with the trends observed in the immersion tests. The maximum variations observed among the units were 21 mV for the EM electrode, 40 mV for the EPCP electrode, and 17 mV for the EMMO electrode. These results emphasize the stability and reproducibility of the units within each electrode type. This consistency is further supported by findings in the literature, which highlight the performance characteristics of these electrodes under various conditions. Reference [14] shows that the potential variation between units of the EM electrode remains below 30 mV in concrete tests. Reference [15] demonstrates the excellent long-term stability of the EMMO electrode exhibits good short-term stability, but after 250 days in mortar subjected to contamination, its potential readings became less reproducible. This potential drift is also noted in NACE Publication 11100 [7], which classifies this type of pseudo-electrode as effective in concrete but unsuitable for measuring absolute potentials.

In practice, monitoring OCP, whether in the field or laboratory tests, does not rely solely on analyzing absolute values to determine the onset of active corrosion. Instead, it focuses on tracking behavioral changes, particularly detecting significant drops in potential values—abrupt or gradual—as indicated by a threshold of  $\geq$ 150 mV [15]. Therefore, the absolute accuracy of values obtained using embeddable reference electrodes in concrete is not critical for effective corrosion monitoring.

#### 4 CONCLUSIONS

The EM and EPCP electrodes exhibited the most significant variations among the tested electrodes. The EM electrode showed a notable potential shift in a Ca(OH)<sub>2</sub> solution at pH 9.5, simulating carbonated concrete. In contrast, the EPCP electrode displayed significant differences in a solution at pH 12.5 with 0.4 % NaCl, simulating chloride-contaminated concrete. In contrast, the EMMO electrode demonstrated stable behavior, with no significant deviations in its potential values, including those obtained from assessment tests.

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The results indicate that, although internal electrodes tend to stabilize over time and are suitable for corrosion monitoring, their reference potential can shift depending on the conditions of the aqueous medium. Therefore, selecting the appropriate electrode type for each application and monitoring its behavior is required, mainly when significant changes in concrete conditions or electrode readings are observed. In such cases, the embedded half-cell potential can be compared with the values obtained using a conventional true reference electrode placed on the concrete surface. This approach ensures more accurate potential measurements in the long term. It enhances the practice of detecting the onset of corrosion by monitoring gradual or abrupt potential drops in atmospherically exposed reinforced concrete structures.

Future research should focus on evaluating the durability of electrodes in the field, considering Brazilian environmental factors that may influence their reference potential, and verifying their role in preventive and corrective maintenance strategies for reinforced concrete structures.

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