



Review

Long-Term Marine Corrosion Under the Influence of Microbiologically Influenced Corrosion and Calcareous Conditions

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Abstract

Calcareous deposits on and within corrosion products tend to inhibit the (abiotic) corrosion of steels in seawater. Herein, it was considered whether this inhibition effect extends to microbiologically influenced corrosion (MIC) for extended (long-term) exposure periods. Quantitative estimates of corrosion rates were made from reported observations for 46 iron and steel shipwrecks, and other iron and steel objects immersed in seawater at various depths and for extended periods (many around 60 years and some up to 160 years). The observations are correlated with observations of the occurrence of calcareous deposits and information about dissolved inorganic nitrogen (DIN), a critical micronutrient for MIC. The results show that calcareous deposits can inhibit both long-term abiotic corrosion and long-term corrosion influenced by conditions suitable for MIC. The practical implications are briefly reviewed.

Keywords: corrosion; calcareous deposits; MIC; DIN; inhibition; long-term



Academic Editors: Philippe Refait,
Scott Wade and Ladislav Vrsalović

Received: 23 July 2025

Revised: 13 August 2025

Accepted: 20 September 2025

Published: 25 September 2025

Citation: Melchers, R.E. Long-Term Marine Corrosion Under the Influence of Microbiologically Influenced Corrosion and Calcareous Conditions. *Corros. Mater. Degrad.* **2025**, *6*, 46. <https://doi.org/10.3390/cmd6040046>

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1. Introduction

It has long been recognized that calcareous materials such as calcium carbonate and magnesium carbonate within marine rust products can have the beneficial effect of reducing the rate of corrosion of steels and irons [1,2]. This effect has mostly been described for shorter-term observations. The effect very much depends on the (near) saturated concentrations of carbonates in natural seawater and reduction in oxygen permeability afforded by the presence of carbonates in and on rust layers [3]. Calcareous deposition has also been associated with impressed current cathodic protection applied to reduce the seawater immersion corrosion of steel piles [4]. In such a case, the effectiveness of calcareous deposition formation is demonstrated by the inhibition effect continuing for some 3 months after the cathodic polarization current that drives the polarization potential, usually considered the primary factor in cathodic protection, has been turned off. It was only after the visible calcareous deposit had (slowly) disintegrated that corrosion was observed. Calcareous deposition has also been associated with sacrificial anode cathodic protection, seen as an external ‘coating’ over paint coatings [5]. An example is shown in Figure 1 for the exterior of a steel pipeline after 9 years in 10 °C offshore natural seawater.

These observations and the mechanisms involved [3,6,7] were all relatively short-term observations and did not involve microbiologically influenced corrosion (MIC). Similarly, a study on whether impressed current cathodic protection could affect biofilm formation in the process of also producing calcareous deposits considered only short-term effects (200 h) [8].



Figure 1. Calcareous deposition on exterior protective coating of a steel pipeline (200 mm (nom.) diam.) with sacrificial anode-protection after 8 years continuous exposure in 10 °C offshore natural seawater (copyright R.E. Melchers).

The present paper has its origins in underwater scuba diving observations that steel shipwrecks in the relatively polluted coastal zone of the Belgian North Sea (BNS) showed little corrosion loss and pitting despite having been exposed in some cases for up to 100 years [9]. This is remarkable since in the high nutrient content of those coastal waters [10], significant microbiologically influenced corrosion (MIC) would be expected, particularly for longer-term exposures [11]. Some samples of rusts and steels were extracted from nearby similar-aged steels, and the microbiological (wet reaction) testing of rusts showed little or no evidence of the microorganisms typically associated with MIC [9]. This strongly suggests that for the wrecks in the same vicinity, MIC was not a significant contributor to corrosion. Also observed was a higher-than-expected presence of calcareous material on and within the rusts of the samples [9]. The diving inspections suggested that this was also the case for the wrecks themselves. These various observations led to the proposition that the low incidence of corrosion loss and pitting, and the low incidence of microorganisms (and hence MIC) was likely the result of the presence of calcareous material (calcium carbonate, magnesium carbonate, ferrous carbonate) on and within the rusts [9]. This proposition is supported by observations at a considerable number of other sites where both calcareous deposition within and on the exterior rusts, and low corrosion losses and low corrosion rates were noted for long-term exposures [12]. It suggests that the presence of calcareous deposits within and on rusts inhibits not only electrochemical (abiotic) corrosion, but also MIC. In this context, it is helpful to note that while MIC is often attributed a status of its own, it must obey fundamental electrochemical/chemical principles, including the Gibbs free energy requirement for the reactions to be possible and to continue to be possible as corrosion and corrosion products (rusts) develop. Both MIC and electrochemical (abiotic) corrosion produce similar corrosion products predominantly of the FeOOH type, irrespective of whether the cathodic reaction is oxygen reduction or hydrogen evolution [13,14]. Of course, MIC is also associated with other, secondary, rust products [15].

The present paper considers whether the above proposition can be supported, including for extended (long-term) exposure periods. It uses quantitative estimates of corrosion rates based on (subjective) observations for 46 iron and steel shipwrecks, and other iron and steel objects immersed in seawater at various depths and for extended periods (many around 60 years and some up to 160 years). The focus is on longer-term observations since for infrastructure applications, these are the most relevant [16]. The next section (Data) describes the use of open-source underwater videos, and still images of steel and iron shipwrecks and other ferrous objects immersed for extended periods to make estimates of the amount of corrosion loss to estimate the long-term rates of corrosion. Some of the estimates are quantitative while others are qualitative owing to the limited available information as

limited by constraints of access (particularly for deep diving), limits of underwater rust removal, and limits of underwater metal surface examination at depth, all within practical economic constraints. Nevertheless, as will become evident, clear distinctions can be made between the rates of longer-term corrosion loss for the different exposure environments.

2. Data

As noted, in total, 46 shipwrecks and other objects immersed in seawater were available for assessment, as shown in the Supplementary Materials. They are mostly for 60–70-year exposures, and in one case (case 25), for an estimated exposure of 347 years. The dataset covers a range of immersion depths. It includes cases exposed to seawaters away from coastal or other potential sources of water pollution, or from natural high-nutrient sources (e.g., ocean vents). It also includes cases with elevated concentrations of the nutrients critical for microbiological metabolism and, by implication, the microbiologically influenced corrosion (MIC) of steels [11,17]. These are principally dissolved inorganic nitrogen (DIN), i.e., nitrates, nitrites and ammonia, and phosphorous (P) [11].

Numerical values for the annual average concentration of dissolved inorganic nitrogen (DIN) and phosphorous (P) were available only for some sites. Such environmental data and climatic data have seldom been seen as central for previous investigations of shipwrecks, given the expense of obtaining it over extended periods. Fortunately, for some locations, such as those in the North Sea and the Gulf of Mexico, some information was available, albeit area-wide. Also, because corrosion in natural conditions tends to be a slow process, it is sufficient for the present analysis to consider only annual average environmental conditions, similar to what has been done in the past [11,18,19].

Long-term corrosion rates were derived from the available information for corrosion losses and are shown in the Supplementary Materials. Specifically, for the extended exposure periods of interest here, the usual bi-modal corrosion behavior (Figure 2) [11,13,18,20] may be simplified to a linear model, applicable for longer exposures. The latter is consistent with empirical data from multiple sources [21,22], including where calcareous deposition is known to be involved [9]. The simplified model may be parameterized using the intercept c_s and the long-term rate r_s (Figure 2). The values for these parameters are shown in columns (9) and (10) in the Supplementary Materials. For completeness, Figure 2 also shows, schematically, the effects of increasing seawater temperature and average DIN concentration [11].

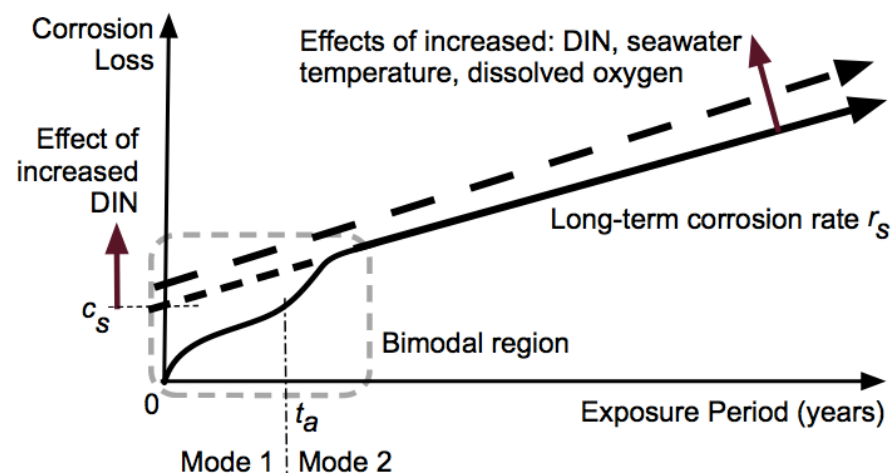


Figure 2. Longer-term bimodal corrosion trending, showing the parameters c_s and r_s that define the linearized model. The effects of increased DIN, increased mean seawater temperature, and increased dissolved oxygen are shown schematically with the broken line [20].

Unless otherwise noted, the data in the Supplementary Materials are for field observations where these were available, not laboratory experiments. Because field conditions are not easily controlled, the observations are subject to a certain amount of, but not easily quantifiable degree of, uncertainty. This is not unusual and, as will be seen, by having available a wide range of data in sufficient numbers and an understanding of the processes involved, it becomes possible to make analyses of the patterns in the data and derive robust observations and conclusions. In brief, these are that the available evidence points to calcareous deposition, from whatever source, inhibits MIC effects as well as abiotic corrosion, and does so remarkably well with, in many cases, little or no general or pitting corrosion in evidence even after extensive periods of exposure.

The data in the Supplementary Materials cover observations in natural seawater, typically well away from coasts or offshore facilities. It also covers observations in natural seawater for which DIN concentrations greater than expected background levels were observed or could be reasonably inferred from the available information. This includes cases where the site is likely subject to exposure to fertilizer run-off, for example, conveyed by river waters discharging into the relevant seawaters, or where discharges or leaks of oil or other chemicals were observed. There are also observations in natural seawater for which calcareous conditions were observed (e.g., [9]), and, further, cases with both calcareous conditions and evidence of elevated DIN conditions. The sources of the information are as noted.

The data and corrosion observations in the Supplementary Materials are for shipwrecks and steel piling. Exceptions include the 100-year old steel mooring chains (case 2), coupons taken from steel sheet-piling in harbors (cases 15–20), 160-year-old cast iron cannon balls (case 24) and 347-year old cast iron cannons (case 25). For the shipwrecks, those at very deep locations were investigated (by others) with the aid of remotely operated vehicles (ROVs). Most of the available reports predominantly deal with archaeological or historical war-related aspects, although most provide underwater photographs. This is also the case for the more accessible shallower wrecks, but information about underwater condition tends to be limited.

The cases in the Supplementary Materials are confined to those for which there is (a) adequate historical information to make reasonable estimates of the duration and of the subsea conditions of exposure, (b) information sufficient for estimating the prevalent DIN concentration at the site and/or calcareous deposition on the steel objects (and if possible their variation throughout the year), and (c) underwater videos or photographs sufficiently detailed to assess the occurrence and, if possible, the severity of corrosion. The cases shown in the Supplementary Materials include those in earlier reports [9,11,12,23,24]. The others were selected opportunistically. Many additional cases exist, particularly shallow wrecks, but for these, there is mostly a lack of sufficiently detailed environmental and water quality information.

3. Data Analysis

The observations were divided into two groups: those in which there is no visual or other evidence of calcareous deposition and those for which there is such evidence. It should be noted that repeated underwater observations of the wrecks showed the former very clearly, with displays of bright red-brown rusts (e.g., [23]) under the illumination of diving lights compared to dull reflections and ‘woolly’ surface topography, either of the calcareous layer itself or rusts with embodied calcareous material. Each category was subdivided into cases for which there is evidence that the waters at the wreck site had been subject to elevated concentrations of DIN and those for which there was no such evidence. This left the four groups shown in the Supplementary Materials.

In the Supplementary Materials, column 1 denotes the case number and reference information, column 2 the name and location of the vessel (or object), column 3 the depth D , and columns 4 and 5 the exposure time period t and the effective exposure time period t_{eff} . The latter makes allowance for the likely period of loss of paint coating due to deterioration. Column 6 provides very brief comments about observations of any visible rusts and any calcareous deposition. These comments are based on examination of the available videos and photographs. Column 7 shows the average concentration of DIN at the site measured or estimated from the source material. Similarly, column 8 provides the best estimate of the annual average seawater temperature T at the site. Column 9 shows the corrosion loss c_T at the site under temperature T . The average corrosion rate obtained as c_T/t_{eff} is shown in column 10. Columns 11 and 12 show the corrosion losses and rates corrected to average water temperature $T = 12.5\text{ }^{\circ}\text{C}$, the latter chosen for consistency with earlier results for the southern North Sea [9]. The correction factors for other mean seawater temperatures are shown at the foot of Table S1 in the Supplementary Materials.

Earlier studies have shown a linear correlation between moderately elevated concentrations of DIN and corrosion losses principally in Mode 2 (Figure 2) [11,25]. This has been attributed to increased concentrations of DIN raising the metabolism of microorganisms, including the sulphate reducing bacteria (SRB). In turn, this has been considered responsible for increasing the severity of corrosion, both pitting and more generally [12]. Examples include the severe longer-term corrosion, attributed to MIC, in seawaters with high levels of nutrient pollution [26]. Consistent with these and other observations, SRB-induced MIC is, herein, considered critical for the extended exposure of steel in seawater conditions, recognizing that there is evidence for other drivers for, and forms of, MIC [27,28].

Using the data in the Supplementary Materials, Table 1 distinguishes the sites with the effects of calcareous deposition and those with seawater DIN concentration. Table 2 uses this distinction to provide the relevant numerical estimates of r_s where possible. These results are summarized in Figure 3.

Table 1. Qualitative comparison of corrosion estimated from the severity of corrosion products (rusts) categorized by occurrence of calcareous deposition and evidence for elevated DIN concentration (based on data in the Supplementary Materials).

1	Calcareous deposition	No	No	Yes	Yes
2	Seawater nutrient quality	Natural	Elevated DIN	Natural	Elevated DIN
3	Case numbers (total number)	1–13 (13)	14–22 (9)	23–35 (13)	36–46 (11)
4	Inferred corrosion severity (from observed rust)	Typical	Heavy	Little or none	Some

Table 2. Quantitative estimates of long-term corrosion rates r_s , categorized by occurrence of calcareous deposition and evidence for elevated DIN concentration (based on data in the Supplementary Materials).

1	Calcareous deposition	No	No	Yes	Yes
2	Seawater nutrient quality	Natural	Elevated DIN	Natural	Elevated DIN
5	Case numbers for estimates of r_s	1–3	14–20	23–25	36–37
6	Number of individual data sets for r_s	>25	>8	>3	>11
7	Approx. rate r_s (mm/y) @ 12.5 °C	0.042–0.05	0.06–0.14	0.016–0.02	0.016–0.03
8	Approx. mean r_s (mm/y) @ 12.5 °C	0.049	0.077	0.016	0.022
9	Approx. standard deviation (mm/y)	0.0035	0.010	0.0017	0.0062

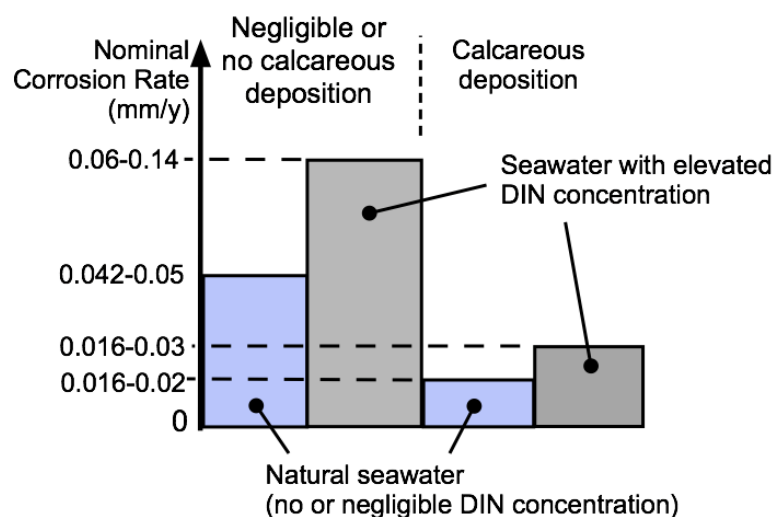


Figure 3. Schematic comparison of nominal longer-term marine immersion corrosion rates r_s and the effect of calcareous deposition, and the presence of elevated concentrations of dissolved inorganic nitrogen (DIN), based on Table 2.

The numbers of individual data in Table 2 (line 6) were extracted from the categories in the Supplementary Materials. While those numbers vary considerably between the four exposure situations, Figure 3 shows that the long-term corrosion rates r_s are very different between natural seawaters with little or no calcareous deposition, and those for which calcareous deposition was observed, directly or indirectly. The ratio ‘calcareous’ to ‘natural’ is about 1:3–1:4, irrespective of the concentration of DIN. Similarly, there is a difference in r_s between the different DIN environments. In this case, the ratio of the corrosion rate between no or negligible DIN and elevated DIN is about 3:8, irrespective of whether calcareous deposition was involved. These ratios and associated interpretations do not change very much if the mean and standard deviation values are used to plot a diagram similar to Figure 3.

For seawaters with little or no calcareous deposition, the relative values of the long-term corrosion rate r_s with and without the influence of MIC (Figure 3) are consistent with earlier results [11,26] which showed that elevated concentrations of DIN in the seawater increase corrosion losses. Importantly, Figure 3 also shows that calcareous deposition reduces the rate of long-term corrosion, and that it also does so under elevated DIN concentrations, with a proportionally similar effect. In short, in seawaters calcareous deposition inhibits MIC as well as abiotic corrosion (i.e., without involvement of MIC).

4. Discussion

The data in the Supplementary Materials were drawn, as noted, largely from field exposures over extended periods of time. They are not, therefore, results from controlled experiments such as laboratory tests with controlled nutrient seeding, steady water temperature, zero or low steady water velocities, etc., and are supplied with quite specific bacteria or other microorganisms. Many such results are available in the literature, mostly for short-term exposures (e.g., 6 months) [29]. Experimentation under controlled conditions over much longer periods of time (e.g., 20 or more years) might be desirable in order to obtain data that would be directly relevant for infrastructure applications; however, it is unlikely that such extended exposure tests would be feasible, organizationally or economically. One alternative is to examine the condition of existing, older steel structures such as shipwrecks that have already been subjected to extended periods of exposure, and then use these to make estimates of corrosion rates coupled with, and in parallel with, obtaining

relevant environmental data. In this approach the 'experimental' environment at any particular (wreck) site was outside the control of the observers. However, with a sufficiently wide set of data from a variety of sources, it is possible to make inferences and to draw conclusions with a reasonable degree of confidence. Such a practical approach, as also used here, has a long history [19,30].

Many of the cases in the Supplementary Materials are shipwrecks located near the mouths of rivers. At these locations, the quality of the water may depart considerably from that of most natural seawaters. One example is the Mississippi River. It discharges hard fresh river waters, enriched with multiple nutrients derived from upstream fertilizer runoff into the near fully carbonate-saturated coastal waters of the Gulf of Mexico [31]. Water quality may also be affected by the presence of limestone-rich coastal zones with submarine groundwater discharge (SGD) from coastal land zones discharging into DIN-enriched coastal seawaters [32]. In open sea conditions, well away from coastal sources of pollution, the quality of waters can be expected to be consistent [33] but may be subject to some degree of uncertainty. Some observations indicate that the concentration of DIN, for example, is generally negligible in open seawaters away from anthropogenic pollution [33] and that DIN concentrations tend to decrease rapidly with depth [34]. This appears to be reflected in observations that the seawaters at the depth of the *Titanic* (case 3) have been considered 'nutrient-poor' [35].

Regarding calcareous deposition, the data in the Supplementary Materials indicate that, overall, shipwrecks located at a considerable depth show little evidence of calcareous deposition. This appears to be consistent with observations that the concentration of CaCO_3 in seawater tends to decrease with depth ([36], pp. 455–457), even though the rates of such decreases vary considerably between and within ocean basins. From a practical viewpoint, these observations are important because the presence of calcareous deposition has been associated with inhibition of immersion corrosion [4,9]. In turn, this is considered the result of the development of relatively impermeable ferrous carbonate layers (FeCO_3) within the rusts [3,37]. In terms of the (bimodal) model for corrosion (Figure 2), the main effect is the inhibition of oxygen diffusion inward from the external environment causing reduced corrosion as corrosion in mode 1 progresses [38]. The reduced corrosion effect carries over to longer-term corrosion in mode 2 after allowing for the transition between the modes [13,14]. In addition, in mode 2, the calcareous material will inhibit the outward diffusion of hydrogen that results from the cathodic hydrogen evolution reaction that controls the (relatively slow) rate of corrosion in mode 2 [12,14].

In addition to the above, carbonate deposition may have a role in inhibiting the diffusion of nutrients necessary for MIC, such as the components of DIN (ammonia, nitrite and nitrate), from the external environment. The quantitative degree to which this is the rate controlling mechanism remains to be investigated. However, there are at least three other mechanisms by which calcareous material may exert control over the rate of MIC [39], all being the direct result of the elevated pH induced by calcareous deposition. Specifically, an elevated pH may reduce microbiological growth and survival, reduce the rate of microbial metabolism from the usual optimum at around pH 3–4, and may interfere with the kinetics of cathodic reactions, particularly the hydrogen evolution reaction. Again, the quantitative effect of each of these on the degree of MIC remains to be investigated.

The inhibition of corrosion in seawaters with elevated DIN through the formation of calcareous deposits may have implications for cathodic protection of steels. Conventionally the protective effect of cathodic protection is attributed to it reducing the electrochemical potential sufficiently to inhibit corrosion [40]. However, the reduction in potential, if sufficient, also causes an increase in local pH, and thus favors the formation of a protective passive film [7], seen in seawater as calcareous deposition (Figure 1). For impressed current

cathodic protection, this has been shown to remain protective for many weeks after the current had been turned off, during which time the calcareous deposits slowly became physically damaged by wave action [4]. The usual criterion for corrosion inhibition in practice is to specify a potential of (approx.) -850 mV (SHE), but where MIC is likely to be involved, the recommended potential is around -950 mV (SHE), although there is considerable controversy whether such extra potential is warranted [41,42]. It involves considerable extra cost and, in any case, would be necessary only in the early stages of cathodic protection. Many investigations of impressed current CP effectiveness have used short-term testing or electrochemical tests [43,44] that focused mainly on corrosion initiation and biofilm formation in the presence of pre-selected microorganisms such as SRB [45]. In practice, however, long-term behavior is of most interest. Evidently, there is room for further investigation considering the role of calcareous deposition in the longer-term effectiveness of cathodic protection.

5. Conclusions

The following conclusion are drawn:

1. The rate of the long-term corrosion of steel in natural seawaters is inhibited in the presence of calcareous deposits on or within corrosion products, irrespective of the presence or otherwise of nutrients critical for microbial metabolism associated with microbiologically influenced corrosion.
2. No experimental evidence was found that the reduction in corrosion loss by calcareous deposition is affected by environmental conditions such as depth of immersion, dissolved oxygen concentration and average seawater temperature. However, as previously established, these parameters do influence the rate of longer-term corrosion, including under microbiologically influenced corrosion conditions.
3. The relative inhibiting effect of calcareous material, measured by the ratio of maximum corrosion rates under calcareous and non-calcareous conditions, is similar for the corrosion of steel in natural seawaters and steel in seawaters with elevated nutrient conditions.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/cmd6040046/s1>, Table S1: Field data and estimated long-term exposure rates for steels in various waters. [46–113] were cited in the Supplementary Materials.

Funding: This research was funded in part by the Australian Research Council, grant number DP210102073.

Data Availability Statement: No new data were created or analyzed in this study. Data sharing is not applicable to this article.

Acknowledgments: The work reported herein is part of the outcomes of a Discovery Project (DP210102073) supported by the Australian Research Council.

Conflicts of Interest: The author declares no conflict of interest.

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