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Rust removal from steel coupons after short term marine immersion

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Abstract

The quantification of mass loss, surface topography, depth of pitting and localized corrosion for steels subject to marine corrosion requires the removal of rusts, preferably without causing additional mass loss, pitting or other damage. Conventional procedures using inhibited hydrochloric acid or Clarke's solution are shown to remove short-term marine rusts but also to cause new corrosion including pitting and related rust products. Both increase with longer exposure to the cleaning solutions. Water washing with soft water left calcareous materials on the metal surface. Ultrasonic removal did not cause additional corrosion but was effective only for very light rusts. Although relatively slow, electrolytic cleaning (electrolysis) produced clean surfaces without new corrosion.

Keywords: Steel, Coupons, Cleaning, Mass loss, Pitting.

1. Introduction

This paper is concerned with the effectiveness of procedures to remove rusts from steel coupons exposed in seawater for relatively short periods of time (days-weeks) so as to allow estimation of the mass loss and also the quantification of localized corrosion such as pitting and surface topography changes. In an earlier experiment [1] involving much longer exposures (0.3-2.6 years) use was made of conventional coupon cleaning procedures. These include mechanical, chemical and electrolytic cleaning [2]. Mechanical cleaning is very suitable for removal of easily removable (outer) rusts but is unsuitable for rusts adherent to the metal because of the risk of damage to the metal. Chemical cleaning at normal room temperatures typically involves inhibited hydrochloric acid (HCl), either as the well-known Clarke's solution or inhibited with hexamethylene tetramine [2]. Both are toxic and require suitably safe conditions for application. The more toxic and dangerous procedures [2] using sodium hydroxide at 80-90°C are not considered herein. Electrolytic cleaning is a safe but slow procedure that uses an electric current to create conditions that causes the rusts to lift off the steel. Washing in clean, preferably soft, water either as a gentle wash or under a water jet may be used to remove rusts that are relatively weak. In addition, ultrasonic cleaning has been used to remove light rusts, particularly for non-ferrous corrosion products [3].

Protocols for the mechanical, chemical and electrolytic procedures have been developed over many years [4] and, if properly applied, are considered to produce coupon surfaces essentially free from the original corrosion products. For the chemical procedures there is a known risk of some loss of the uncorroded steel. Usually such losses are small relative to the corrosion losses from field exposures and often low enough to be ignored in practice. Such losses can, however, be estimated by subjecting blank, uncorroded coupons to identical cleaning procedures [2]. These procedures have a long history and have been used extensively for experiments to estimate corrosion loss, for immersion, tidal, atmospheric zones, invariably using coupons to determine mass loss. However, apart from some interest in pit depth [5] the topography of the corroded surfaces has not received much attention.

For extended exposure periods the mass losses, the changes in topography and the volume of rust products tend to be relatively large and short periods of cleaning using inhibited HCl solution have no noticeable effect on the mass losses of the coupons and on the topography of the corroded surfaces [1]. However, this is unlikely to be the case for shorter-term exposures. For these, the amount of rust generated, the corrosion losses and the depth of pitting will be small and, as shown in a preliminary study [6], the process used to remove rusts may have an influence on surface topography, unless great care is taken or non-aggressive cleaning techniques are used.

The motivation underlying the present study is to consider the effect of biofilms and MIC on short-term corrosion (days, weeks), measured by mass loss and by the severity of localized corrosion and pitting. So far these matters have been studied mainly in laboratory experiments using culture media [7]. The development of corrosion and pitting with increased exposure time has been considered recently but still in culture media [8]. Little attention has been given to the influence of natural exposure conditions. This includes the influence of the nutrient content in seawater on mass loss and pitting, considered mainly in laboratory settings [9] for short term exposures but more recently considered for much longer exposure periods under field conditions [1, 10].

For short exposure periods (0-100 days or so) the corrosion mass losses and the depths of pitting are quite small and the marine rusts are not voluminous and may be weak structurally compared to rusts from extended exposures. Removal of such rusts is not covered specifically by conventional procedures. The present paper is concerned with ascertaining the effectiveness and the imitations of the various techniques for removal of rusts from coupon surfaces. The next section outlines the materials used and the methods employed for coupon cleaning and for subsequent examination of the corroded surfaces. This is followed by a description of typical observations and a discussion of the results and the implications of the test results.

2. Materials and methods

Mild steel coupons 25 mm by 25mm and 1.4mm in thickness (composition % by weight: 0.08 C; 0.21 Mn; 0.017 S; <0.001 P; 0.03 Ni; 0.059 Al; 0.03 Cu; 0.02 Sn; <0.001 Nb; <0.001 Cr; <0.001 Mo; <0.001 Ti) were used throughout. Prior to the coupons being laser-cut from the parent plate, each of the two surfaces of the plate were mirror polished uniformly over the surface, using a series of fine silicon-carbide abrasives, finishing with a 1 micron polishing paste, then washed in triply distilled water and dried with methanol (methyl alcohol) and then air-dried. After laser cutting the coupons were again washed and dried and then stored in sterile desiccators until required. The mass of all coupons was determined immediately before exposure. Careful examination showed that there was no corrosion product on the surfaces of the coupons at the time of exposure.

Apart from those coupons set aside for blank tests, all coupons were exposed fully immersed at a depth of 50-100mm in fresh, slowly flowing natural seawater in the natural seawater exposure tank at the corrosion laboratory at Taylor's Beach, NSW, Australia. These coupons were suspended using plastic CTMP1000 Smart Tube UV resistant cable ties and coded for identification. The seawater chemistry (Table 1), velocity and temperature conditions and the general environment have been documented previously [11].

Parameter	Units	Typical value	Parameter	Units	Typical value
Alkalinity(CaCO ₃)	ppm	409 - 419	Nitrite	ppm (wt)	< 0.003 - 0.011
Ammonia	ppm	0.017 - 0.090	pН		8.1 - 8.3
Calcium	ppm	374 - 409	Salinity	ppt (wt)	25.7 - 31.8
Chloride	ppm	21,000	Sulphate	ppm (wt)	1600 - 2750
Diss. Oxygen	%	90	Total Phosphorus	ppm (wt)	0.003 - 0.07
Nitrate	ppm	0.017 - 0.050	Water Temp.(annual mean)	°C	20

Table 1. Typical seawater chemistry at Taylor's Beach Research facility.

At each of 1, 3 and 4 weeks after immersion, a set of exposed coupons was recovered and gross marine growth and major rust components removed using brisk brushing. Immediately thereafter each coupon was placed in an individually labelled sterilized plastic container completely filled with the natural seawater from the exposure tank and sealed in such a way that air was excluded. All coupons were then transported to the laboratory (about 1 hour). In the laboratory, the four cleaning methods summarized in Table 2 were applied to duplicate coupons. They were labelled for unique subsequent identification by cleaning code as A1, A2 (inhibited acid); C1, C2 (Clarke's solution); T1, T2 (tap water); E1, E2 (electrolysis) and U (ultrasonics) corresponding to the intended exposure periods shown in Table 2. For the inhibited HCl solution (Table 2) laboratory grade HCl was used together with a commercial inhibitor (Hexamethylene Tetramine 99% – $(CH_2)_6H_4$ Chem-Supply / Australian Scientific). Clarke's solution was obtained from a commercial supplier

(ACI Labscan / Australian Scientific). The electrolysis was performed in tap water modulated with sodium carbonate, sodium hydroxide and sodium sulphate (quantitative (details in Table 2). The coupon to be cleaned was connected to the negative terminal of a 12 volt DC power supply via a potentiostat and current meter and using a piece of SS304 stainless steel as the anode, following conventional procedures [12]. For cleaning with water, potable Newcastle tap water (a 'soft' water) was used (Table 3). Immediately following the cleaning process, each coupon was rinsed thoroughly with deionised water, then immediately dried, weighed and placed in sterile containers and sealed. The coupons were then taken for scanning electron microscope (SEM) examination.

Procedure	Coupon	Time	Method / Solution	Observations		
Inhibited	A1	10 min	500 ml hydrochloric acid, sp gr 1.19	Doughnut shaped features (Fig 2).		
acid*	A2	30min	3.5 g hexamethylene tetramine Reagent water to make 1000 ml	Higher density of doughnut shaped features over most of surface.		
Clarke's	C1	10 min	1000 mL hydrochloric acid, sp gr 1.19	Similar to above but greater density.		
solution*	C2	30 min	50 g of Stannous Chloride (SnCl ₂)	Similar to above.		
Electrolysis*.	E1	1 hour	200 A/m ² cathodic treatment 75 g of Sodium Hydroxide (NaOH) 25 g of Sodium Sulphate (Na ₂ SO ₄)	Most of the metal surface revealed, with some shell-like features remaining on surface (Fig. 3).		
	E2	2 hours	75 g of Sodium Carbonate (Na ₂ CO ₃) 1000 mL of reagent water	As E1 but no shell-like features.		
Ton Watan	T1	1 min	Held under a gentle stream of tap water	As E1 but more shell-like features (Fig. 4).		
Tap water	T2	1 min	Held under a strong jet of tap water	Similar to T1.		
Ultrasonic	U	12 hours**	Branson 40 kHz Sonogen Automatic Cleaner Immersed in Tap water at room pressure and temperature	Extended exposure required to remove rust products.		
Combined	C1+U	10+15min	Immersion in Clark's solution followed	Removal of all surface features.		
Combined	C2+U	30+15min	by ultrasonic cleaning.	As above.		

Table 2. Summary of the methods and tests used for removal of 4 weeks-old marine rust products and observations of effect.

* Based on ASTM G1-03 [2011].

** Time required to remove all rust products.

Table 3. Water chemistry of tap water used for cleaning coupons with 4 week-old marine rusts.

Ammonia	Chloride	O_2	Nitrate	Nitrite	pН	Salinity	Sulphate	Sulphite	Р	Hardness
mg/L	mg/L	%	mg/L	mg/L	-	-	mg/L	mg/L	mg/L	mg/L CaCO ₃
0.017	34	96.3	0.013	0.002	7.18	0.1	27	< 0.05	0.009	50-100

Water hardness provided by Hunter Water Corporation.

Several control tests were carried out on unexposed polished coupons, that is coupons that had no marine corrosion products on their surfaces. In one series of tests inhibited HCl was applied to several unexposed coupons for 2, 5 and 10 minutes but otherwise subjected to the same protocol, including SEM examination. The severity of corrosion was then assessed by examination of the amount and appearance of corrosion

product on the metal surface as a function of exposure period. In another series of tests, unexposed coupons were subjected to Clarke's solution for 10 and for 30 minutes using the same protocol as for the corroded coupons including examination by SEM but immediately afterwards they were subjected to ultrasonic treatment and thereafter they were again examined using SEM. The relevant protocol otherwise is identical to that shown as Combined in Table 2. For the ultrasonic treatment coupons were immersed in tap water at room pressure and temperature in a Branson 40 kHz SONOGEN Automatic Cleaner. Also, control tests were carried out on unexposed coupons to assess the effect, if any, of the electrolytic process and, separately, of ultrasonics on the condition of the metal surface. No corrosion was expected but the tests were necessary to ascertain whether this expectation was correct.

3. Results

All the coupons were well-covered with rusts after their seawater immersion exposures irrespective of the period of exposure (1, 3 or 4 weeks). Before these were subject to the cleaning protocols A, C, E and W (Table 2) they all had very similar exterior appearance being well-covered with marine rusts, although more so for those exposed for 4 weeks. For this reason only the results for coupons exposed to seawater for 4 weeks are described in the following. Also, in most cases the coupons showed similar but atypical corrosion patterns in a narrow strip (approx. 1 mm wide) at the edges of the faces of the coupons. These regions have been noted before [13,14] but are ignored in the following.

SEM images representative of the surface topography of the coupons are shown in Figs. 1-4 for each of the cleaning procedures. As noted, the images were chosen as typical of the features over the coupon surface area.

The typical surface topography of a coupon surface after inhibited HCl for 10 minutes is shown in Fig. 1. There is a background clearly recognizable as the surface of the steel, with, scattered over the surface, the presence of isolated rust products, similar in appearance to rusts typically generated by localized pitting corrosion [15]. After 30 minutes very little of the steel surface was visible and there was a much greater density of corrosion products. Very similar observations were made for coupons that had been subject to cleaning with Clarke's solution for 10 minutes (Fig. 2) and 30 minutes, although the density of rust product features was greater.

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Figure 1. Coupon A1 after 10 minutes agitated immersion in Inhibited HCl (Table 2). The background is the steel surface. The doughnut-shaped and smaller features were identified by EDS as predominantly Fe. Scale bars: left = $200 \,\mu m$, right = $20 \,\mu m$.



(a)

(b)

Figure 2. Coupon C1 after 10 minutes agitated immersion in Clark's solution (Table 2). A few areas of the background steel can be observed. The doughnut-shaped features are more numerous and conglomerated compared to the coupons cleaned with inhibited HCl. They were identified by EDS as predominantly Fe. There is some evidence of remaining hard marine growth (top centre). Scale bars: left = $200 \,\mu m$, right = $20 \,\mu m$.

In the case of electrolytic cleaning the coupon surface after 1 hour exposure, when examined by SEM, shows a predominantly metallic surface with some evidence of pitting and localized corrosion and with the presence of several small shell-like features (Fig. 3). These were identified by energy dispersive X-ray spectroscopy (EDS) as predominantly Ca and C. This suggests the presence of CaCO₃. Exposure to electrolytic cleaning for 2 hours reduced the size of the shell-like features but otherwise there was little difference. In both cases the microstructure of most of the steel coupon surface was clearly visible. Exposure of unexposed coupons to electrolytic cleaning produced no noticeable changes to the metal surface, even after 2 hours exposure.



Figure 3. Coupon E1 after 1 hour of cleaning using Electrolysis (Table 2). The background is the steel surface depicting pitting and a few peculiar shell-like features. The features shown at left were identifies by EDS as predominantly Ca and C and thus may be presumed to be CaCO₃. The steel microstructure is visible (right). Scale bars: left = $200 \,\mu$ m, right = $20 \,\mu$ m.

Coupon cleaning with a gentle stream of tap water left a surface that appears to be predominantly steel but with what night be called shell-like features (Fig. 4). These were identified by EDS as predominantly Ca and C. Water washing with a strong stream (jet) of water produced generally similar results but with smaller remaining shell-like features. For both the water washing protocols the surface after cleaning was not completely clear of surface deposits. In neither case was the metal microstructure completely revealed over the whole surface.



(a)

Figure 4. Coupon T1 after gentle washing in a stream of soft tap water (Table 2). The background is the steel surface. The features shown at left and the magnified feature at right are predominantly Ca and C and thus may be presumed to be CaCO₃. Scale bars: left = $200 \,\mu\text{m}$, right = $20 \,\mu\text{m}$.

The surfaces of the blank coupons (i.e. those not exposed to seawater and thus without marine corrosion products) exposed for 2 minutes to inhibited HCl showed very little other than clean metal. Detailed microscopic examination of the surfaces showed some very small signs of corrosion features in isolated areas.

This was also the case for blank coupons subjected to 5 minutes of inhibited HCl (Fig. 5). The small features remaining have doughnut-like appearance, generally similar but smaller than those observed in Figs. 1-2. Blank coupons exposed to inhibited HCl for 10 minutes showed features and surface characteristics generally similar but more severe than those shown in Fig. 5.



Figure 5. Blank coupon subject to 5 minutes of inhibited HCl showing, at high magnification, the majority of the coupon surface. Scale bar: $10\,\mu\text{m}$.

The effect of application of chemical treatment followed by ultrasonics applied to blank coupons can be seen in the example using Clarke's solution applied for 10 minutes in accordance with the protocol (Table 2). The corrosion features caused by Clarke's solution are similar to those shown in Fig. 5 but after 15 minutes application of ultrasonics all these were observed to have been removed. There was clear evidence of pitting (Fig 6).

Exposure of blank coupons directly to only ultrasonic cleaning produced no noticeable changes to the metal surface, even after 4 hours exposure. Similarly, exposure of blank coupons to electrolysis produced no discernible effect on the steel surface. These results show that ultrasonic cleaning and also cleaning using electrolysis have no side-effects such as corrosion loss, localized corrosion or pitting.



Figure 6. Surface of blank coupon after 10 minutes exposure to Clarke's solution, followed by removal of products by ultrasonics, showing pitting. Scale bar: $10 \,\mu m$.

As noted in Section 2, all coupons were weighed immediately prior to exposure to seawater and again after application of the cleaning protocols. Similarly, the blank were weighed before and after the application of a cleaning protocol (if used) and again after exposure to ultrasonic cleaning. At each occasion the coupons were weighed a number of times. The average mass is that shown for each in Table 4. Also shown are the corresponding mass losses for each cleaning protocol and the accuracy to which weight measurements were made. The standard deviation for the weighing process was estimated from repeated sample measurements as 0.0008 g. The mass loss results for the tests on blank coupons exposed to the protocol for Clarke's solution and subsequently to ultrasonic cleaning are shown in Table 5. These show that increased exposure to Clarke's solution causes increased mass losses. Strictly these should be termed 'apparent mass losses' since in all cases the application of ultrasonic cleaning further increased the mass loss.

	A1	A2	C1	C2	E1	E2	T1	T2	U
Before marine exposure (g)	14.1292	14.1095	14.1608	14.1773	14.1167	14.1973	14.2023	14.1189	14.1608
After both marine exposure and cleaning protocol (g)	14.0421	14.0135	14.0742	14.0793	14.0342	14.1123	14.1425	14.0526	14.0961
Mass Loss (g)	0.0871	0.0960	0.0866	0.0980	0.0825	0.0850	0.0598	0.0663	0.0648

Table 4. Average weights and calculated total mass losses for coupons after 4 weeks marine exposure and cleaning protocol.

Table 5. Average weights and mass losses for blank coupons subjected to Clark's solution and subsequent ultrasonic cleaning.

Time in Clarke's solution (min)	Mass of blank coupon (g)	Mass after Clarke's solution (g)	Apparent mass loss (g)	Mass after ultrasonic cleaning (g)	Total mass loss (g)
10	14.0379	14.0293	0.0086	14.0281	0.0098
20	14.1620	14.1521	0.0099	14.1504	0.0116

The total mass losses, including that from marine corrosion over 4 weeks exposure, relative to the duration of each cleaning process are summarized in Fig. 7. If the cleaning processes had no effect the total mass losses would be expected to be very similar. However, this is not the case. The coupons subjected to a gentle stream wash of tap water show the lowest mass loss, followed by the coupons cleaned using a strong jet stream of tap water. The coupons cleaned using the electrolytic method show very similar mass losses, irrespective of cleaning duration (60 min and 120 min). Coupons cleaned by the inhibited HCl or using Clarke's solution show higher (apparent) mass losses and these increase with longer exposure to the cleaning solutions. Application of ultrasonic cleaning after the use of Clarke's solution for 10 and for 30 minutes, considerably increased the corresponding total mass losses.



Figure 7. Comparison of coupon mass losses after 4 weeks of marine corrosion and after application of different cleaning protocols. See text for meaning of trends MN and KL.

4. Discussion

The results of the present investigation are summarized in Fig. 7. Both inhibited HCl and Clarke's solutions are effective in removing the original marine rust products but also cause additional corrosion and associated corrosion products, both made worse by longer exposure to these solutions (10 minutes vs. 30 minutes). This is indicated also by the light trend lines shown for these results in Fig. 7.

Application of ultrasonic cleaning to the coupons already subjected to Clarke's solution increased the mass losses and more so for the 30 minute exposure to Clarke's solution than for the 10 minute exposure (Fig. 15). These higher losses are a more accurate reflection of the effect of Clarke's solution on the steel. A similar result would apply for the inhibited HCl. Further, the trend line MN shown through the total mass loss results represent the rate of increase in the metal loss caused by the cleaning process. Conversely, an estimate of the mass loss that resulted from marine exposure, without the influence of chemical cleaning mass loss is given by the projection of the trend MN back to the left axis.

The electrolysis cleaning method removed most rust products after application for one hour (Fig. 3) and effectively all rust products and other surface features after 2 hours. For both exposure durations the metal grain structure was clearly revealed. After one hour electrolysis a small number of shell-like features were seen on the surface (Fig. 3) but these were not apparent after 2 hours exposure. EDS showed these features to contain Ca and C, suggesting they are CaCO₃. This is consistent with observations that marine rusts contain CaCO₃ [16]. However, it is also possible that the fragile shell-like features are the remnants of shells or other hard marine growth, composed of CaCO₃, that were attached to the surface of the coupons, with electrolysis insufficient to remove them within one hour but sufficient to remove them within 2 hours. This is consistent with the well-established reduction of CaCO₃, such as in water hardness reduction, using electrolysis [17]. In Fig. 7 the lower apparent mass loss at 1 hour compared with 2 hours can now be seen to be the result of the remnant CaCO₃ features. Hence the line KL projected back to the left hand axis represents the limit of mass loss that should have been obtained in all cleaning procedures.

The coupons for which cleaning was attempted by a gentle wash with tap water and by a strong stream of tap water revealed considerable areas showing metal grain structure (Fig. 4). However, it was only partly effective as some isolated deposits remained in both cases. These have a fragile shell-like appearance and EDS examination showed the presence of Ca and C, again suggesting they consist of CaCO₃ and could have arisen from the same mechanisms.

The water cleaning procedure cannot be considered sufficiently effective for 4 week-old marine rusts to be removed. Also, as a result of the remaining deposits, the measured mass after washing would be greater than if all the rusts had been removed from the coupons.

In the present experiments no specific attention was given to the composition of the corrosion products. For steels in seawater immersion conditions these are well-known and for the Taylors Beach site have been earlier reported [18]. Similarly, the corrosion products resulting directly from the application of inhibited

HCl and of Clarke's solution also are well-known [4,19]. The experimental evidence reported herein clearly separates out the corrosion associated with the marine rusts and the localized corrosion and associated corrosion products caused by the action of the cleaning solutions.

5. Conclusion

1. The conventional ASTM inhibited HCl solution and also Clarke's solution removed a proportion of the rusts from 4 weeks of marine immersion exposure, caused localized corrosion of their own and caused deposition of new corrosion products. The severity of the new localized corrosion and the associated corrosion products increased with longer exposure to the cleaning solutions.

2. Gentle washing with tap water removed most of the marine rusts but left calcareous material. More, but not all calcareous materials were removed using a strong jet of tap water.

3. Electrolytic cleaning was the most effective cleaning process for removing 4 week old marine rusts but it took some 2 hours for the metal surface to be fully revealed. No additional corrosion or additional corrosion products were observed. Features consistent with calcium carbonate remained after a shorter application of electrolytic cleaning.

4. Provided the exposure period is sufficient, ultrasonics was found to be effective in removing weak corrosion products such as those from acid cleaning. It did not cause additional corrosion or metal surface damage. However, it was ineffective in removing more substantial rusts even after 12 hours application.

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