Corrosion Testing of Carbon Steel Exposed to Sludge Heel Chemical Cleaning Solutions – 16109

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ABSTRACT

As efforts continue to treat and dispose of millions of gallons of legacy radioactive materials from the production of nuclear weapons, non-compliant waste storage tanks will gradually be emptied of the bulk waste volume leaving heel materials requiring removal prior to tank closure. The waste heel slurries are distributed on the floor of large, million gallon tanks, which frequently contain numerous obstructions that limit the effectiveness of mechanical removal methods. As a result, chemical cleaning methods are needed for the effective removal of the heels as well as chemical scales that are present on the tank walls and other interior surfaces.

Two tank cleaning technologies have already been implemented at the Savannah River Site (SRS): Low Temperature Aluminum Dissolution (LTAD) and Bulk Oxalic Acid Cleaning (BOAC). Recent chemical cleaning efforts on SRS Tank 12 were very successful with regard to bulk sludge heel (especially for Al, Fe, and U phases) and beta/gamma radionuclide removal. Although chemical cleaning using these technologies has been shown to be effective, no disposition path has been identified for oxalate added during BOAC, and insoluble oxalate salts are accumulating within the SRS tank farm and waste processing facilities (evaporators, etc.). Extensive sludge washing is also required to remove moderately soluble sodium oxalate salts prior to sludge vitrification in the SRS Defense Waste Processing Facility (DWPF). As a result, oxalate additions to the tank farm need to be minimized by the use of supplementary acids to assist sludge removal in OA or the use of other cleaning reagents or processing strategies.

Previous SRNL testing revealed the importance of pH control for BOAC and recommended the use of a supplementary acid (dilute HNO_3) with OA to minimize oxalate additions. Primary drivers in SRS Tank Closure Performance Assessments, which evaluate the fate and impact of tank sludge residuals on a geological timescale, are the removal of alpha emitting radionuclides present at low concentrations such as Pu, Am, and Np, which are not highly soluble in currently utilized chemical cleaning reagents. Scoping studies conducted at SRNL revealed promising methods to dissolve the actinides within the HLW tank heels. Oxidation of the actinides with permanganate in either strong caustic (10 M NaOH) or dilute

acidic (0.2 M HNO$_3$) solutions was shown to result in dissolution of oxy/hydroxide phases of these metals in the absence of major sludge phases.

The waste tanks at SRS and Hanford are constructed of carbon steel. Carbon steel corrodes rapidly in most acidic and/or oxidizing environments. Previous electrochemical corrosion testing indicated that the corrosion rates in dilute HNO$_3$ with OA may result in manageable corrosion rates, over short exposure periods. Additional corrosion testing was performed to provide data for the nitric acid/OA blend as well as the acid/permanganate and caustic permanganate based chemical cleaning solutions.

Corrosion rates from the passive coupon tests for the nitric acid/oxalic acid blend were significantly greater than those observed previously with the electrochemical testing. This result may be due to simultaneous electrochemical and chemical dissolution of the steel. The corrosion rate data from the coupon tests in the nitric acid/oxalic acid blend were evaluated to determine the degree of potential structural damage. The corrosion rates, although relatively high, would not be expected to cause damage that would reduce the capacity of the tank primary wall if the process is completed within a month.

The corrosivity of sodium permanganate in four proposed cleaning solutions, 1 nitric acid solution and 3 sodium hydroxide solutions, was studied by electrochemical methods at room temperature. The corrosion rates were significantly less aggressive than in the nitric acid/oxalic acid blend corrosion tests. Passive coupon tests needs to be performed in these solutions to ensure that chemical dissolution of the metal is not occurring simultaneously.

INTRODUCTION

The U. S. Department of Energy (DOE) Office of Environmental Management (EM) has tasked the Savannah River National Laboratory (SRNL) with developing strategies and technologies to chemically clean radioactive High Level Waste (HLW) tanks prior to tank closure.[1] Two tank cleaning technologies have already been implemented at the Savannah River Site (SRS): Low Temperature Aluminum Dissolution (LTAD) and Bulk Oxalic Acid Cleaning (BOAC). Although chemical cleaning using these technologies has been shown to be effective, no disposition path has been identified for oxalate added during BOAC, and insoluble oxalate salts are accumulating within the SRS tank farm and waste processing facilities (e.g. evaporators).[2] Extensive sludge washing is also required to remove moderately soluble sodium oxalate salts prior to sludge vitrification in the SRS Defense Waste Processing Facility (DWPF). As a result, oxalate additions to the tank farm need to

be minimized by the use of supplementary acids to assist sludge removal in oxalic acid (OA) or the use of other cleaning reagents or processing strategies.

Previous SRNL testing[3-4] revealed the importance of pH control for BOAC, recommended the use of a supplementary acid (i.e. dilute HNO_3) with OA to minimize oxalate additions,[5] and indicated that marginal corrosion rates would be observed with these acid blends.[6] The heel pH was maintained to near the ideal value for sludge dissolution during BOAC (~pH 2) in SRS Tank 12, but a supplementary acid was not utilized.

Primary drivers in SRS Tank Closure Performance Assessments, which evaluate the fate and impact of tank sludge residuals on a geological timescale, are the removal of alpha emitting radionuclides present at low concentrations such as Pu, Am, and Np, which are not highly soluble in currently utilized chemical cleaning reagents. Scoping studies conducted at SRNL revealed promising methods to dissolve the actinides within the HLW tank heels.[7] Oxidation of the actinides with permanganate in either strong caustic (i.e., 3, 5, and 10 M NaOH) or dilute acidic (i.e., 0.18 M HNO_3) solutions was shown to result in dissolution of oxy/hydroxide phases of these metals in the absence of major sludge phases. Either of these two permanganate-based methods for alpha removal might be suitable for incorporation into a chemical cleaning flow sheet, though they would likely be utilized at different times in the processing sequence. Utilization of permanganate-based methods results in the addition of manganese oxide solids to the waste, so minimization of permanganate additions is needed.

This paper presents the results of the corrosion evaluation component of the Alternative Chemical Cleaning program. The corrosion evaluation was divided into two tasks. Task 1 focused on one month corrosion tests that included electrochemical probe measurements and passive, weight loss coupons to evaluate corrosion loss using the nitric acid/oxalic acid chemical cleaning solution along with sludge waste simulants at two dilution conditions. Task 2 is a corrosion screening test that concentrated on the corrodibility of sodium permanganate in the conditions that would be used for in-tank treatment of the sludge types of interest.

EXPERIMENTAL METHODS

American Society of Testing and Materials (ASTM) A285 carbon steel materials were utilized for the Task 1 and Task 2 corrosion tests. The Type I and II SRS waste tanks, which are the initial tank groups targeted for chemical cleaning and closure, were constructed of A285 carbon steel. The coupons were polished to a 600 grit finish to provide a uniform, reproducible surface prior to testing.

The test material, 304L stainless steel, was also used for the corrosion tests. SRS tank farm transfer lines and ventilation system materials were constructed of 304L stainless steel. The coupons were polished to a 600 grit finish to provide a uniform, reproducible surface prior to testing.

For Task 1 the cleaning solution utilized for these tests contained a mixture of 0.18 M nitric acid (NA) and 0.056 M (0.5 wt. %) oxalic acid (OA) based on previous recommendations for OA cleaning.[7] The test temperature was 50 °C and the two acid: sludge volume ratios tested were: 20:1 and 50:1. These ratios were assumed to simulate the multiple acid strikes (cumulative acid to sludge volume ratio 50:1) typically used for the waste tank chemical cleaning process and BOAC baseline (20:1).

Two sludge simulant formulations, which are representative of SRS PUREX and HM sludge types, were evaluated. The recipes were developed to simulate the dissolution characteristics of waste tank heels by the addition of portions of the major Fe and Al sludge components as oxide or hydroxide phases. The HM sludge slurry simulant recipe was comprised primarily of aluminum, iron, and manganese oxides and hydroxides with numerous of secondary metal oxides and hydroxides. In addition, the sludge simulant also included the hazardous metals Ag, Ba, Cd, Cr, Hg and Pb. The PUREX sludge slurry simulant was comprised primarily of iron and manganese oxides and hydroxides with numerous of secondary metal oxides and hydroxides, but also included Ag, Ba, Cd, Cr, Hg and Pb. Minor metal species present in these simulant formulations could have significant impacts upon the corrosivity toward steel. In order to simulate conditions in the waste transfer lines, tests on 304L stainless steel were conducted using spent acid solutions produced by the tests above.   The vessels containing the acid and simulant were used without stirring leaving the coupons exposed to the spent acid solutions. The coupons were exposed to these NA/OA solutions for 4 weeks at a test temperature of 50 °C.

For Task 2, testing was performed utilizing A285 Grade C carbon steel working electrodes. Four test solutions were utilized:

1)   10 M sodium hydroxide / 0.05 M sodium permanganate
2)   5 M sodium hydroxide / 0.05 M sodium permanganate
3)   3 M sodium hydroxide / 0.05 M sodium permanganate
4)   0.18 M nitric acid / 0.05 M sodium permanganate

In addition to tests in these solutions, tests were performed in these solutions combined with the PUREX and HM simulants. These conditions will simulate the actual chemical cleaning process. The liquid to solid phase ratios used for testing were 20:1. Tests were conducted at ambient temperature with temperature

monitoring. In order to obtain better dissolution of the solids, the waste is typically agitated by pumps. Previous laboratory testing has also suggested that agitation results in higher general corrosion rates. Therefore, the simulants were agitated during the testing.[6]

Task 1: Corrosion Tests in Nitric Acid/Oxalic Acid

Changes in the solution corrosivity (i.e., aggressiveness of the environment) and alloy corrodibility (i.e., corrosion susceptibility, passive layer formation and/or degradation, etc.) can be determined with a planned interval testing program. In these tests, the waste simulants are contacted with the NA/OA cleaning reagent for a period of four weeks and steel coupons are immersed in this slurry for the intervals indicated. A minimum set of 3 flat, rectangular coupons with a surface area of 34.95 cm^2 (5.42 in^2) is initially exposed to the corrosive environment of interest. Duplicate or more sets may be included as well for statistical validity. Coupon #1 was removed after 1 week of exposure, coupon #2 was to be removed after 3 weeks of exposure, but was inadvertently removed after 2 weeks. Coupon #3 was removed after 4 weeks of exposure. Coupon #4, another flat, rectangular coupon, was placed in at 3 weeks and was removed along with Coupon #3 after week 4.

In-situ monitoring of the open circuit potential and the general corrosion rate during the test was performed with electrochemical probes. The electrochemical probes were prepared from the same A285 carbon steel material as the coupons. The pre-mounted probe was nominally 0.25″ x 0.25″ x 0.3″ (i.e. 0.425 in^2 or 2.73 cm^2)(see Figure 1.)

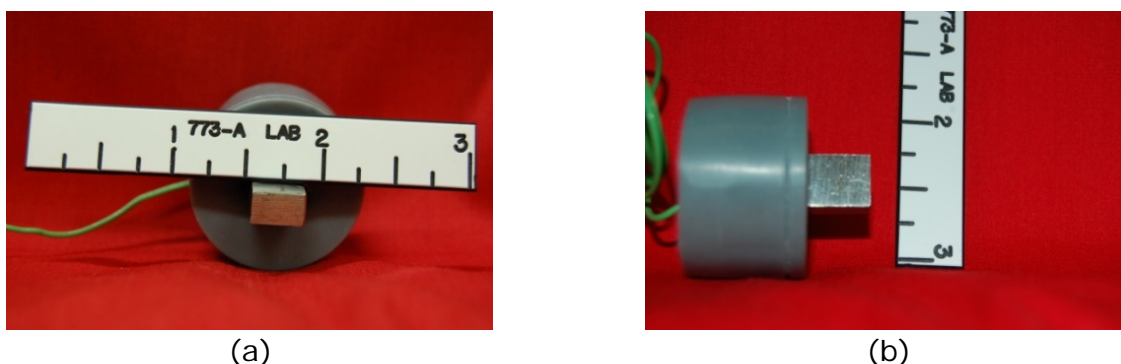| (a) | (b) |

Figure 1. Electrochemical probe for tests: (a) end view and (b) side view.

The tests with the electrochemical probes were performed concurrently with the coupon tests. To perform the corrosion measurements within each test vessel, an electrochemical cell was designed.

Task 2: Corrosion Tests in Permanganate Solutions

The electrochemical tests were conducted using a flat square electrode of A285 Grade C carbon steel embedded in epoxy with a flat exposed surface area of 3.629 cm^2. The electrode was connected by a piece 22 gauge wire that was spot welded to the sample before embedding. The sample was then polished to a 600-grit finish. After each set of measurements, the sample was resurfaced by wet polishing first with 240-grit sand paper, then 600-grit paper.

Four electrochemical tests were performed at each condition:

1)   monitoring of the open-circuit potential (OCP),
2)   linear polarization resistance testing (LPR),
3)   cathodic polarization testing (CP), and
4)   cyclic polarization testing (CPP).

These tests were conducted with a PAR 273A potentiostat. Graphite rods were used as counter electrodes. In the first three tests with sodium hydroxide and sodium permanganate only, an $Ag/AgCl_2$ reference electrode was used, but the permanganate ion was observed to penetrate the electrode and appeared in the electrode solution. A luggin bridge was used with a saturated calomel electrode (SCE) to prevent continuous maintenance of reference electrodes and preserve data quality. The tests were measured with respect to the reference potentials.

The OCP reflects a measure of the activity at the metal surface for all oxidation and reduction reactions, i.e. whether it is actively corroding or passive, while the LPR testing will give a direct measure of the instantaneous corrosion rate. The cathodic polarization (CP) is performed to understand the nature of the cathodic reaction, while the cyclic potentiodynamic polarization (CPP) testing was performed separately to reveal any vulnerability to localized corrosion, such as stress corrosion cracking and/or pitting. The open circuit potential was measured during a 10-minute rest period between techniques to monitor for any significant changes during testing. These tests provide necessary mechanistic information to support the results of future coupon tests as well as screen for potential process conditions, which may result in lower corrosion rates. The conditions with lower corrosion rates could then be the focus of the coupon tests.

RESULTS AND DISCUSSION

Task 1: Nitric Acid/Oxalic Acid Results

The open circuit potentials were measured daily for the active probes in each vessel. Figure 2 shows the trend of the open circuit potentials for the duration of the testing for electrodes. The potential remained relatively constant below -300 mV (vs. Ag/AgCl). The potentials are comparable to those for oxalic acid alone. Based on this data, hydrogen evolution is thermodynamically possible as the potential is less than that for hydrogen evolution. Cathodic polarization studies would be needed to study the kinetics of the cathodic reaction to determine the rate of hydrogen evolution.

In addition, a linear polarization resistance measurement was collected daily from which the corrosion rate could be calculated. Figure 3 through Figure 6 compare the corrosion rates measured from the LPR tests. In Figure 3, the HM 20:1 test shows the corrosion rate rose to a maximum after 12 days of testing, settling then around 100-120 MPY before decreasing to 50 MPY at the end of the testing. This behavior is similar to the PUREX 20:1 in Figure 5 where the corrosion rate increases from about an average of 50 MPY, peaks around 90 to 115 MPY then gradually decreases to the 40 to 60 MPY range at the end of the experiment. From Figure 4, the HM 50:1 corrosion rates exhibit a greater disparity between the electrodes. Electrode 3 shows a downward trend in the corrosion rates as the rate begins at 55 MPY and decreases to around 33 MPY at the end of the testing. Electrode 4, however, show more aggressive corrosion rates in the third quarter of the testing, but had a similar decreasing trend at the end of the testing. The PUREX 50:1 test results, shown in Figure 6, exhibit a decline in the corrosion rate that is somewhat progressive. The corrosion rate appears to decrease steadily from around 150 MPY in the early stages of testing to about 55 MPY at day 12. Around day 15, the rate decreases again and remains steady around 40 MPY and then drops to below 30 MPY at the end of testing.

The integrated corrosion rate was calculated by integrating the daily measured instantaneous corrosion rates and dividing by the duration of the test. The results are presented in Table 1, along with the maximum corrosion measured during the testing.
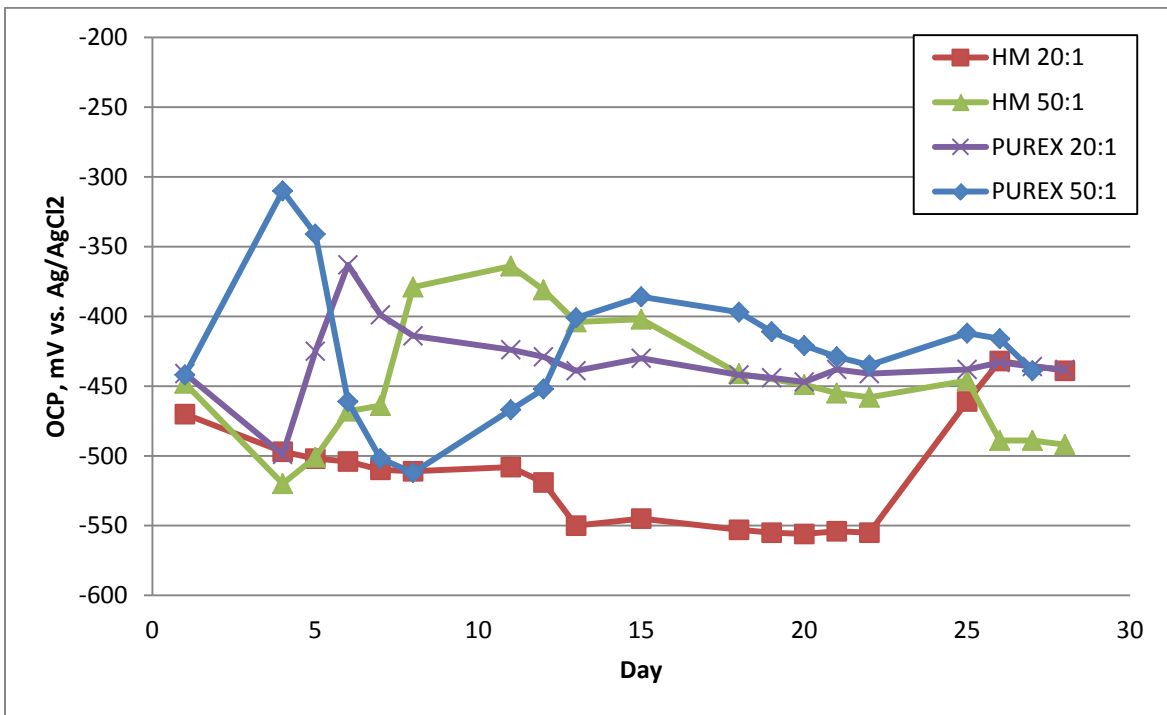
Figure 2. Open Circuit Measurements for the Active Electrodes in Each Test Solution.
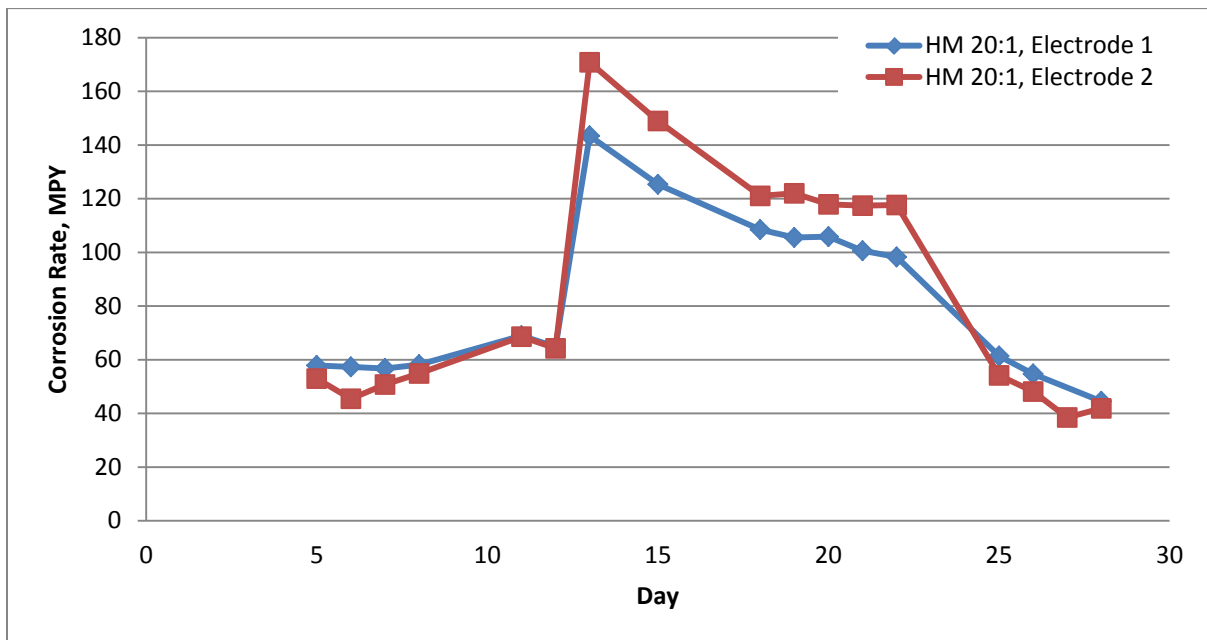

Figure 3. Corrosion Rate for HM 20:1 Test Solution Calculated from the Linear Polarization Measurement.
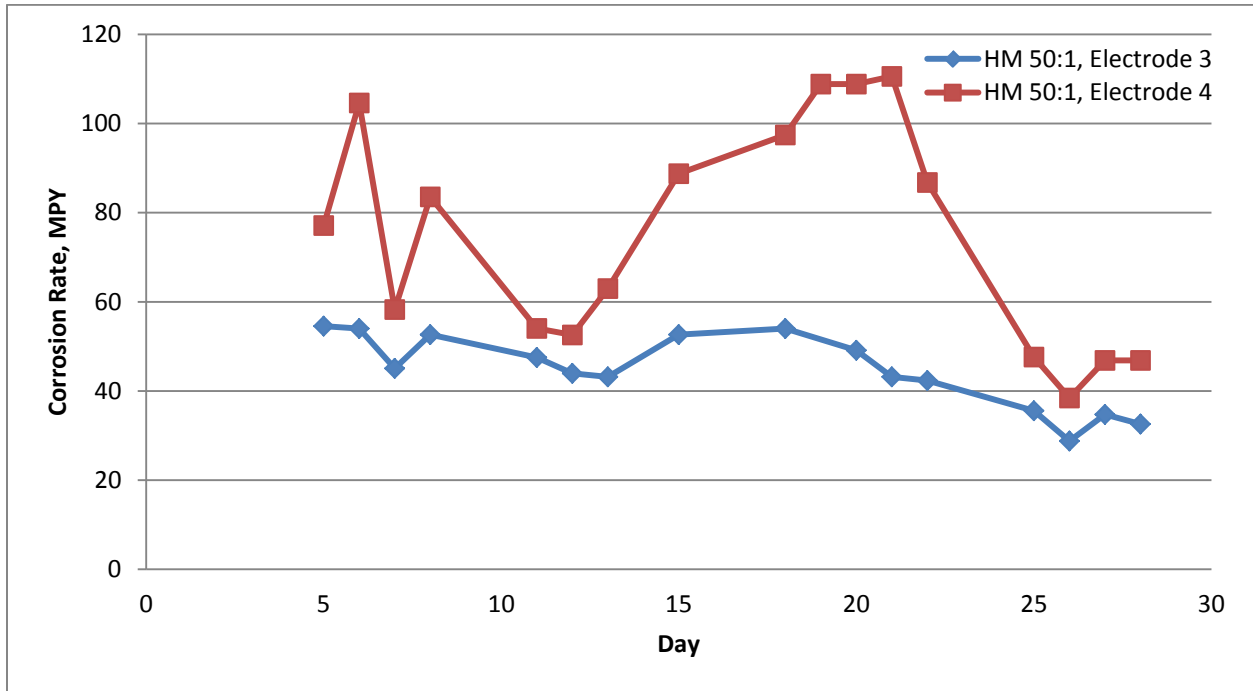
Figure 4. Corrosion Rate for HM 50:1 Test Solution Calculated from the Linear Polarization Measurement.
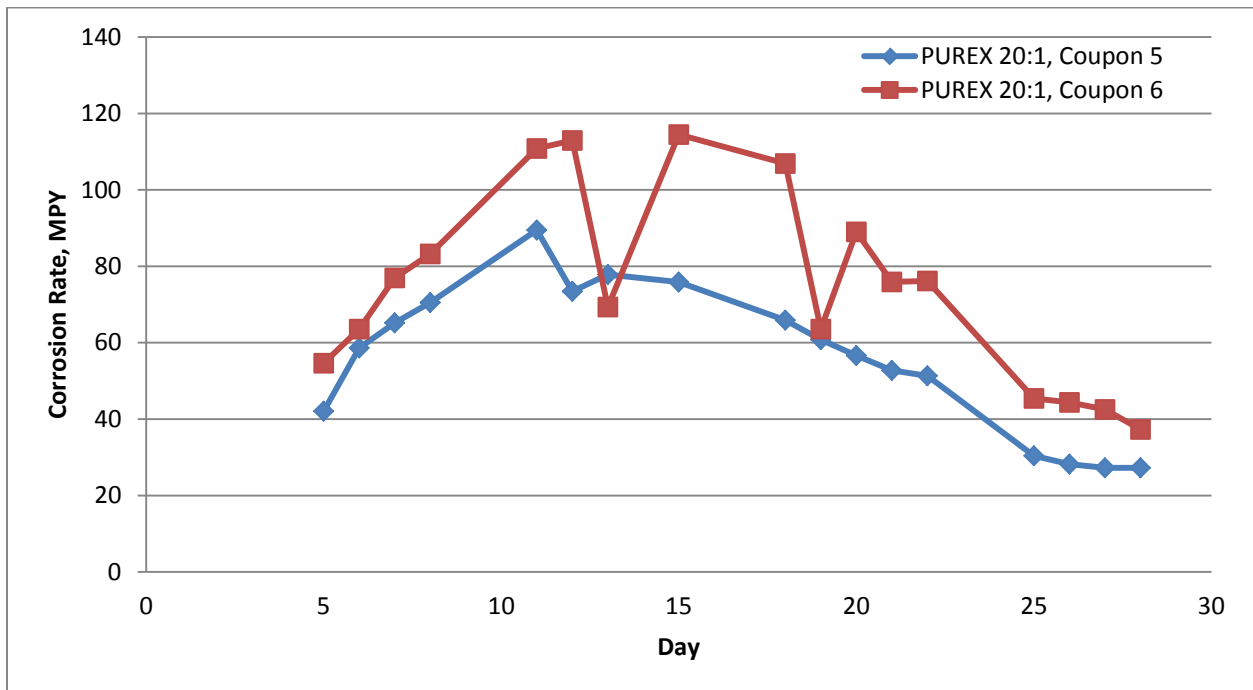

Figure 5. Corrosion Rate for PUREX 20:1 Test Solution Calculated from the Linear Polarization Measurement.
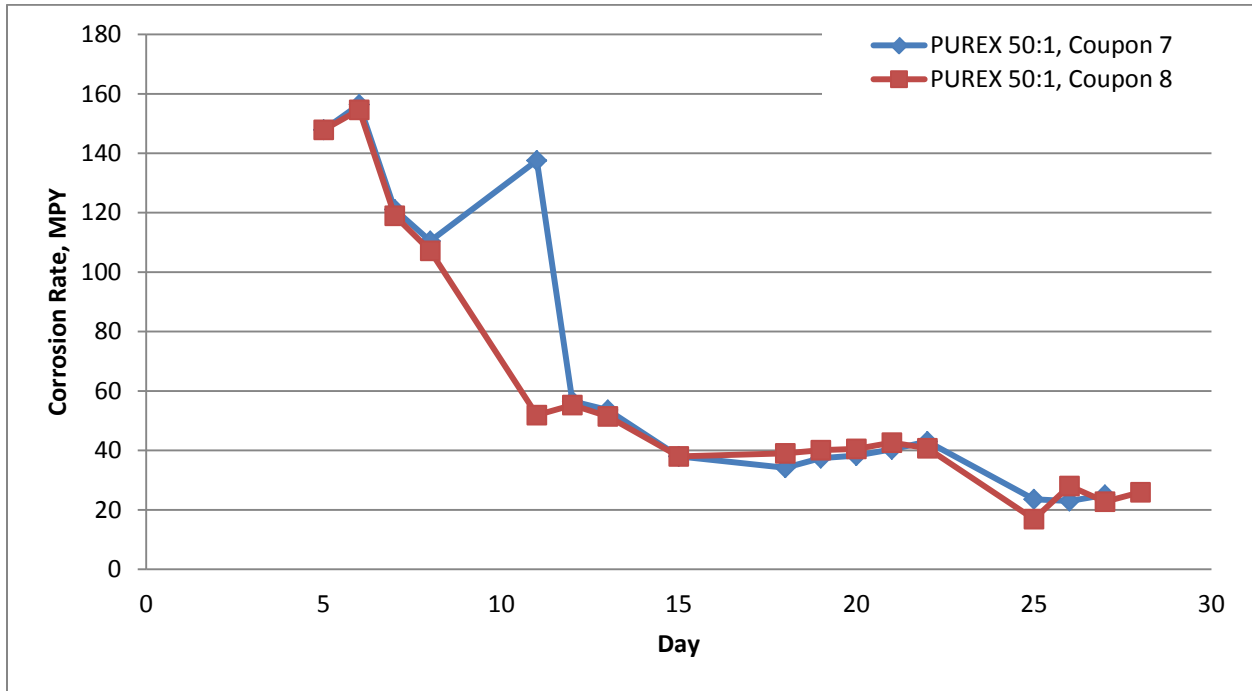
Figure 6. Corrosion Rate for PUREX 50:1 Test Solution Calculated from the Linear Polarization Measurement.

Table 1. Integrated and Maximum Corrosion Rates Calculated from the Linear Polarization Measurements.

	Electrode	Maximum Measured Corrosion Rate, MPY	Integrated Corrosion Rate, MPY
HM 20:1	1	143	70.2
	2	171	75.7
HM 50:1	3	111	36.5
	4	55	62.2
PUREX 20:1	5	114	49.3
	6	89	65.9
PUREX 50:1	7	156	52.0
	8	155	45.7

The data presented in Table 2 are the results of the weight loss measurements from the passive corrosion coupons that were exposed to the testing solutions. The first 3 coupons were exposed for 1, 2, and 4 weeks, respectively. The corrosion rates exhibited the following general trends. The 4 week coupon test data can be

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compared with the integrated corrosion rate data shown in Table 1. The dissolution of the PUREX sludge in NA/OA results in a simulant that is more corrosive than the solution that results from dissolution of the HM sludge. Corrosion rates for the PUREX simulants ranged from approximately 115 to 490 MPY, while for the HM simulants, the rates ranged from approximately 75 to 330 MPY. It should be noted that the corrosion rates for coupons 2 and 3 for the HM 50:1 were likely influenced by an adjustment that was made during the test. This test solution had a pH that was higher than expected after the first week of testing. At the time, 1 liter of acid solution was removed from the vessel and replaced with a new liter of solution. This step brought the pH back into alignment with the pH expectations for the testing. However, this likely increased the corrosion rates that were measured for coupons 2 and 3. If this is the case, for all comparable test conditions, the corrosion rates were higher in the PUREX simulants.

The high acid to sludge ratio (50:1) results in a solution that is more corrosive than the low acid to sludge ratio (20:1). For example, the corrosion rates for the PUREX simulant at a 50:1 ratio ranged from approximately 250 to 490 MPY, whereas for the 20:1 ratio the rates ranged from approximately 120 to 240 MPY. A similar trend was observed for the HM simulant.

Table 2. Corrosion Rates in Mils Per Year from Passive Exposure Coupons Determined by Weight Loss.

Coupon	Time (hrs)	HM 20:1	HM 50:1	PUREX 20:1	PUREX 50:1
1	168	152.3	265.2	240.2	486.0
2	384	75.8	327.9	115.7	248.9
3	672	162.1	159.2	156.1	310.4
4	168	233.3	138.6	232.6	159.9

Overall, there was a discrepancy with the corrosion rates from the passive coupons and the corrosion rates measured by LPR. In general, the corrosion rates from the passive coupons are 2-6 times greater than those measured by LPR. This phenomenon has been observed for iron, chromium, and their alloys in acidic media.[8] Examples of this occurrence include: 1) iron in sulfuric acid that contains additions of potassium chromate, 2) stainless steel in hydrofluoric-nitric acid solutions, and 3) an iron-chromium alloy in sulfuric acid containing hydrogen peroxide.[9] In these cases, metal dissolution occurs not only by an electrochemical mechanism, but in parallel with a chemical mechanism. No current is passed by the chemical dissolution mechanism and hence the LPR measurements would underestimate the actual weight loss. Thus, electrochemical measurements cannot

be utilized to determine corrosion rates without major corrections. One commonality of these environments is that a reducing acid is in the presence of a strong oxidizer. In the present case, oxalic acid is a reducing acid and nitric acid is a strong oxidizer. Additional tests (i.e., potentiostatic and potentiodynamic) would need to be performed to confirm the chemical mechanism, but the discrepancy between the coupon and LPR results suggests that this is a possibility.

This observation has ramifications for both structural and flammability analysis. The OCP data suggests that hydrogen evolution is possible. However, due to chemical dissolution of the material, the hydrogen evolution rate may not be as high as predicted by the corrosion rates measured by the coupons. On the other hand, structural damage would likely exceed anticipated values if the corrosion rates from the LPR tests were accepted. Based on the present data, the corrosion rates from the coupon tests should be utilized for structural evaluations, while further kinetic studies are needed to evaluate hydrogen evolution.

The stainless steel tests yielded little to no corrosion for the exposure tests of 2 and 4 weeks. The corrosion rates determined by weight loss were negligible with the highest corrosion rate being 0.2 MPY.

Task 2: Sodium Permanganate Tests

Table 3 shows the results of the OCP measurements with respect to the saturated calomel reference electrode (SCE). Each test was run in duplicate. During the first tests with the 10 M, 5 M, and 3M NaOH solutions, a green Mn^{6+} species penetrated the glass frit of the Ag/AgCl reference electrodes.   The OCP values are significantly more noble than those values that would be observed in either a simple sodium hydroxide solution or nitric acid solution. This observation is reflective of the permanganate, which is a strong oxidizer. The potentials are at values well above the value for hydrogen evolution. The potentials do become more noble as the pH decreases.

The addition of the simulant to the cleaning solutions did not impact the OCP more than 100 mV for the most extreme case. The 5 M NaOH solutions with HM and the PUREX showed the greatest separation at 106 mV at the most extreme values. The other 3 groups fall within a 60 mV window where the greatest variation between the maximum and minimum values is 55 mV.

Table 3. Open Circuit Measurements Sodium Permanganate Solutions

Test	NaOH (M)	Simulant	OCP, mV vs. SCE	Corrosion Rate (MPY)
1	10	None	246	2.14
2	10	PUREX	238	2.99
3	10	HM	241	1.22
4	5	None	342	0.878
5	5	PUREX	344	7.83
6	5	HM	269	0.457
7	3	None	407	22.5
8	3	PUREX	381	28.8
9	3	HM	400	24.6
0.18M HNO₃				
10	0.18	None	1197	1.77
11	0.18	PUREX	1173	2.73
12	0.18	HM	1167	1.17

Table 3 also gives the general corrosion rates for the LPR tests on the test solutions. The highest corrosion rates found were for the 3M NaOH: 0.05M $NaMnO_4$ solution. These corrosion rates are very low, much lower than observed with the oxalic acid and oxalic acid/nitric acid blend, and would not present an issue. Coupon tests would be necessary to confirm, however, that a chemical dissolution mechanism is not occurring due to the presence of the strong oxidizer (i.e., permanganate).

The results of the CPP curves showed primarily active dissolution of the metal that reaches a high limiting current value with only a small degree of polarization. There was a slight indication of positive hysteresis in the CPP curve. It is possible the hysteresis could be an artifact of another process, such as deposition of a metal from solution.

Cathodic polarization testing was performed to determine the kinetics of the cathodic reaction and if hydrogen will be evolved as an active process when the acid interacts with the waste. In order for hydrogen evolution to occur, the value a must be within a specific range and below the potential for the hydrogen reduction reaction. In the basic conditions, where the potential is the lowest, the potential was positive for all solutions. Therefore, the free potential is too noble for hydrogen evolution. Permanganate reduction or oxygen evolution is the more likely candidate for the cathodic reaction.

CONCLUSIONS

The testing presented in this report examined the corrosion of A285 carbon steel exposed to a chemical cleaning solution composed of 0.18M nitric acid and 0.5 wt. % oxalic acid at 50℃. This solution has been proposed as a chemical cleaning solution for the hard heel portion of the sludge in the waste tanks. When this solution was combined with the HM or PUREX simulated sludge, the corrosion rates determined from passive coupon tests were nearly 500 mils per year in the worst case (for PUREX simulant at a 50:1 acid to sludge ratio), but decreased with time in 28-day tests. The highest corrosion rate determine by electrochemical probes was 76 mils per year (for HM 20:1.) The tests showed corrosion behavior to be active general corrosion.

Based on weight loss determination, the high acid to sludge ratio (50:1) results in a solution that is more corrosive than the low acid to sludge ratio (20:1). Additionally, the dissolution of the PUREX sludge results in a simulant that is more corrosive than the solution that results from dissolution of the HM sludge.

Both dissolved sludge simulants resulted in similar trends in solution corrosivity and metal corrodibility. The ratio of acid to sludge determined the evolution of the solution corrosivity and metal corrodibility. For the 20:1 acid to sludge ratio, the solution corrosivity increases with time, whereas for the 50:1 ratio the solution corrosivity decreases with time. For both 20:1 and 50:1 sludge ratios, metal corrodibility increases with time. This suggests that the ferric oxalate film that forms on the steel surface degrades with time and offers less protection.

Corrosion rates measured by the passive coupon tests for the nitric acid/oxalic acid blend are significantly greater than those from the LPR measurements. This result may be due to simultaneous electrochemical and chemical dissolution of the steel. As a result, the results from the LPR tests would not be useful without significant correction. Further testing would be necessary to determine if chemical dissolution were a factor.

The corrosivity of sodium permanganate in four proposed cleaning solutions, 1 nitric acid solution and three sodium hydroxide solutions, was studied by electrochemical methods at room temperature. The corrosion rates were less aggressive than in the nitric acid/oxalic acid tests. The most aggressive solution was the 3M sodium hydroxide with permanganate which had corrosion rates greater than 20 mils per year based on linear polarization tests. The cyclic potentiodynamic polarization tests indicated that primarily general corrosion occurs. Passive coupon tests need to be performed in these solutions to ensure that chemical dissolution of the metal is not occurring simultaneously.

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