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Performance of Vapor Corrosion Inhibitors on Mitigating Corrosion of Secondary Liner in Double Shell Storage Tanks at Hanford

Pavan K. Shukla, Roderick E. Fuentes, Bruce J. Wiersma
Savannah River National Laboratory®
Aiken, South Carolina, 29808
USA

and

Crystal Girardot, Natalie Young, and Theodore Venetz
Washington River Protection Solutions
2425 Stevens Center Pl
Richland, Washington, 99352
USA

ABSTRACT

Hanford high-level waste storage site has 28 carbon-steel double shell storage tanks (DSTs), where a secondary shell surrounds the primary shell. The bottom plate of the secondary shell rests on a concrete pad with grooves for leak detection. There have been instances of corrosion on the secondary liner bottom plate in contact with the concrete pad where accumulation of the groundwater solution in the pad grooves has caused corrosion. In previous studies, several commercially available vapor corrosion inhibitors (VCIs) were tested for their ability to mitigate concrete-pad side corrosion of the secondary liner bottom. The studies involved either immersing or placing coupons in vapor space of the groundwater solution dosed with VCIs. However, most likely field conditions of the tank bottom are envisioned to have weathering which would include accumulation of corrosion products in corroded areas. A study was conducted to determine effectiveness of the VCIs in mitigating corrosion of weathered carbon-steel coupons. Many sets of coupons were exposed to the groundwater solution for several months in three separate corrosion tests. Half of the coupons were extracted after approximately two months of exposure and analyzed for corrosion. The remaining sets of coupons were exposed to the commercially available VCIs for an additional two months by adding the VCIs to the groundwater solutions in the tests. The remaining coupons were extracted after completion of the tests and analyzed for pitting corrosion. Statistical analysis of the pre- and post-VCI exposed coupons' corrosion rates were compared to evaluate effectiveness of the VCIs for mitigating corrosion on the weathered coupons. The paper presents experimental data and results on the effectiveness of VCIs in mitigating corrosion on weathered carbon-steel surfaces.

Key words: Vapor Corrosion Inhibitors, Hanford, Double Shell Tanks, Bottom Plate, Pitting Corrosion.

INTRODUCTION

High-level radioactive waste was generated when reprocessing of spent nuclear fuel was carried out at Hanford. The waste has been stored in several single- and double-shell tanks. There are 28 double shell tanks (DSTs) at Hanford. Each DST consists of a primary shell (inner) surrounded by secondary (outer) liner. The secondary

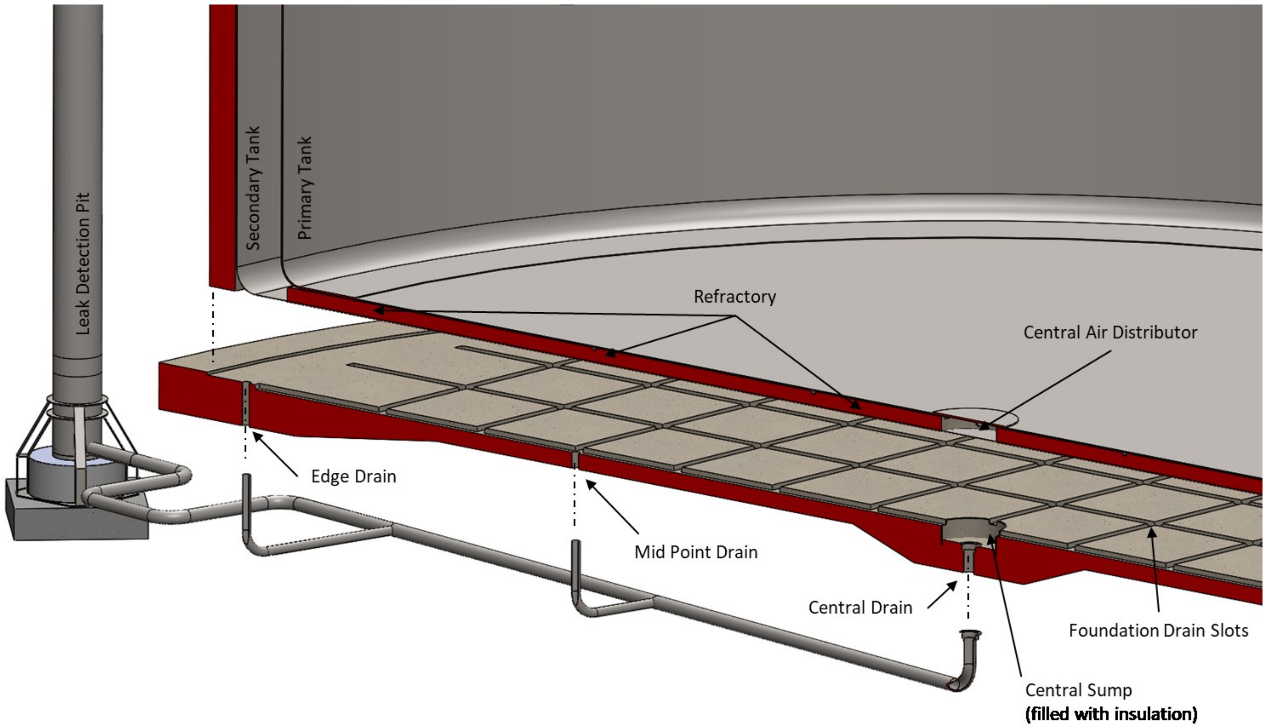


Figure 1: Schematic of a double shell tank depicting primary and secondary tank shells, concrete foundation, and drain slots.

liner rests on a concrete foundation. The schematic diagram, which shows the concrete foundation and drain slots, is presented in Figure 1.

Water is known to accumulate in the drain slots, and corrode the exterior of the secondary liner. Evidence of corrosion has been detected via ultrasonic inspections of the annulus floor. The inspection is confined to the annular space between the primary and secondary tank shells; there is a concern that corrosion is widespread on the underside of the bottom plate. Since the water level can vary in the drain slots based on accumulation, corrosion could be caused by direct contact with the accumulated water; when the leak detection pit (LDP) water level is below the structural limit, vapor space corrosion (VSC) could also occur. Accumulated water is drained through the sumps in the LDP. The LDP water was analyzed for its constituents, and two simulants were developed considering the chemical composition range of the accumulated water. The simulants are identified as leak detection pit and ground water (GW); compositions are listed in Table 1. The pH of both simulants was adjusted using sodium carbonate and acetic acid to 7.6 after preparation. A previous study established that GW simulant is more corrosive than the leak detection pit, therefore GW was used in the VCI effectiveness study.[1]

Table 1
Composition of the Leak Detection Pit and Ground Water Simulants

Source chemical	Concentration (M)	
	Leak Detection Pit	Ground Water
Sodium bicarbonate	1.12×10^{-3}	1.75×10^{-3}
Calcium hydroxide	1.21×10^{-4}	1.50×10^{-3}
Potassium nitrate	6.75×10^{-5}	2.40×10^{-4}
Magnesium Nitrate, 6hydrate	1.52×10^{-5}	−
Strontium Nitrate	4.04×10^{-6}	2.87×10^{-6}
Sodium sulfate	1.83×10^{-6}	−
Ferric sulfate	−	6.25×10^{-4}
Sodium Metasilicate, 5hydrate	4.57×10^{-5}	6.00×10^{-4}
Ferric chloride	2.67×10^{-6}	7.67×10^{-5}7E-05
Manganese Chloride	−	3.100E-04
Acetic Acid	3.00×10^{-4}	3.000E-04
Adjusted pH	7.6	7.6

Laboratory experiments were conducted to address the concerns of both immersed-phase corrosion and VSC of the tank steel exposed to GW simulant. Vapor corrosion inhibitors (VCIs) were also tested to determine their efficacy in mitigating the corrosion on pre-corroded surfaces. VCIs have been used for corrosion mitigation in numerous applications for decades. However, VCIs' application for above ground storage tank bottom corrosion control is recent, and several studies have documented effectiveness of VCIs for tank bottom plate corrosion control.[2 3 4 5]

EXPERIMENTAL

Three experiments were set up using GW simulants as electrolytes. VCIs were added to each experiment mid-course. Two VCIs were used and are identified as VCI-A[(1)] and VCI-B[(2)]. The experiments included (i) initially GW simulant, and then 10% of the recommended dosage of VCI-B after 2 months, (ii) initially GW simulant, and then 100% of the recommended dosage of VCI-A after 2 months, and (ii) initially GW simulant, and then 100% of the recommended dosage of VCI-B after 2 months.

Disk coupons, machined from a legacy carbon steel plate, were used in the experiments. The legacy carbon steel is based on specifications of Association of American Railroads[(3)] Tank Car (AAR TC 128) steel, and its chemistry and microstructure are similar to the vintage steel from which the tanks were fabricated UNS K02401 (i.e., American Society for Testing and Materials (ASTM) [(4)] A515 Grade 60 carbon steel). The chemical composition of the legacy carbon steel is listed in Table 2. All elemental compositions except for Mn and Si meet the ASTM A515 Grade 60 specification. The coupons were 25 mm (1 inch) diameter with a thickness of 3 mm (0.125 inch) and polished to a 600 grit finish. The coupons were potted in a mold prepared with a two-part clear epoxy solution (EpoKwick® from Buehler) so that one face of the coupon was exposed to the test electrolyte. An image showing two test coupons is presented in Figure 2. A wire was attached to the potted face of each coupon; the coupons were hung at various heights with the aid of the attached wires.

Table 2
Chemical Composition of AAR TC 128 Steel (wt.%)

	C	Mn	P	S	Si	Fe
Specification	0.24 (max.)	0.9 (max.)	0.035 (max.)	0.04 (max.)	0.13 to 0.33	Balance
Measured	0.212	1.029	0.012	0.013	0.061	Balance

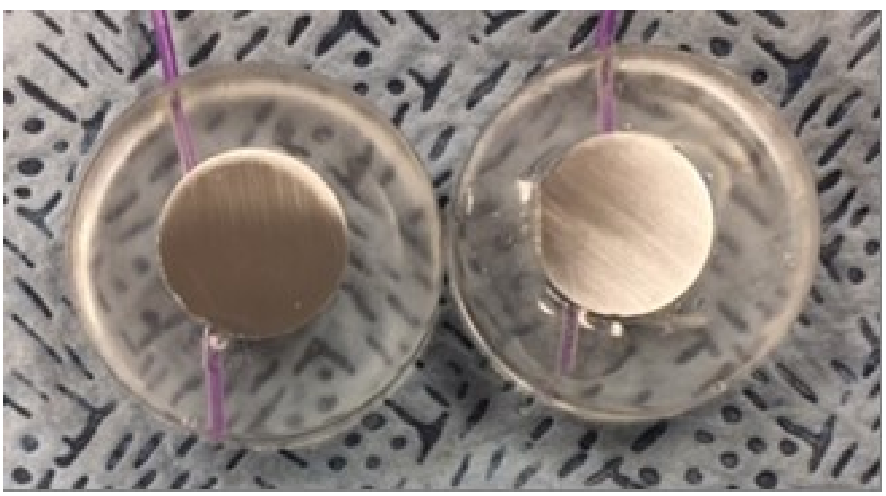


Figure 2: Image of the coupons used in the study. A purple wire was attached to each coupon, and coupons were suspended above the liquid interface using the wires.

A glass vessel of dimensions 1.0 m (3.3 ft) tall and 14 cm (5.5 inch) diameter was used for each experiment. Approximately 1.25 L of GW simulant was added to a vessel for each experiment. Each vessel has a water jacket

(1) VCI-A was VpCI-337 manufactured by Cortec Corporation.
(2) VCI-B was a mixture of VpCI-649 MF and VpCI-609, both manufactured by Cortec Corporation.
(3) American Association of Railroads, 425 3rd Street SW, Washington, DC 20024
(4) ASTM International, 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959

around the simulant holding area which was used to circulate warm water to maintain the simulant temperature at 45 ± 2 °C. Each vessel also has several ports, which were used to insert thermocouples and electrical resistance (ER) probes. An image showing the two vessels used is presented in Figure 3(a). Coupons were exposed to the electrolyte and vapors of the electrolyte in each experiment by suspending them using a rod shown in Figure 3(b). The rods holding the coupons were placed inside the vessels. Several coupons were immersed in each vessel, and coupons were also placed in vapor space of each vessel. The vapor space coupons were placed at several height levels with respect to the electrolyte using the rods. The coupons' positions, with respect to electrolyte in each vessel, simulated different vapor space conditions and water levels in the drain slots. These levels, representing the drain slot characteristics and its position with respect to the bottom, are described:

Level 1: Bottom or low level. Coupons were dipped in the simulant for five minutes prior to testing. The coupons were hung at the bottom fixed ring of the rod shown in Figure 3(b). These coupons were suspended approximately 25 mm (1 inch) above the liquid level of the simulant. Every two weeks, the coupons were lowered into the simulant for 5 minutes. This level is representative of the situation when the secondary liner bottom plate experienced periodic wetting/drying.

Level 2: Intermediate or middle level. Coupons were dipped in the simulant for five minutes prior to testing. The coupons were hung at the middle-fixed ring approximately 46 cm (18 inch) above the liquid simulant in each vessel. This level is representative of a vapor space region of the secondary liner bottom that at one time was exposed to water, but has infrequent or no contact with the water. However, this region is exposed to the humidified air.

Level 3: Top or high level. This set of coupons was not exposed to the solution prior to testing. The coupons were suspended approximately 91.4 cm (36 inch) above the simulant. This level is representative of the secondary liner bottom plate region that is only exposed to the humidified air and any volatile species from the solution.

(a) (b)

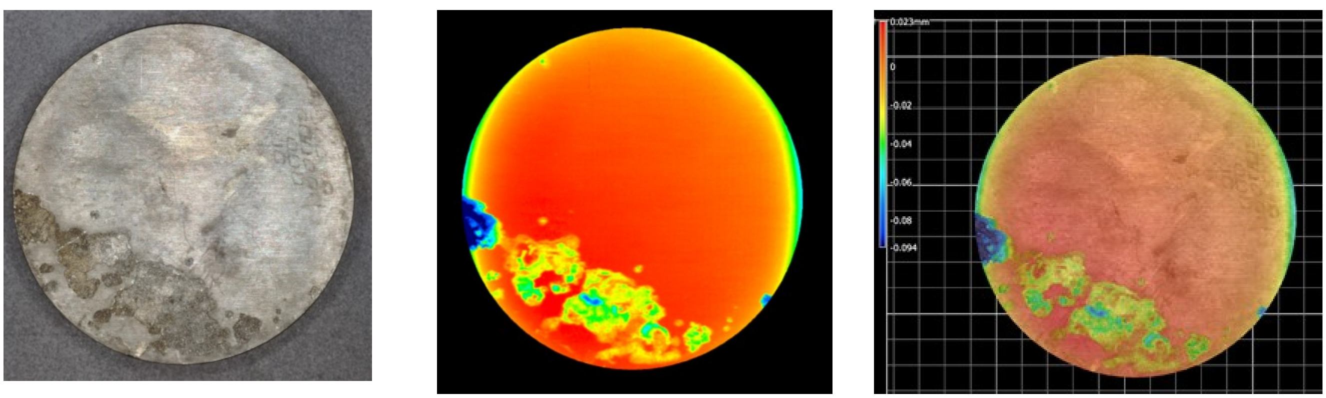
Figure 3: Images of the (a) experimental configuration, and (b) steel rod to suspend the coupons inside the vessel containing electrolyte.

Description of the vessels for each electrolyte is provided in Table 3. Electrical Resistance (ER) probes were placed in Vessels 7 and 8; placement positions are detailed in Table 3. ER probe data were collected periodically. Coupons were removed after several months of exposure, cleaned with Clarke's solution[6] to remove corrosion products, and measured for weight losses.

Table 3
Electrolyte Description, Vessel Identification, and Coupons and ER Probe Information

Electrolyte	Corrosion Cell	Notes
Initially GW simulant, and then 10% of the recommended dosage of VCI-B after 2 months	Vessel 6	• 6 coupons each in immersed, Level 1, Level 2, and Level 3 positions, total 24 coupons. • No ER probes
Initially GW simulant, and then 100% of the recommended dosage of VCI-A after 2 months	Vessel 7	• 6 coupons each in immersed, Level 1, Level 2, and Level 3 positions, total 24 coupons. • One ER probe in immersed and another at Level 1 position
Initially GW simulant, and then 100% of the recommended dosage of VCI-B after 2 months	Vessel 8	• 6 coupons each in immersed, Level 1, Level 2, and Level 3 positions, total 24 coupons. • One ER probe in immersed and another at Level 1 position

Coupons were extracted mid-course and after completion of the experiments, and after cleaning with Clarke's solution, coupons were analyzed for material wastage. Pitting and patch-like corrosion occurred on several coupons. Coupon surfaces were profiled and the deepest pit in each coupon was measured from the surface profile data. In addition, each coupon's mass change was also recorded. A representative image of an exposed coupon and its profiled surface are presented in Figure 4.



(a) (b) (c)

Figure 4: (a) Image of a representative coupon, (b) false color map, and (c) digital profile of the coupon surface

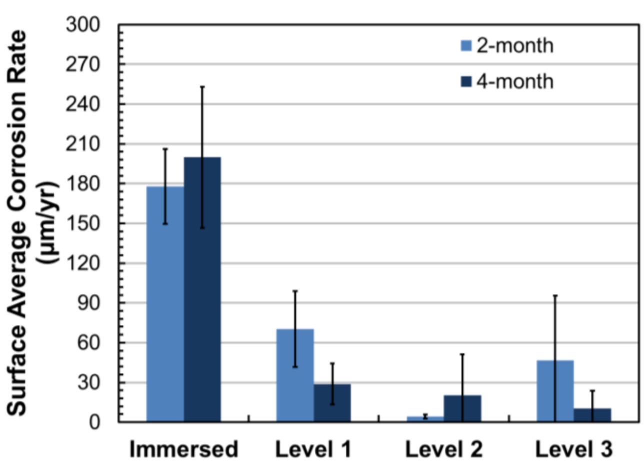
EXPERIMENTAL DATA AND RESULTS

The corrosion rate data for the coupons in Vessel 6 are listed in Table 4. The table has corrosion rate data, including surface average and pitting corrosion rates for the coupons exposed to GW and then GW plus 10 % recommended dosage of VCI-B. The data are for coupons exposed for two and four months; the coupons exposed for two and four months are referred as 2-month and 4-month coupons, respectively, hereafter. The 2-month coupons were exposed to GW simulant whereas 4-month coupons were exposed to GW plus 10% VCI-B dosage. Table 4 data also include averages of surface average and pitting corrosion rates and corresponding standard deviations; the corrosion rates and standard deviations are presented in Figure 5. As seen in the figure, the average of the surface average corrosion rate of the immersed 4-month coupon is higher than the 2-month data. The same is observed in the surface average and pitting corrosion rates of the coupons that were at Level 2. This indicates that 10% VCI-B recommended dosage may not be enough to arrest the corrosion initiated by the GW simulant.

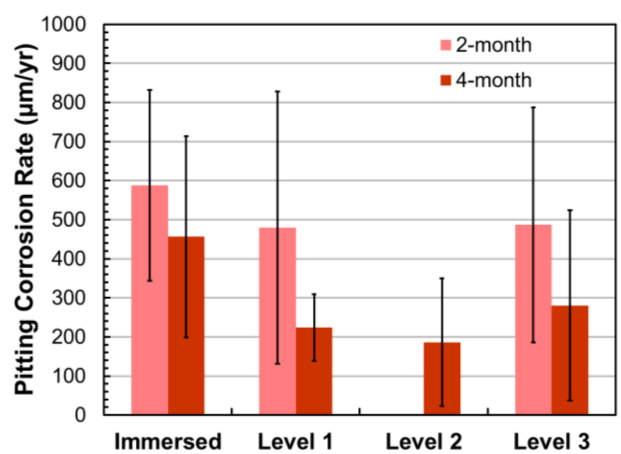
Table 4
Vessel 6 Coupon Corrosion Data

Corrosion Type	Corrosion Rate (µm/yr)*							
	Immersed		Level 1		Level 2		Level 3	
	2-month	4-month	2-month	4-month	2-month	4-month	2-month	4-month
Surface Average Corrosion	183	241	102	15	5	56	38	5
	147	218	64	25	3	3	99	0
	203	140	46	46	5	3	3	25
Average** ± std***	178 ± 28	200 ± 53	70 ± 29	29 ± 16	4 ± 2	20 ± 31	49 ± 49	10 ± 13
Pitting Corrosion	653	716	401	140	0	251	719	439
	318	452	861	310	0	307	594	0
	792	201	178	221	0	0	147	401
Average** ± std***	588 ± 244	456 ± 258	480 ± 348	224 ± 85	0 ± 0	186 ± 164	487 ± 301	280 ± 243

*25 µm/yr = 1 mil/yr = 1 mpy
**Average values are calculated for 3 coupons
***std denotes standard deviation of the corrosion rate data used to calculate the average



(a)



(b)

Figure 5: Average of (a) surface average, and (b) pitting corrosion rates for coupons in Vessel 6. The black line in each bar represents the standard deviation.

The corrosion rate data for the coupons in Vessels 7 and 8 are listed in Tables 5 and 6, respectively. Table 5 has corrosion rate data, including surface average and pitting corrosion rate, for the GW (2-month) and then GW plus 100% recommended dosage of VCI-A (4-month), and Table 8 has the data for the GW (2-month) and then GW plus 100% recommended dosage of VCI-B (4-month). The data are for the 2-month and 4-month coupons. The 2-month coupons were exposed to GW simulant whereas 4-month coupons were exposed to GW plus 100% VCI dosages. The table data also include average of surface average and pitting corrosion rates corresponding standard deviation. These average values and standard deviations of the averages are presented in Figures 6 and 7. Vessels 7 and 8 also had ER probes, one immersed in electrolyte and another one just above the electrolyte interface in each vessel. The ER probe data are presented in Figure 8.

Table 5
Vessel 7 Coupon Corrosion Data

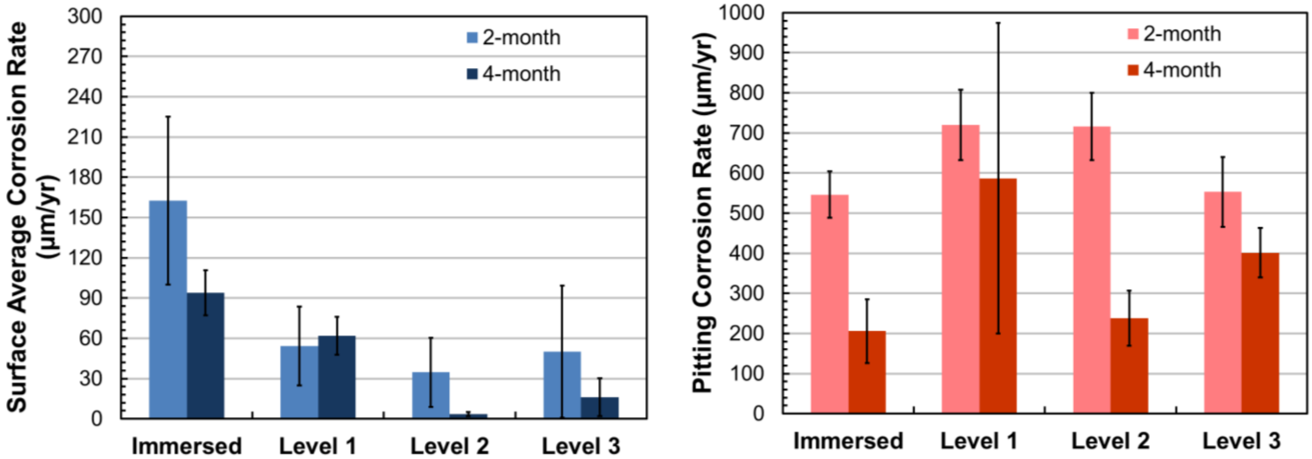
Corrosion Type	Corrosion Rate (µm/yr)*							
	Immersed		Level 1		Level 2		Level 3	
	2-month	4-month	2-month	4-month	2-month	4-month	2-month	4-month
Surface Average Corrosion	170	79	69	48	5	3	107	25
	221	112	20	61	48	3	20	0
	97	91	74	76	51	5	23	23
Average** ± std***	163 ± 63	94 ± 17	54 ± 29	62 ± 14	35 ± 26	3 ± 2	50 ± 49	16 ± 14
Pitting Corrosion	569	251	757	368	813	216	516	330
	589	251	782	358	673	183	653	434
	480	114	620	1034	663	315	490	439
Average** ± std***	546 ± 58	206 ± 79	720 ± 87	587 ± 387	716 ± 84	238 ± 69	553 ± 87	401 ± 62

*25 µm/yr = 1 mil/yr = 1 mpy
**Average values are calculated for 3 coupons
***std denotes standard deviation of the corrosion rate data used to calculate the average

Table 6
Vessel 8 Coupon Corrosion Data

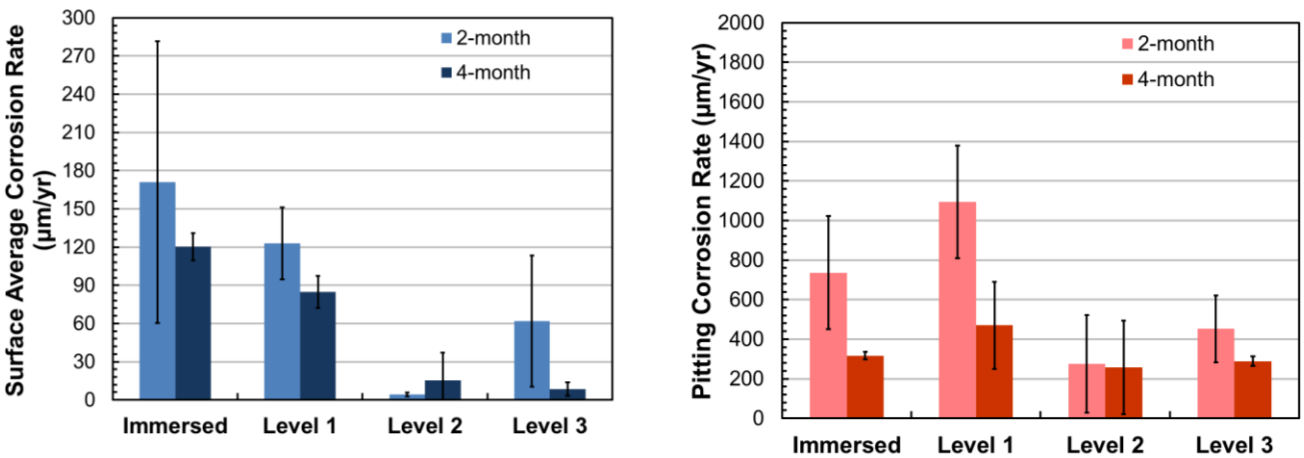
Corrosion Type	Corrosion Rate (µm/yr)*							
	Immersed		Level 1		Level 2		Level 3	
	2-month	4-month	2-month	4-month	2-month	4-month	2-month	4-month
Surface Average Corrosion	236	112	155	76	3	41	89	3
	43	117	102	99	5	3	3	13
	234	132	112	79	5	3	94	10
Average** ± std***	171 ± 111	120 ± 11	123 ± 28	85 ± 13	4 ± 2	15 ± 22	62 ± 51	9 ± 5
Pitting Corrosion	747	338	1351	620	0	310	445	269
	445	310	1143	218	475	0	287	315
	1016	302	787	572	351	462	625	282
Average** ± std***	736 ± 286	317 ± 19	1094 ± 285	470 ± 219	276 ± 246	257 ± 236	452 ± 169	289 ± 24

*25 µm/yr = 1 mil/yr = 1 mpy
**Average values are calculated for 3 coupons
***std denotes standard deviation of the corrosion rate data used to calculate the average



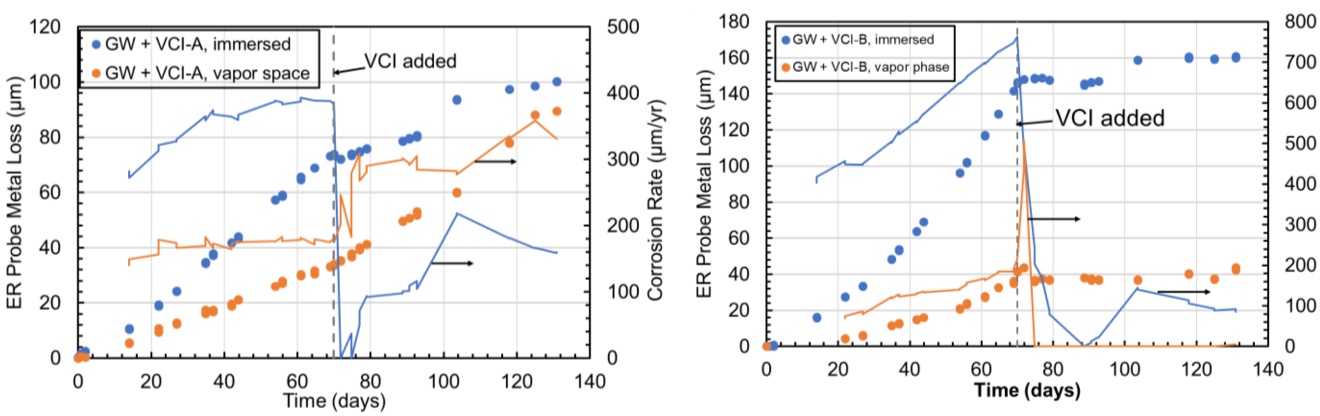
(a) (b)

Figure 6: Average of (a) surface average, and (b) pitting corrosion rates for coupons in Vessel 7. The black line in each bar represents the standard deviation.



(a) (b)

Figure 7: Average of (a) surface average, and (b) pitting corrosion rates for coupons in Vessel 8. The black line in each bar represents the standard deviation.



(a) (b)

Figure 8: (a) Vessel 7 and (b) Vessel 8 ER probe data metal loss and corresponding corrosion rates. The ER Probe data is represented by filled circles, and corrosion rates by solid lines.

Vessel 7 corrosion rate data in Figure 6(a) show that VCI-A dosage lowered the corrosion rate except for the Level 1 coupons. Two ER probes were also placed in Vessel 7; one was immersed in the solution and another one at Level 1. The ER probe metal loss data and corresponding corrosion rates are presented in Figure 8(a). The Figure 6(a) corrosion rate data are consistent with the ER probe data in Figure 8(a). As per the ER probe data, the immersed coupons' corrosion rate should have decreased, and Level 1 coupons' corrosion rate should have increased after VCI-A addition. In Figure 6(a), the 4-month immersed coupon corrosion rate is lower than the 2-month coupon, and 4-month Level 1 coupon corrosion rate is higher than the 2-month coupon, and thus, a consistency is observed between the ER probe and coupon data. It is, however, noted that the pitting corrosion rate decreased for all four sets, i.e., immersed and Levels 1-3, of coupons after VCI-A addition as seen in Figure 6(b), indicating that addition of VCI-A helps arrest propagation of pitting corrosion.

Vessel 8 corrosion rate data in Figure 7(a) show that VCI-B dosage lowered the corrosion rate except for that of the Level 2 coupons. Two ER probes were also placed in Vessel 8; one was immersed in the solution and another one at Level 1. The ER probe metal loss data and corresponding corrosion rates are presented in Figure 8(b). The Figure 7(a) corrosion rate data are consistent with the ER probe data in Figure 8(b). As per the ER probe data, the immersed and Level 1 coupons' corrosion rate should have decreased after VCI-B addition. In Figure 7(a), the 4-month immersed and Level 1 coupons' corrosion rates are lower than the 2-month coupons, and thus, establish consistency between ER probe and coupon data. The addition of VCI-B also decreased pitting corrosion on the coupons, as seen in Figure 7(b). A decrease in pitting corrosion was observed for immersed and Levels 1, 2 and 3 coupons.

A statistical analysis was conducted to determine significance of the decrease in corrosion rate due to addition of 100% recommended dosages of VCI-A and VCI-B. The statistical method used was Student's t-test, which is based on the hypothesis that there is no statistically significant difference between the corrosion rates for the two electrolyte parameters used in the t-test—that is, that they are essentially identical to each other in terms of the coupon corrosion rates. The statistical result calculated by the test, P value, is the probability that the hypothesis is true. The higher the P-value, the greater the chance that the two sets of corrosion rates for the 2- and 4-month coupons are statistically similar. If the P-value is equal to or less than 0.05, it indicates that there is a less than 5% chance that the two sets of coupons have similar corrosion rates—that is, it means, with 95% confidence, that there is a statistically significant difference between the two 2- and 4-month coupons. The P-values are listed in Table 7.

Table 7
Student's t-Test P-values* for Comparison Between Coupons Before and After VCI Treatment

Corrosion Cell	Corrosion Type							
	Surface Average Corrosion				Pitting Corrosion			
	Immersed	Level 1	Level 2	Level 3	Immersed	Level 1	Level 2	Level 3
Vessel 6 (10% VCI-B)	0.57	0.11	0.46	0.32	0.56	0.33	0.19	0.41
Vessel 7 (100% VCI-A)	0.19	0.71	0.17	0.36	0.01	0.62	0.00	0.08
Vessel 8 (100% VCI-B)	0.51	0.13	0.48	0.21	0.13	0.04	0.93	0.23
*P-values of 0.05 or less indicate statistically significant differences with 95% confidence								

Pitting corrosion of the secondary shell is the main hazard for a tank failure, leading to breach of the secondary containment. The P-values in Table 7, obtained by statistical analysis of the pitting corrosion rates, are discussed. The P-values for Vessel 6 pitting corrosion are much higher than 0.05, indicating that there is high likelihood that pitting corrosion was not mitigated by the 10% VCI-B dosage. The P-values for Vessel 7 immersed and Level 2 coupons are less than 0.05, and the P-value for Level 3 is 0.08, slightly above 0.05. This indicates with 95% confidence that immersed and Level 2 coupons' pitting corrosion were mitigated, however, there is only 92% confidence that Level 3 coupons' pitting corrosion was mitigated. In addition, Vessel 7 Level 1 coupons' pitting corrosion rates were not mitigated, as indicated by P-value of 0.62. In Vessel 8, the P-value for the Level 1 coupons is 0.04; there is 95% confidence that Level 1 coupons' pitting corrosion was mitigated. The P-values for immersed and Level 3 coupons are 0.13 and 0.23, respectively, this indicates that the confidence levels in the reduction are 87 and 77% for the immersed and Level 3 coupons, respectively. Vessel 8 Level 2 coupons' pitting corrosion rates were not mitigated, as indicated by the P-value of 0.93.

CONCLUSION

A study was conducted to determine effectiveness of two commercially available vapor corrosion inhibitors in mitigating corrosion on bottom side of the secondary liner of double shell tanks. VCIs were added during mid-course of experiments, i.e., after coupons have experienced corrosion in the untreated simulant. Three tests were conducted using VCI-A and VCI-B. The first two tests were conducted using 100% recommended dosages of VCI-A and VCI-B. The third test was conducted at 10% of the recommended dosage of VCI-B. Following conclusions are made from the experimental data and results:

- The corrosion rate data indicated that 10% of the recommended dosage is not enough in mitigating corrosion. This observation is consistent with a prior study which also concluded that VCIs' effectiveness vanish at 10% of the recommended dosages for the aboveground tank bottom underside application.[5]

- The data also showed that 100% recommended dosages of VCI-A and VCI-B mitigated pitting corrosion of weathered coupons. Specifically, VCI-A mitigated pitting corrosion in immersed, Level 2, and Level 3 coupons, whereas VCI-B mitigated pitting corrosion in immersed, Level 1, and Level 3 coupons.

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