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Inhibition and Control of Microbiologically Influenced Corrosion in Oilfield Materials

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Abstract: Most engineering systems experience unexpected problems, premature failures and costly repairs due to damages wrecked by Microbiologically Influenced Corrosion (MIC). This research is aim at establishing the influence of certain microorganisms in increasing corrosion rate in steel coupons, characterizing and isolating the microbial strains associated with the accelerated corrosion in waterlogged clay soil area and determining the corrosion inhibitors that can effectively reduce the MIC, acting as a biocide instead of a nutrient source. Microbial count tests in both dry and waterlogged soils and Biochemical tests on isolates were conducted. Results show that effective prevention and control of MIC can be achieved through proper characterization of the microorganisms involved and understanding their specific roles in the clay soil environment. The Inhibitive Efficiency of the three inhibitors used in this study decreased in the following order: Copper (II) Chloride, Glutaraldehyde and sodium molybdate with $CuCl_2$ giving 78.9% efficiency. The effective use of biocides helps reduce the equipment damage by lowering the rate of corrosion attack and thereby saving maintenance cost such as repair costs, lost production and lost sales.

Key words: Corrosion, inhibition, equipment, waterlogged clay, micro-organisms, Nigeria

INTRODUCTION

Corrosion of oilfield equipment and piping can lead to potentially hazardous system malfunctions as well as costly damage and repair costs. Microbiologically Influenced Corrosion (MIC) can rapidly accelerate corrosive growth leading to these problems even in buildings <5 years old. Unfortunately, inspections for MIC and Corrosion generally are often overlooked until expensive problems such as damaging leaks occur or the corrosion is so prevalent that large areas of the system have to be replaced. This corrective maintenance approach is a retro-active strategy. The task of the maintenance team in this scenario is usually to effect repairs as soon as possible in a reactionary mood. Microbiologically influenced corrosion is the term used for corrosion influenced by microbes.

The primary concern is that the influence of these microbes is often an extremely accelerated rate of corrosion. In a general sense, MIC fall into one of two groups based upon their oxygen requirements; one being aerobic such as sulfur oxidizing bacteria and the other being anaerobic, such as sulfate reducing bacteria (Huggins, 2010). There is presently no indication that MIC is confined to any specific geographical area. MIC almost always occurs concurrently with other corrosion mechanisms and it is virtually impossible to separate them. Global mentioned that this is in part due to the fact that microbes help create conditions under which other corrosion mechanisms can occur such as crevice corrosion, pitting and under-deposit corrosion.

Hundreds of different microbe types have been found to be capable of thriving in what are considered to be extraordinarily hostile environments. According to Rice and Wilkes (1991) and Chung *et al.* (1995) these microbes have to become established first, since effective prevention and control of MIC involves an underlying knowledge of the microorganisms responsible for increased corrosion rates as well as methods that can be implemented to detect and prevent microbial growth. Microorganisms have long been known to influence corrosion, causing through-wall corrosion of piping and heat exchanger tubes 10-1000 times faster than normal (Licina, 2001).

Wet soils containing clay have played a major role in the occurrence of underground MIC problems. Formation of a biofilm creates a microenvironment that is dramatically different from the bulk surroundings. Pope and Zintel (1988) discussed that changes in pH, dissolved oxygen, organic and inorganic compounds in the microenvironment can lead to electrochemical reactions which increase corrosion rates.

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Table 1: Common microorganisms involved with microbiologically influenced corrosion

Genus or species	pH	Temp (°F)	Oxygen requirement	Metals affected	Metabolic
Desulfovibro	4-8	50-105	Anaerobic	Iron and steel, stainless steels, aluminium, zinc, copper alloyed	Use H_2 in reducing SO_4^{2-} to S^{2-} and H_2S; promote formation of sulfide films
Desulfotomaculum	6-8	50-105 (some 115-165)	Anaerobic	Iron and steel, stainless steel	Reduce SO_4^{2-} to S^{2-} and H_2S
Desulfomonas	-	50-105	Anaerobic	Iron and steel	Reduce SO_2^{2-} to S^{2-} and H_2S
Sphaerotilus	7-10	70-105	Aerobic	Iron and steel, stainless steel	Oxidizes Fe^{2+} to Fe^{3+} and manganious to manganic ions; promote tubercle formation
Pseudomonas	4-9	70-105	Aerobic	Iron and steel, stainless steel	Some strains reduce Fe^{3+} to Fe^{2+}
Acidithiobcallus thioxidans	0.5-8	50-105	Aerobic	Iron and steel, copper alloys, concrete	Oxidizes sulfur and sulfides to form H_2SO_4 damages protective coatings

Microorganisms may also produce hydrogen which can cause damage in metals. Most microorganisms form an extracellular membrane which protects the organism from toxic chemicals and allows nutrients to filter through. MIC problems have been widely documented in piping systems, storage tanks, cooling towers and aquatic structures. Mild steels are widely used in these applications due to their low cost but are some of the most readily corroded metals. Mild steels are normally coated for corrosion protection while cathodic protection may also be used for selected applications Galvanization (zinc coating) is commonly used to protect steel in atmospheric environments.

Bituminous coal tar and asphalt dip coatings are often used on the exterior of buried pipelines and tanks while polymeric coatings are used for atmospheric and water environments. However, biofilms tend to form at flaws in the coating surfaces. Furthermore, acid producing microorganisms have been found to dissolve zinc and some polymeric coatings (Mansfeld and Little, 1990). Videla (2002) elaborates on biocorrosion and stated that microorganisms cause debonding of coatings from the underlying metal.

Delamination of the coating, in turn, creates an ideal environment for further microbial growth. Poor quality water systems and components with areas that accumulate stagnant water and debris are prone to MIC. This has been seen to occur in underground pipes that have been left unused for periods of time. Dexter (2003) discussed several groups of microorganisms that contribute to MIC as shown in Table 1.

The exterior of buried pipelines (water, wastewater) and tanks especially in wet clay environments are persistently being exposed to microorganisms which influence corrosion (Videla, 2002). It is possible that corrosion inhibitors will have biocidal effects on bacteria. Bacteria are also well-known for their ability to oxidize a wide variety of chemicals and use them as nutrients (sources of carbon and nitrogen) under a variety of conditions (Hamilton, 2003).

Therefore, corrosion inhibitors could act as a nutrient source and enhance populations of bacteria. It would be an advantage to know if the corrosion inhibitor is acting as a nutrient source, a biocide or indeed if there is any effect at all (Scott, 2004).

Inhibition of microbiologically influenced corrosion

Microbial inhibition of corrosion: Corrosion inhibition is the slowing down of the corrosion reaction and is usually performed by substances (corrosion inhibitors) that when added in small amounts to a given environment, decrease the rate of attack by this environment on a metal.

Microorganisms can induce or accelerate drastically the electrochemical conditions at the metal-solution interface or even inhibit corrosion. Microbial effects that could enhance corrosion include the stimulation of the anodic reaction by acidic metabolites or the cathodic reaction by microbial production of a new alternative cathodic reactant (e.g., H_2S), the microbial breakdown of protective films and the increase in conductivity of the liquid environment (Videla, 1996).

Microorganisms can contribute to corrosion inhibition by different mechanisms: neutralizing the action of corrosive substances present in the environment; forming protective films or stabilizing a pre-existing protective film on a metal and inducing a decrease in the medium corrosiveness. Videla (2003) discuss general key features of microbial inhibition of corrosion can be summarized as follows: MIC and its counter-process, microbial inhibition of corrosion are rarely linked to a single mechanism of a single species of microorganisms. Either the corrosive or the inhibitory actions of bacteria develop on biofilmed metal surfaces where complex biofilm/protective films occur.

Microbial corrosion inhibition is frequently accomplished through: a decrease in the cathodic rate by microbial consumption of a cathodic reactant (e.g., oxygen consumption by respiratory activity), decreasing the medium aggressiveness in restricted areas of the metal solution interface (e.g., by neutralizing acidity) and providing or stabilizing protective films on the metal (e.g., biofilm exopolymers with metal-binding capacity). Finally, a proper understanding of the identity and role of microbial contaminants in the specific environment of a

Table 2: Biocides properties and usual concentrations (Zou, 2007)

Biocides	Properties	Usual conc. (mg L^{-1})
Chlorine	Effective against bacteria and algae; oxidixing; pH dependent	0.1-0.2 (continuous treatment)
Chlorine dioxide	Against bacteria, in a lesser extent against fungi and algae; oxidixing; pH-independent	0.1-1.0
Bromine	Effective against bacteria and algae; oxidizing; wide pH range	0.05-0.1
Ozone	Effective against bacteria and biofilms; oxidizing; pH-dependent	0.2-0.5
Isothiazolones	Effective against bacteria, algae and biofilms; non-oxidizing; pH-independent	0.9-10
Glutaradehyde	Effective against bacteria, algae, fungi and biofilms; non-oxidizing; wide pH range	10-70
QUATs[a]	Effective against bacteria and algae; non-oxidizing; surface activity	8-35
THPS[b]	Effective against bacteria, algae and fungi; low environmental toxicity; specific action against sulfate-reducing bacteria	-

[a]Quarternary ammonium compounds; [b]Tetrakis-hydroximethyl phosphonium sulphate

metal surface may be exploited to induce corrosion inhibition by bacteria as a useful tool to prevent frequent MIC effects encountered in practice (Javaherdashti, 2008).

MIC prevention and control: One of the classic concepts for maintaining an industrial system free of the deleterious effects of MIC is to keep the system clean. Physical methods include flushing which is perhaps the most simple although of limited efficacy. A special case is the use of flushing supported by cleaners or jointly with chemical agents that induce biofilm detachment. Abrasive or non-abrasive sponge balls are frequently employed in industry. However, abrasive sponge balls can damage protective passive films and non-abrasive sponge balls are not very effective with thick biofilms. The most common chemical method for controlling biocorrosion in systems is the use of biocides.

These can be either oxidizing or non-oxidizing biocides. Chlorine, ozone and bromine are three typical oxiding agents of industrial use. Non-oxidizing biocides are reported to be more effective than oxidizing biocides for overall control of algae, fungi and bacteria as they are more persistent and many of them are pH independent. Combinations of oxidizing and oxidizing biocides or of two non-oxidizing biocides are often used to optimize the effect of microorganisms. Typical biocides of the second type are formaldehyde, glutaraldehyde, isothiazolones and quaternary ammonia compounds (Table 2).

MATERIALS AND METHODS

Corrosion rate determination for the uninhibited mild steel coupons in dry and water-logged clay soil samples: Dry and water-logged clay soil samples from Abak, Akwa-Ibom state of Nigeria were used in this experiment. Prior to the experiment, the coupons were abraded to get rid of superficial rust, cleaned with acetone to remove any trace of dirt and grease, dried in the dessicator and then weighed. Three Coupons were then planted into each of the clay soil samples (dry and water-logged) and left for a period of 14 days before the first set of coupons were withdrawn, rinsed in deionized water, allowed to dry at room temperature overnight in the dessicator and weighed afterwards. After an additional 7 days, the next set of coupons were withdrawn from both Clay Samples, cleaned and weighed for weight loss. The corrosion rates of the metal coupons were calculated in mils (thousandths of an inch) per year according to the following formula: the corrosion rate is calculated assuming uniform corrosion over the entire surface of the coupon:

$$\text{Corrosion rate} = \frac{wk}{AtD} \quad (1)$$

Where:

w = Weight loss (g)
k = 3450000 (corrosion rate const in mils per year)
t = Time of exposure (h)
A = Expanded surface area (cm^2)
D = Density of metal coupon (g cm^{-3})

Isolation and characterization of the MIC-microorganisms in the water-logged clay soil sample: The last set of metal coupons were withdrawn and crushed without prior cleaning and conveyed to the laboratory in a polyethylene bag for analysis. The crushed samples to be cultured were serially diluted.

Serial dilution: A four-fold serial dilution was carried out on both samples. About 1 g of the crushed samples was measured into 9 mL of distilled water and shaken thoroughly to obtain a homogeneous mixture (aliquot). About 1 mL of the aliquot was then transferred into 9 mL of distilled water, shaken thoroughly until homogeneity was achieved. This process was repeated in 2 other successions to obtain a third diluents used for culturing. The mineral salt medium was carefully weighed, prepared and sterilized aseptically using an autoclave set at 180°C for I5 min. The medium was allowed to cool after sterilizing. About 1 mL of the final diluent was transferred into the sterile petridish containing the medium and swirled. After the medium solidified it was kept in an incubator set at 37°C for 20 days. After 20 days, the discrete colonies were observed and counted. The microbial count was measured in colony forming unit per

gram (CFU g^{-1}) and standardized according to the power of dilution (10^3). There after, isolates were picked and characterized through several biochemical tests. The biochemical tests were used to confirm the species and types of microorganisms located around the metal coupon buried in the water-logged clay soil sample.

Catalase test: About 0.5 mL of hydrogen peroxide solution was put in a test tube. The colony was carefully picked and rubbed onto the inside wall of the test tube above the surface of the hydrogen peroxide solution. The tube was capped and tilted to allow the hydrogen peroxide solution cover the colony.

Indole test: The filter paper was moistened with the spot test reagent and smeared with the isolated colony. The paper was observed for colour change.

Nitrate test: About 1 drop of the nitrate reagent was added to the inoculated plate and observed for any change.

Urease test: The urea disk was inoculated heavily with the isolate and left for 24 h to detect any colour change.

Sodium formate stimulation test: The isolated colony was picked and streaked on a plate with mineral medium containing sodium formate and incubated for 24 h to observe colony growth.

Desulfovibrin test: The colony was carefully picked onto a plate and 1 drop of 2 N NaOH was added. The plate was observed for any colour change.

SIM (Sodium Indole Motility) test: SIM media was inoculated with the isolated colony and observed for colour change.

Corrosion rate determination for the inhibited mild steel metal coupons in the water-logged clay soil sample: Materials used were:

- Mild steel metal coupons (7.5 cm long, 1.2 cm wide, 0.15 cm thick)
- Water-logged clay soil sample from Abak, Akwa-Ibom state
- De-ionized water
- Selective inhibitors (based on characterized microbes)
- Glutaraldehyde
- Dessicator
- Mass balance (adventure AR3130 model)

The already prepared metal coupons were weighed, coated with the selected inhibitor and then planted into the water-logged clay soil for 14 days after which they were weighed for weight loss and corrosion rate determination. Corrosion rate was calculated according to Eq. 1.

Inhibitive Efficiency (IE) determination of the selected inhibitors: After determining the corrosion rate of the inhibited mild steel coupons, the inhibitive efficiency of the different inhibitors used were found out as follows:

$$\text{Inhibitive Efficiency (IE)} = \frac{CR_{uninhibited} - CR_{inhibited}}{CR_{uninhibited}} \times 100\% \quad (2)$$

Where:

$CR_{uninhibited}$ = Corrosion rate of the uninhibited coupon in water logged clay soil sample

$CR_{inhibited}$ = Corrosion rate of the inhibited coupon in water logged clay soil sample

RESULTS AND DISCUSSION

Comparing the corrosion rate results in Table 3 and 4, it is noticed that the corrosion rate of the uninhibited mild steel metal coupons in the water-logged clay soil sample is approximately five times more than that obtained for the uninhibited mild steel metal coupons buried in the dry clay soil sample.

These values suggest that the accelerated corrosion rate obtained for the uninhibited coupons in the water-logged clay soil sample must have been influenced by MIC-microorganisms. These results are not conclusive hence other tests were necessary.

Analysis of Table 5: The result in Table 5 establishes the earlier suspicion of the prevalence of MIC-microorganisms in the water-logged clay soil sample. The figures show that the microorganisms present in the water-logged clay soil is about seven times that obtained in the dry clay soil sample.

This corroborates the corrosion rate results outlined in Table 3 and 4 suggesting further that the accelerated corrosion rate noticed for the uninhibited mild steel metal coupons buried in the water-logged clay soil must have been microbiologically influenced. Hence, the observed microorganisms in the water-logged clay soil needs to be further characterized.

Table 3: Corrosion rates of the uninhibited mild steel metal coupon in the dry clay soil sample

Coupon ID	Density (g cm^{-3})	Initial weight (g)	Final weight (g)	Surface area (cm^2)	Exposure time (h)	Corrosion rate (mpy)
D1	7.85	9.502	9.491	20.61	336	0.70
D2	7.85	8.892	8.877	20.61	504	0.63

Table 4: Corrosion rates of the uninhibited mild steel metal coupon in the water-logged clay soil sample

Coupon ID	Density (g cm^{-3})	Initial weight (g)	Final weight (g)	Surface area (cm^2)	Exposure time (h)	Corrosion rate (mpy)
W1	7.85	9.401	9.339	20.61	336	3.93
W2	7.85	9.521	9.456	20.61	504	2.75

Table 5: Microbial count of the discrete colonies in the immediate surroundings of the buried uninhibited metal coupons in both clay soil samples

Clay soil sample	Microbial count (CFU g^{-1})	Standardization (CFU g^{-1})
Water-logged	264	2.64×10^5
Dry	36	0.36×10^5

Table 6: Biochemical tests result on isolates

Biological tests	Observation	Results
Catalase	No reaction occurred	-
Indole	No colour change	-
Nitrate	A red colouration occurred indicating the reduction of nitrate to nitrite	+
Urea	Colour change from yellow to pale-pink	+
F/F	Bacterial growth was stimulated by sodium formate after 24 h	+
Desulfoviridin	Red fluorescence was observed indicating the presence of desulfovibrin pigment	+
SIM	A black colour in the SIM media was observed indicating H_2S production	+

F/F-Sodium formate/sodium fumarate stimulation; SIM-Sulfide-indole-motility medium for detection of H_2S

Analysis of Table 6: Table 6 shows the result of the biochemical tests on one of the isolates. From the table, the isolate produced H_2S as seen from the SIM test. The urea test showed that the microorganism detected could breakdown urea to ammonia using the enzyme urease. The suspected microorganism grew in the presence of sodium formate.

The nitrate test was positive showing that the suspected microorganism reduced nitrate to nitrite and desulfovibrin pigments were also detected. According to William (2000) the suspected microorganism belongs to the genus *Desulfovibrio* sp. an SRB. Zuo (2007) showed that molybdenum-containing compounds have the ability to inhibit the growth of sulphur-reducing bacteria. Sani *et al*. (2001) studied the copper inhibition of growth of Desulfovibrio desulfuricans.

The toxicity of copper [Cu (II)] to Sulphate Reducing Bacteria (SRB) was studied using desulfovibrio desulfuricans in a medium developed specially to test metal. The result clearly showed significant Cu (II) toxicity to SRB at concentrations 100 times lower than previously reported. From the fore-going, the selected inhibitors include:

- Glutaraldehyde (0.1 M)
- Sodium Molybdate, SM (0.1 M)
- Different concentrations of Copper (II) Chloride (0.1, 0.2 and 0.3 g mL^{-1})

Analysis of Table 7: Glutaraldehyde, a general inhibitor was effective in reducing the corrosion rate of the mild steel metal coupon in the waterlogged clay soil sample. Sodium molybdate was not as effective as glutaraldehyde (Table 7).

Sodium molybdate, probably acted as a nutrient source with its sodium suspected to have reacted with trioxosulphate (iv) ions present in the soil water in the following way:

$$2Na^{+1} + SO_3^{-2} \rightarrow Na_2SO_3$$

Forming sodium sulphite which can be oxidized by the sulphur-reducing bacteria to sodium sulphate as shown below:

$$Na_2SO_3 + 0.5O_2 \rightarrow Na_2SO_4$$

The observed reduction in the corrosion rate on using Copper (II) Chloride as an inhibitor was due to the susceptibility of *Desulfovibrio* sp. to the antimicrobial agent. This indicates that copper (II) chloride is more of a biocide in this case than a nutrient source. Hence, in selecting an inhibitor for this system, copper (II) chloride will be most preferred.

Analysis of Table 8: The Inhibitive Efficiencies of each of the selected inhibitors are shown in Table 8. The ability of the selected inhibitors to reduce the corrosion rate in the waterlogged clay soil decreased in the following order: copper (II) chloride, glutaraldehyde and sodium molybdate.

Increasing the concentration of the copper (II) chloride from 0.1-0.2 g mL^{-1} led to an increase in the effectiveness of inhibition of $CuCl_2$. No significant change in the corrosion rate was observed with a further increase in the concentration of $CuCl_2$ from 0.2-0.3 g mL^{-1}. Nevertheless, $CuCl_2$ (the inhibitor chosen based on characterization) researched effectively as a biocide in this system having the highest inhibitive efficiency.

Table 7: Ability of the various selected compounds to inhibit mic in the water-logged clay soil

Coupon ID	Density (g cm^{-3})	Inhibitor used	Initial weight (g)	Final weight (g)	Expended area (cm^2)	Exposure time (h)	Corrosion rate (mpy)
1	7.85	Glutaraldehyde (0.1 M)	9.216	9.191	20.61	336	1.59
2	7.85	Sodium molybdate (0.1 M)	9.392	9.359	20.61	336	2.09
3	7.85	Copper (II) chloride (0.10 g mL^{-1})	9.209	9.196	20.61	336	0.83
4	7.85	Copper (II) chloride (0.2 g mL^{-1})	9.500	9.488	20.61	336	0.76
5	7.85	Copper (II) chloride (0.3 g mL^{-1})	9.259	9.247	20.61	336	0.76

Table 8: Inhibitive efficiencies of the selected inhibitor (s)

Selected inhibitor	Inhibitive Efficiency (IE) (%)
Glutaraldehyde (0.1 m)	59.5
Sodium molybdate (0.1 m)	46.8
Copper (II) chloride (0.1 g mL^{-1})	78.9
Copper (II) chloride (0.2 g mL^{-1})	80.7
Copper (II) chloride (0.3 g mL^{-1})	90.7

CONCLUSION

The results have shown that efficient protection of the metal against microbial attack is achieved through proper pre-characterization of the MIC-microorganisms in the waterlogged clay soil which in turn assisted in the selection of a proper inhibitor in controlling MIC.

The use of the selected inhibitors based on pre-characterization resulted in a higher efficiency in the control of MIC in the selected water-logged clay soil sample compared to the use of glutaraldehyde, a general, widely used industrial MIC inhibitor.

It can therefore be concluded that pre-characterization of the MIC-microorganisms assists the engineer and scientist in the effective control of Microbiologically Induced Corrosion (MIC) by helping to select an inhibitor which will act as a biocide and not a nutrient source.

APPENDIX I

$$\text{Corrosion rate} = \frac{wk}{AtD} \quad (1)$$

Where:

w = Weight loss (g)
k = 3450000 (corrosion rate const. in mils per year)
t = Time of exposure (h)
A = Expanded surface area (cm^2)
D = Density of metal coupon (g cm^{-3})

The density of all the mild steel metal coupon samples = 7.85 g cm^{-3}

Expanded surface area (A) of the mild steel metal coupons = 2 (LB + LT + BT)

Where:

L = Length of the rectangular coupon
B = Breadth of the rectangular coupon
T = Thickness of the rectangular coupon

Therefore:

Surface area of the coupon samples = 2 [(7.5×1.2)+ (7.5×0.15)+(1.2×0.15)] = 20.61cm^2

$$\text{Corrosion rate for coupon Dl} = \frac{(9.502-9.491)\times 3450000}{20.61\times 336\times 7.85} = 0.70 \text{ mpy}$$

$$\text{Corrosion rate for coupon Wl} = \frac{(9.401-9.339)\times 3450000}{20.61\times 336\times 7.85} = 3.93 \text{ mpy}$$

Standardization for the aliquot is set to the power of the third diluents, 10^3. Therefore, for the waterlogged clay soil sample, the standardized microbial count is given as 264×10^3 CFU g^{-1}. This is equivalent to 2.64×10 s CFU g^{-1}. Also, for the dry clay soil sample, the standardized microbial count is given as 36×10^3 CFU g^{-1}. This is equivalent to 3.6×10^4 CFU g^{-1}:

$$\text{Corrosion rate for coupon 1 + Glutaraldehyde (0.1M)} = \frac{(9.216-9.191)\times 3450000}{20.61\times 336\times 7.85} = 1.59 \text{ mpy}$$

$$\text{Corrosion rate for coupon 2 + Sodium Molybdate (0.1M)} = \frac{(9.392-9.359)\times 3450000}{20.61\times 336\times 7.85} = 2.09 \text{ mpy}$$

$$\text{Inhibitive Efficiency (IE)} = \frac{CR_{uninhibited} - CR_{inhibited}}{CR_{uninhibited}} \times 100\% \quad (2)$$

Where:

$CR_{uninhibited}$ = Corrosion rate of the uninhibited coupon in water logged clay soil sample
$CR_{inhibited}$ = Corrosion rate of the inhibited coupon in water logged clay soil sample
Time basis = 336 h
$CR_{uninhibited}$ = 3.93 mpy (at 336 h)

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