International Journal of Ecology and Environmental Sciences 39 (1): 51-58, 2013
ISSN: 0377-015X (Print); 2320-5199 (Online)

Biologically Active Solid Deposits in Biocide Treated Oil and Gas Pipelines from a Nigerian Onshore Oil Production Facility[1]

C.C. OKORO[1]*, O.O. AMUND[2] AND O.B. SAMUEL[3].
[1] *Department of Biological Sciences and Biotechnology, Caleb University, Imota Lagos, Nigeria*
e-mail: chuma2k2001@yahoo.com
[2] *Department.of Microbiology, University of Lagos, Lagos, Nigeria (Email: kay_amund@yahoo.com).*
[3] *Department of Marine Sciences, University of Lagos, Lagos, Nigeria. (Email: osamuel@unilag.edu.ng)*

* Corresponding author;

ABSTRACT:

Pipelines transporting crude oil, produced water and gas from an onshore oil producing facility in Nigeria are subject to frequent corrosion failures despite treatment programs with chlorine (0.5mg L^{-1}) and sodium azide (0.2 mg L^{-1}). Solid deposit samples from 6 corroded pipelines were analyzed chemically and biologically to determine whether the corrosion episodes were as a result of microbial activity. Microbiological analysis showed relatively high concentrations of microorganisms associated with corrosion such as SRBs (10^3-10^5 cells g^{-1}) and APBs (10^3-10^7 cells g^{-1}) while chemical analysis showed evidence of corrosion products such as iron II, calcium, sulfide, carbonate and bicarbonate. Chemical analysis also revealed that environmental conditions such as availability of nutrients, pH, moisture and redox potential were favorable for the growth and proliferation of these microorganisms. All the pipeline samples that were investigated recorded remarkable corrosion rates that ranged from 0.065-0.30 mm yr-1 but the rates were higher in chloride treated pipelines. Our investigation revealed that the solid deposit samples were biologically active and pipeline corrosion was as a result of microbial activity. Thus the prevalence of bio-corrosion is likely to be higher in cases where routine pipeline cleaning and checks are not strictly adhered to despite regular biocide treatment programs.

Key Words: Corrosion; Solid Deposits; Pipelines; Sulfate Reducing Bacteria; Acid Producing Bacteria; Biologically Active.

1INTRODUCTION:

The major problems associated with pipelines carrying produced water and crude are scaling, corrosion, dirt, solid deposit accumulation and microbial growth. Any of these problems or combination of them can result in costly operational problems like high maintenance costs, expensive parts replacement, acid cleaning operations, reduced flow capacity, increased water usage and unscheduled shut down (Videla and Herrera 2005). Solid deposit accumulation and bacteria growth in oil and gas pipelines otherwise referred to as biological active deposits (BAD) in produced water and oil pipelines or 'black powder' in gas pipelines is often ignored in the industry despite its potential for costly problems. Both 'BAD' and 'black powder' are produced in oil and gas pipelines by direct chemical reactions with pipe walls or following biological production of hydrogen sulfide which is usually accompanied by the presence of water (Baldwin 1998, Shi et al. 2011). The major biological component of the solid deposits are microorganisms, mostly sulfate reducing bacteria (SRB), acid producing bacteria (APB) and hydrocarbon utilizing bacteria while the chemical components are iron sulfide, iron oxides, hydrogen sulfide, carbonates and bicarbonates (Jack 2002, Videla and Herrera 2005). According to Davis

[1]Research work carried out at the Petroleum Microbiology Research Laboratory, Caleb University, Imota, Lagos, Nigeria

(1987), operations that involve pipelines with solid deposits can lead to loss of efficiency over time and there may be changes in internal diameter and surface roughness causing increased pressure drop. Gas and oil quality may also be affected by the solid deposits and the deposits may increase corrosion rates by offering a suitable environment for corrosion causing micro-organisms like sulfate reducing bacteria (SRB) to thrive or by preventing corrosion inhibitors that are dosed to the pipelines to protect it.

Pipeline and facility corrosion is a major issue globally and about 20-30% of corrosion is related to microbial activity (Fleming 1996, Videla 1996). Other microbial induced problems according to Videla (1996) are the slime produced by microorganisms which can lead to blockages and filter plugging. The responsible microbial populations originate from hydrocarbon and ground water sources within the subsurface or are introduced into reservoirs during water flooding or secondary oil recovery operations (Videla 1996, Videla and Herrera 2005, Park et al. 2011). Corrosions due to sulfate reducing bacteria (SRB) alone results in annual loss of about $4-6 billion in US (Syrett et al. 2001).

Microbially influenced corrosion (MIC) or bio-corrosion can be defined as an electrochemical process where the participation of the microorganisms is able to initiate, facilitate or accelerate corrosion by changing the electrochemical conditions at the metal - solution interface (Videla and Herrera 2005). Microorganisms are ubiquitous in oil and gas environment and may influence both the initiation and propagation of all known types of metallic corrosion. As a consequence, MIC poses a serious risk for the integrity, performance and reliability of nearly all metals used in oil and gas operations such as iron, steel, aluminum, copper, titanium and their alloys. The main types of microorganisms associated with corrosion failures of cast iron, mild and stainless steel structures are sulfate reducing bacteria (SRB), sulfure oxidizing bacteria, iron oxidizing/reducing bacteria, manganese oxidizing bacteria as well as bacteria secreting organic acids and extracellular polymeric substances (EPS) or slimes (Beech et al. 2000). These organisms co-exist in naturally occurring biofilms often forming communities that are able to affect electrochemical processes through cooperative metabolism which individual species have difficulty to initiate (Beech et al. 2000). Guidelines for the identification of MIC according to Jack (2002) are:

- Metallurgical, i.e. the appearance of the corrosion damages such as corrosion pits.

- Biological analysis which include the numbers of viable SRBs, APBs and other anaerobic and aerobic bacteria present.

- Chemical analysis which include Fe^{2+}, Fe^{3+}, Calcium, Sulfide, Carbonate and Bicarbonate.

Simple chemical tests of the sample according to Jack (2002) can detect carbonate by extensive burble formation and sulfide by the characteristics repulsive odor of hydrogen sulfide or by its reaction with a color indicator such as lead acetate. Fe^{2+} and Fe^{3+} can be detected in the resulting acid solution by using standard ferricyanide and thiocyanate tests. Addition of oxalate to the test solution after buffering with excess sodium acetate yields a white precipitate if calcium is present. It should be noted that the presence of a hard white calcium carbonate scale is a good evidence that effective corrosion product potential is achieved on metal surfaces where it is found (Jack 2002).

Current technologies used to control solid deposits and microbial contamination is the use of mechanical scrapping of biofilms formed in pipelines with “pigs” and the use of chemical biocides. Biocides are anti-microbial chemicals that are used to kill microbes or control their proliferation by inhibiting their growth and reproductive cycle and according to Shi et al. (2011), three main mechanisms of biocidal attack on micro-organisms exists;

1. Those that make use of oxidizing agents such as chlorine, ozone, chloroamides, hypoclorites and bromide.
2. Enzyme inhibitors and protein denaturants such as sodium azide, bisthiocyanates, salts of heavy metals, isothiolativezoles and aldehydes.
3. Surface active agents such as quaternary ammonium compounds.

Most biocides have to deal with environmental and health concerns over their use because most of them are toxic and not friendly to the environment. Research have also shown that relative to planktonic microbes, sessile microbes are highly resistant to biocides as the EPS synthesized by some microorganisms tend to act as a barrier by preventing the biocides from reaching their target sites (Videla and Herrera 2005).

In the present study, we tried to establish the level of biological activity of some solid deposits after prolonged treatment with biocides. Direct measurement of biological activity were also carried out on some excavated pipelines that were previously treated with biocides. The aim of our investigation was to establish the level of biological activity of the solid deposits after a prolonged treatment with some biocides. This will help

to predict the extent the biocides can protect the oil and gas pipelines from bio-corrosion. We also drew some comparison between the level of biological activity of solid deposits from gas pipelines (black powder) with relatively low moisture content and those of produced water and oil pipelines with relatively higher moisture content.

MATERIALS AND METHODS.

Source of Samples, Collection and Handling

Solid deposit samples were collected from excavated pipelines that comprised of 20" oil and gas and 10" produced water pipelines from a Nigerian onshore oil production facility. The pipelines were previously treated with chlorine (0.5 mg L^{-1}) and sodium azide (0.2 g L^{-1}) periodically for about 8 years before fouling and leakages were suspected and the pipelines were excavated and replaced. Direct measurement of some parameters like pH and redox potential were also carried out in the excavated pipelines. Samples were collected with 500 mL sterile glass container and preserved with ice bags before transportation to the laboratory for analysis. Full description of sample codes are shown in Table 1.

Physicochemical Analysis of Samples

pH and redox potential of the solid deposits were measured with bright platinum electrodes and a colonel reference electrode. Readings were taken with a potable pH/mV digital meter as described in Patrick et al., (1996). Moisture content was measured by simple gravimetric analysis as described in Eaton et al., (1995). Analysis of total petroleum hydrocarbon (TPH) was carried out as previously described (Okoro 2010), while carbonate, bicarbonate and chlorides were analysed as described in Eaton et al. (1995). SO_4^{2-} was analyzed by high performance liquid chromatography (HPLC) as described in Eaton et al. (1995). Dissolved sulfide was determined using the diamine method (Truper and Schlegel 1964). NH_4^+ was measured using the indole-phenol method while NO_3^-, NO_2^- and organic acids, acetate, propionate and butyrate were analyzed using HPLC as described in the standard methods (Eaton et al. 1995).

Fe^{2+} Assay

Sterile distilled water (100 mL) was added to 100 g of solid deposits and shaken vigorously for 10 min to bring part of the solid deposit sample into solution. Two sets of tubes measuring 13x100mm were used for analysis. The first set was for acid extraction of the samples where 0.5ml of 0.5N HCl was added to each tube followed by the addition of 100µL of the mixed sample. The mixture was vortexed and allowed to stand for 15 min. In the second set of tubes, 3 ml of ferrozine solution was added to each tube followed by the addition of 100µL of the acid extracted sample, the sample was vortexed and absorbance was read immediately at A_{562}. Fe^{2}+ concentration in the samples was extrapolated from the standard curve prepared with known Fe^{2+} concentrations.

Measurement of Corrosion Rates

Sterile distilled water (100 mL) was added to 100 g of solid deposits and shaken vigorously for 10 min to bring part of the solid deposit sample into solution. The corrosion rate (CR) of the liquid samples were determined from the metal weight loss ($\triangle$W) according to the equation (Parks et al. 2011):

$$CR = 87{,}600 \times \triangle W/(A \times T \times D)\ mm\ yr^{-1}$$

Table 1. Sample codes and description

S/N	Sample Code	Sample description
1	GPL-Cl	Solid deposits from gas pipeline(20") treated with Chlorine (0.5 mg L^{-1})
2	GPL-Na-AZ	Solid deposits from gas pipeline(20") treated with Sodium azide (0.2 mg L^{-1})
3	OPL-Cl	Solid deposits from oil pipeline(20") treated with Chlorine (0.5 mg L^{-1})
4	OPL-Na-AZ	Solid deposits from oil pipeline(20") treated with Sodium azide (0.2 mg L^{-1})
5	PWPL-Cl	Solid deposits from produced water pipeline (10") treated with Chlorine (0.5 mg L^{-1})
6	PWPL-Na-AZ	Solid deposits from produced water pipeline (10") treated with Sodium azide (0.2 mg L^{-1})

where A represents the coupon area (cm^2) and D represents the density of the steel (7.85g cm^{-3}) and T is the duration of the experiment in hours.

Most Probable Number (MPN) Estimation

To quantify the presence of SRB in the samples, the API RP-38 broth medium was used. Serial dilution of the samples in API RP-38 broth medium was made with the use of a sterile syringe. 1 mL of each sample was inoculated to the 9 mL of the medium and the sequence was repeated serially to the last tube. Samples were then incubated at 37°C for up to 30 days. Formation of black precipitates of iron sulfide was used as a diagnostic tool to confirm the presence of SRB.

For acid producing bacteria, prepared ZPRA-5 medium (Phenol red-dextrose reagent) with a salinity of 5000 ppm was used. Change in color from orange to yellow shows the presence of acid producers (fermentation of dextrose).

Enumeration of Heterotrophic Bacterial Population

0.1 mL aliquot of each of the 10-fold serially diluted samples was introduced into prepared nutrient agar plates (Oxoid) in duplicate and incubated for 24 hr. at 37 ^{0}C. The bacterial colonies were observed and counted.

Enumeration of Hydrocarbon Utilizing Bacteria

Hydrocarbon utilizing bacterial counts in the samples were carried out as described by Mills et al.. (1978).

Statistical Analysis.

One way analysis of variance (Anova) and Duncan tests were used and the analysis was performed with a computer statistical package, XPSS 10 to determine the standard deviations, standard errors and the level of correlations between SRB and other factors on one hand and corrosion rates and ferrous iron concentration on the other. Regression analysis was done on excel software.

RESULTS

Physicochemical Characteristics of Solid Deposits

All the solid deposit samples showed bubble formation when reacted with concentrated HCl and this indicates the presence of carbonates. Samples from gas pipelines treated with chlorine and sodium azide had relatively lower concentrations of TPH (60-98 ppm) when compared with those from oil and produced water pipelines (68-500 ppm). Sulfate was present at relatively higher concentrations in samples from gas pipelines (8.30-12.48 mM) as opposed to those from oil and produced water pipelines (0.30–1.45 mM) where relatively low concentrations of hydrogen sulfide were detected. Fe^{2+} and NH_4^{2+} were present at considerable concentrations in samples from oil and produced water pipelines while chloride was detected at relatively high concentrations in samples where chlorine was used as a biocide. Organic nutrients such as acetate and propionate were present at significant concentrations in samples from oil and produced water pipelines. No butyrate was detected in all the samples analyzed. Detailed results are shown in Table 2.

Microbiological Characteristics of Solid Deposits

Solid deposit samples from gas pipelines treated with sodium azide showed relatively low concentrations of heterotrophic bacteria (THB), hydrocarbon utilizing bacteria (HUB), Sulfate reducing bacteria (SRB) and acid producing bacteria (APB) when compared with those treated with chlorine (Table 3). Generally, samples from oil and produced water pipelines recorded higher bacterial counts than those from gas pipelines. In a similar scenario, samples from oil and produced water pipelines treated with chlorine recorded higher concentrations of THB, HUB, SRB and APB than those treated with sodium azide. This showed that the sodium azide applied at the concentration of 0.2 mL L^{-1} was more bactericidal than chlorine (0.5 mL L^{-1}) but neither of the biocide was able to demonstrate considerable inhibition of bacterial growth and proliferation in the solid deposit samples.

pH and Redox Potential at Various Depths of the Solid Deposits

The pH of the solid deposit samples at the uppermost layer (0.5-2 cm) ranged between 5.80 and 6.90 while the redox potential ranged between 80-180 mV. It is expected that this environment will be dominated by aerobic neutrophils. At the middle layer (2.5 - 4 cm), the pH ranged between 4.2 and 5.6 while the redox potential ranged between -60 and -140 mV indicating that micro-aerophilic and anaerobic acidophile bacteria are likely to

Table 2. Physicochemical analysis of solid deposits[a].

Parameters	GPL-Cl	GPL-Na-AZ	OPL-Cl	OPL-Na-AZ	PWPL-Cl	PWPL-Na-AZ
TPH (mg kg^{-1})	60-85	68-98	350-500	280-456	68-120	80-145
Redox Potential (mV) (Midlayer)	- 65 - -120	-85 - -160	-120 - -240	-90- -210	-65 -80	-40- -65
Moisture (%)	6-8	12-18	15-23	18-25	20-27	23-30
pH (1-4 cm)	6.40-7.60	6.40-7.70	5.90-7.30	6.50-7.80	6.20 7.40	6.10 7.60
HS^- (mM)	ND	ND	0.45-0.58	0.28-0.30	0.35-0.60	0.10-0.18
Sample Temp.(°C)	28-32	28-30	30-33	28-30	30-33	29-30
NH_4^+ (mM)	ND	ND	0.65-0.70	0.38-0.40	0.28-0.35	0.14-0.28
Fe^{2+} (mM)	0.18-0.58	0.36-0.48	2.58-2.65	0.25-0.36	2.60-3.20	1.30-1.60
$C0_3$ (mg g^{-1})	380-400	245-260	68-80	45-60	450-480	285-300
$HC0_3$ (mg g^{-1})	2300-2400	1800-2000	650-800	860-950	3400-3480	3270-3300
$N0_2$ (mM)	0.55-0.65	0.21-0.33	0.30-0.32	0.56-0.60	0.20-0.35	0.43-0.58
$N0_3$ (mM)	2.80-2.86	3.20-3.32	5.40-5.80	3.30-3.75	2.40-2.51	1.60-1.68
$S0_4$ (mM)	12.48-12.51	8.30-8.38	0.50-1.10	1.20-1.32	0.30-0.48	1.30-1.45
Cl^- (mg kg^{-1})	8566-8600	480-495	6500-6570	320-360	8340-8450	1360-1382
Reaction with conc. HCl	Bubble formation	Bubble formation	Bubble formation Smell of H_2S	Bubble formation Smell of H_2S	Bubble formation Smell of H_2S	Bubble formation Smell of H_2S
Slime formation	none	Present	Present	Present	Present	Present
Acetate (mM)	0.25-0.31	0.54-0.56	1.26-1.30	0.86-1.20	2.56-2.60	2.20-2.36
Propionate (mM)	0	0	0.85-0.88	0.24-0.28	1.23-1.30	0.43-0.48
Butyrate (mM)	0	0	0	0	0	0

[a] Data represents the ranges observed in triplicate solid deposit samples; For sample codes, see Table 1.

Table 3. Mean population densities of microorganisms in solid deposit samples.

Sample Codes	THT Bacteria CFU g^{-1} x 10^5	HCU Bacteria CFU g^{-1} x 10^5	SRB Cells g^{-1}	APB Cells g^{-1}
GPL-Cl	45	06	10^4	10^5
GPL-Na-AZ	16	04	10^3	10^3
OPL-Cl	380	140	10^5	10^5
OPL-Na-AZ	56	22	10^3	10^4
PWPL-Cl	240	116	10^5	10^7
PWPL-Na-AZ	110	48	10^4	10^5

HCU = Hydrocarbon Utilizing; THT = Total Heterotrophic, APB = Acid Producing bacteria; SRB = Sulfate Reducing bacteria

dominate the environment. At the lower layer (5-10 cm), pH ranged between 3.8 and 5.6 while the redox potential ranged between -220 and -340 mV indicating that the obligate anaerobic acidophiles are likely to dominate the environment. Detailed results are shown in Figure 1. In summary, the environment under investigation can encourage the growth and proliferation of varieties of microorganisms comprising mostly of aerobic, micro-aerophilic and anaerobic acidophiles and neutrophiles.

Corrosion Rates.

The corrosion rates of samples from gas pipelines treated with chlorine and sodium azide were relatively low (0.06 and 0.10 mm yr^{-1}). Samples from oil and produced water pipelines treated with chlorine recorded higher corrosion rates (0.22 and 0.28 mm yr^{-1} respectively) as opposed to those treated with sodium azide (0.18 and 0.20). Detailed results are shown in Figure 2.

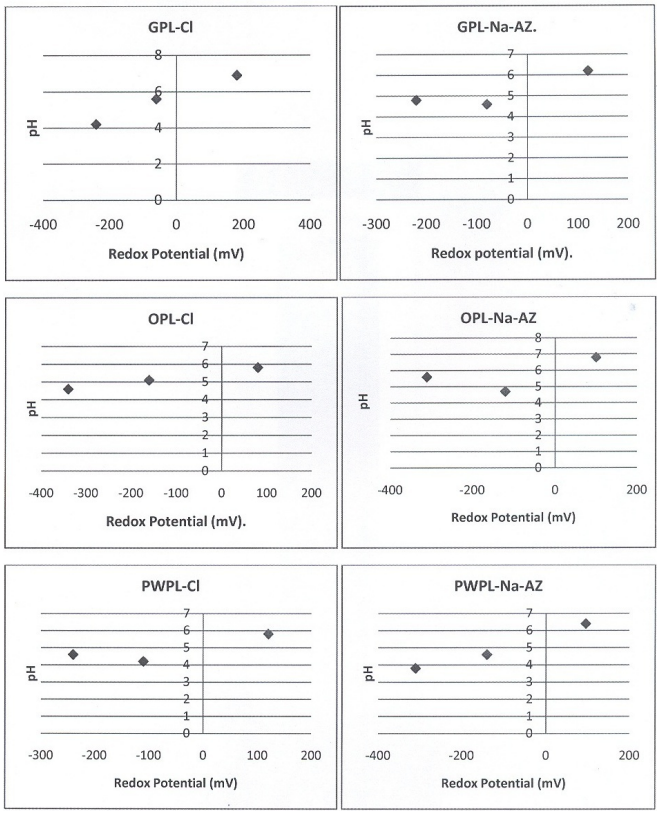

Figure 1. pH and redox potential at upper, middle and lower layers of biologically active solid deposit samples from excavated biocide treated pipelines.

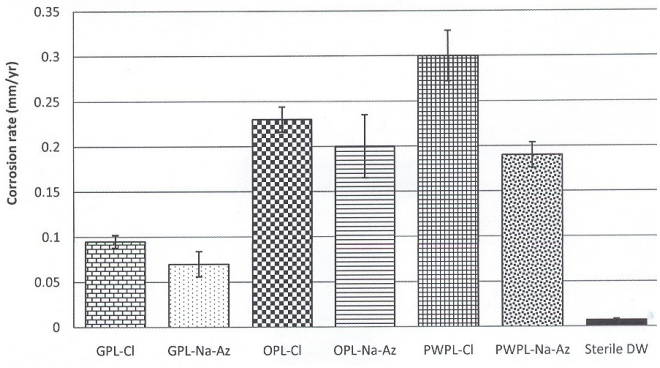

Figure 2. Corrosion rates in (mm year^{-1}) of iron coupons in samples as determined by metal weight loss. Data represent average results from two separate incubations with two coupons each.

Fe^{2+} Concentration in Samples After Incubation with Metal Coupons

The concentration of Fe^{2+} was relatively low in gas pipelines treated with sodium azide (0.56 mM) and chlorine (1.45 mM). Those from oil and produced water pipelines treated with chlorine showed relatively high concentrations of Fe^{2+} (2.55 and 3.65 mM) as opposed to those treated with sodium azide (1.65 and 2.34 mM respectively). Detailed results are shown in Figure 3.

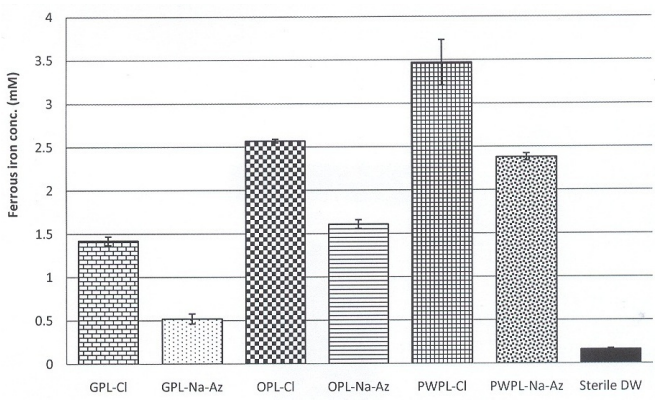

Figure 3. Concentration of ferrous iron (mM) in samples after 5 weeks of incubation with metal coupons

Factors Correlating with SRB, Corrosion Rates and Ferrous Iron Concentrations in Solid Deposits

There was a positive and strong correlation between SRB and APB (+0.635). Sulfate also correlated positively with SRB (+0.500). The correlation between pH and SRB was strong though negative (-0.928). Other factors such as moisture, redox potential and TOC correlated poorly with SRB (Table 4). The regression analysis carried out on the data obtained showed that ferrous iron concentration correlated positively and strongly with corrosion rate (Figure 4).

DISCUSSION AND CONCLUSION

Information provided by the operators of pipelines that were investigated revealed that the pipelines had been in operation for about 8 yr before leakages were observed as a result of corrosion and fouling. This development led to the present study which was designed primarily to establish the role of microbial activity in corrosion.

Table 4. Factors correlating with Sulfate Reducing Bacteria (SRB) and other parameters in solid deposit sample (expressed as correlation coefficient)

	SRB	APB	TOC	Redox Potential	pH	Sulfate	Moisture
SRB	1.000						
APB	0.635	1.000					
TOC	-0.045	-.457	1.000				
Redox Potential	-.107	.462	-.797	1.000			
pH	- .928**	-.446	.172	.013	1.000		
Sulfate	.500	.639	-036	.380	-.355	1.000	
Moisture	.281	.350	.108	.286	-.172	.937**	1.000

** Correlation is significant at the 0.01 level (2-tailed).

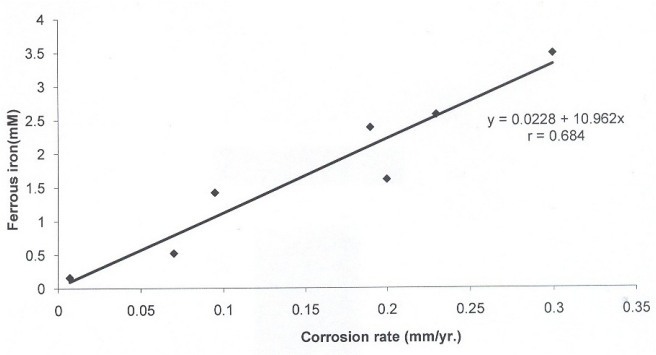

Figure 4. Correlation between ferrous iron concentration and corrosion rates (Correlation coefficient r = 0.684)

Further discussion with pipeline operators also revealed that routine pipeline cleaning and maintenance were not strictly adhered to as a result of poor security situation in the areas where the pipelines were located. Though some investigators have advanced that about 20-30% of corrosion cases are related to microbial activity (Fleming 1996, Videla 1996), we observed that in some cases where routine pipeline cleaning and maintenance are not strictly adhered to, higher prevalence of bio-corrosion are likely to be recorded. According to Lane (2005), maintaining the cleanliness of systems is the best method to prevent MIC and where routine cleanliness of the systems is ignored, bio-corrosion is likely to pose a very serious challenge.

Our present study reveals that there was considerable corrosion in all the pipelines investigated despite treatment with biocides such as chlorine and sodium azide. There was enough evidence also to suggest that the corrosions were as a result of microbial activity because the microorganisms asso-ciated with corrosions such as the SRBs and the APBs were present and active at considerable concentrations in all the pipeline samples investigated. We also observed that environmental conditions such as temperature, pH, moisture and redox potential were favorable for their growth and proliferation. Scotto and Mollica (2000) have observed that the ability to produce a wide spectrum of corrosive metabolic by-products over a wide range of environmental conditions and resistance to biocides used to treat pipelines makes microorganisms a real threat to the stability of metals that have been engineered for corrosion resistance.

Another significant observation in our study is the prevalence of higher corrosion rates in samples with significant moisture content and biofilm formation. Some investigators have advanced that biofilms forms a significant diffusion barrier and thus confers resistance to antimicrobial agents since they cannot penetrate to make active contact with microorganisms (Vidella and Hirrera 2005, Jack 2002). Though we suspected that the microorganisms might have developed resistance to the biocides used for treatment over time because of the considerable microbial populations observed, it was also noted that chloride treated pipelines recorded higher corrosion rates than the sodium azide treated ones. We also observed a strong positive correlation between SRB, APB and sulphate and a strong negative correlation between SRB and pH in the pipelines investigated. SRB correlated poorly with other environmental factors such as moisture, redox potential and TOC. In a similar investigation carried out by Jack (2002) for buried pipelines, SRB correlated strongly and positively with APB, moisture and TOC and poorly with sulfate and redox potential. Our investigation also revealed that the respective ranges of pH and redox potential for aerobic and anaerobic microorganisms in the solid deposits from the pipelines were 3.5 to 6.8 and -380 to + 200. Jack

(2002) noted that where anaerobic forms of microbial metabolism tend to be found, the pH and redox potential ranged between 4.5 to 9.5 and -420 to +200 respectively.

Our investigations also showed relatively high corrosion rates of solid deposit samples that ranged from 0.065 to 0.30 mm yr^{-1}. The highest corrosion rate (0.30 mm yr^{-1}) was observed in produced water pipelines treated with chlorine (PWPL-Cl) while the lowest (0.065 mm yr^{-1}.) was observed in gas pipelines treated with sodium azide. Jack (2002) stated that for buried pipelines in anaerobic soil environments where SRB have not precipitated extensive iron sulfide deposits, corrosion rates range from 0.002 to 0.01 mm yr^{-1}. but where the focused action of SRB have exposed unprotected steel to extensive iron sulfide deposits, very high corrosion rates greater than 0.2 mm yr^{-1}. can be seen. In a similar investigation, Park et al., (2011) observed much lower corrosion rates that ranged from 0.008 to 0.009 mm yr^{-1}, in Canadian brackish water transporting pipelines. Interestingly, our investigations show strong correlation between ferrous ion concentrations in samples incubated with metal coupons for 5 weeks and corrosion rates of metal coupons in samples.

In conclusion, we have demonstrated that corrosion rates and the prevalence of bio-corrosion can be higher in cases where routine pipeline cleaning and checks are not strictly adhered to despite treatment with biocides. Such development can lead to resistance to the antimicrobial agents by the microorganisms over time. This aspect of investigation has not been clearly documented in literature in the Gulf of Guinea where security concerns have consistently prevented routine pipeline checks over the years.

ACKNOWLEDGEMENTS

This work was supported by grants from Global Environmental Fund and Microcheck Nigeria Limited.

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Received 20 December 2012;
Accepted after Revision 28 March 2013