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A FRAMEWORK FOR CONDUCTING ANALYSIS OF MICROBIOLOGICALLY INFLUENCED CORROSION FAILURES

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OVERVIEW OF MIC

Ethane crackers are subject to severe operating conditions that Microbiologically influenced corrosion (MIC) is corrosion that is affected by the presence and/or activity of microorganisms, including bacteria, archaea and fungi.[1] MIC is experienced by assets in the oil and gas industry, paper production, cooling water systems, waste water handling, manufacturing, infrastructure, and many other places. Problems with MIC and fouling in aviation jet fuel tanks and biodiesel storage and delivery systems by bacteria, yeast and filamentous fungi are also well-documented. Since microorganisms require water to demonstrate activity, the potential for MIC is logically associated with locations where exposed metal is in contact with water or solids containing sufficient moisture.

On wetted surfaces, microorganisms typically exist in a diverse biofilm that consists of extracellular polymeric substances (EPS), various types of cells, and organic and inorganic material. Biofilms formed by microorganisms create a microenvironment on the metal surface that can differ significantly from the overall environment, leading to local differences in electrochemical potential resulting in corrosion. Microbial activities in biofilms can also facilitate corrosion by producing corrosive metabolites (acids, elemental sulfur), changing the nature or kinetics of rate controlling reactions, forming mineral scales, and direct uptake of electrons from the steel surface.

Microorganisms and their activities are affected by many environmental factors, including most prominently, temperature, salinity, oxygen concentration, pH, and availability of nutrient compounds. Although most microorganisms can exist over a range of these environmental conditions, they commonly have a set of conditions at which growth is optimal. When conditions are unfavorable, cells may become dormant or die, or transition to a spore form that can last a long time until conditions once again become favorable for growth.

While the chemical and physical environments have a significant effect on microbiology, they also have an equally significant effect on corrosion mechanisms and forms of damage. When diagnosing MIC, the abiotic (i.e., non-biological) effects of the environment must also be considered. Distinguishing the degree to which abiotic or biotic conditions lead to a corrosion failure is one of the greatest challenges in MIC failure investigation. The use of a MIC diagnostic framework; however, can help provide the insight needed to sort out the actual cause.

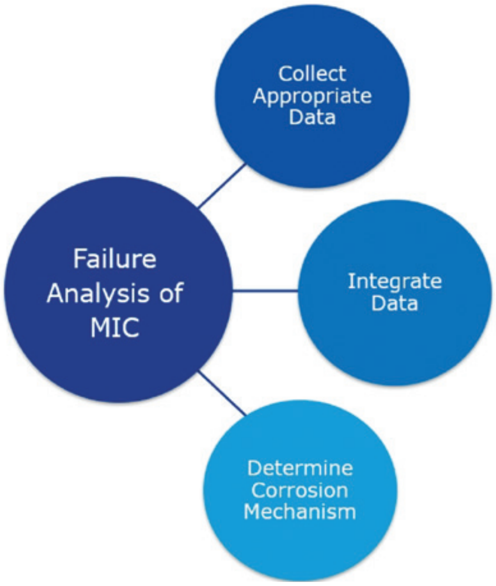

Figure 1. Overview of the failure analysis process

MIC FAILURES

In the oil and gas industry, a significant number of pipeline failures due to either external or internal corrosion have resulted from MIC, some with catastrophic consequences. One widely publicized case occurred in August of 2000 when a 30” diameter high pressure natural gas pipeline near Carlsbad, New Mexico ruptured and caught fire, killing 12 individuals. The rupture was determined in part to be the result of internal MIC. Another high-profile crude oil pipeline leak due to MIC occurred in 2006, leading to a temporary shutdown of most of the crude oil production on the North Slope of Alaska.[2] Where natural gas and crude oil are gathered from producing wells, water (brine) is often commingled, exposing the gathering systems to the threat of MIC.

FAILURE ANALYSIS

Comprehensive and thorough analysis of failures should be conducted to investigate the failure mechanism. A holistic understanding of the failure mechanism is essential for selecting appropriate measures for mitigating or preventing further corrosion from occurring. Additionally, the data collected as part of failure analysis can be used to identify other corrosion threats and assess the risk of failure due to these threats in the system.

The framework for failure analysis is a three-step process (**Figure 1**). The first step is to collect appropriate data from the location

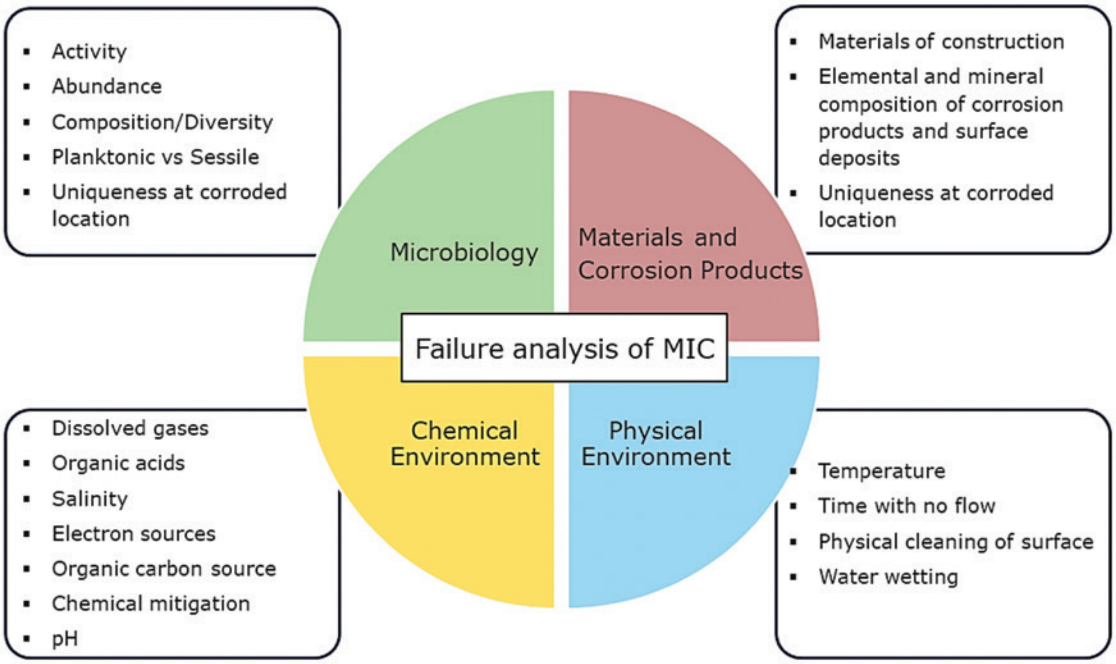

Figure 2. Overview of data to be collected for conducting MIC failure analysis.

of failure and the different categories of data to be collected are detailed in the following section. Additionally, collecting data from the non-corroded locations is worthwhile to examine the differences between the failure location and the non-corroded locations. Further, historical trends in the data and any known changes to the system design or operation should be gathered.

The second step is to integrate the data collected from the failure location and non-corroded locations, along with historical changes to the different parameters.

Once all this data is integrated, the last and the most important step is to determine the corrosion mechanism and to interpret the cause for the failure. Based on the available data, all the possible abiotic corrosion mechanisms should be considered and weighted against MIC and finally, the cause of failure should be assessed.

DATA COLLECTION FOR CONDUCTING FAILURE ANALYSIS

The outcome of failure analysis is as effective as the quantity and quality of information collected.[3] Since MIC is characterized by the interactions of the microbial community with corroding metal, other deposits, and physical and chemical environment, it is crucial to analyze the microbiology, physical and chemical conditions, and materials and corrosion products of the environment to determine if MIC is the actual cause of the failure.[4] These categories of data should be collected to obtain multiple lines of evidence from the failure location (**Figure 2**). The data to be collected and the analytical methods to be used for the data collection are described in this section.

Microbiology

Microbiological analysis plays a vital role in investigating the cause of failures and subsequently attributing the failures to MIC. Several methods are available for microbiological testing and each of these methods reveal different types of microbiological information. Culture-based methods such as serial dilution tests and most probable number (MPN) tests, DNA-based methods such as quantitative polymerase chain reaction (qPCR) and DNA sequencing, and enzyme-based methods such as adenosine triphosphate (ATP) tests and enzyme tests for sulfate reducers are the commonly used methods in MIC-related investigations.

These methods provide information on microbial abundance, composition of the microbial community, or microbial activity. The quality of microbiological information obtained from the failure depends on the sample type and method used. DNA-based methods such as qPCR and DNA sequencing provide the most reliable information on the microbial abundance and composition of the microbial community and hence are recommended as compared to serial dilution tests for obtaining information on microbial presence, abundance, and composition.

Additionally, the type of sample used for the microbiological testing plays a significant role in influencing the reliability of the data used to determine if MIC is the cause of failure. It is well known that MIC is mediated through biofilms attached to the metal surface at the corroding location. Hence, microbiological testing should be preferentially conducted on sessile samples (such as surface swabs and corrosion deposits) as they provide substantially more valuable information than planktonic samples (liquid samples).[5]

To determine if MIC is the cause of a failure, the microbiological tests should be conducted both at the corroded location and a non-corroded location closer to the corroded location. Next, the results of these tests should be compared to demonstrate if the corroded location has higher microbial abundance, increased microbial activity, or greater percentages of known corroding species (e.g., sulfate reducers, acid producers, methanogens, iron reducers, nitrate reducers) in the microbial community than the non-corroded location. If any of these differences are observed between the failure location and the non-corroded locations, this indicates the possibility that MIC might be a cause of failure. However, further analysis of the materials and corrosion products along with physical and chemical conditions is required to conclusively determine MIC as the cause of failure.

Materials and Corrosion Products

The microbial communities present in a system interact with the materials and corrosion products to either initiate or accelerate corrosion reactions. It is important to examine the materials and corrosion products associated with a failure to interpret the corrosion mechanism(s). Different materials have varying degrees of susceptibility to MIC. Hence, the materials used in construction of corroded equipment may help determine the probability of MIC as the cause of failure. For example, steels, the most commonly used materials of construction, are susceptible to MIC. Aluminum alloys, some alloys of copper, and some alloys of nickel are also susceptible to MIC, whereas titanium alloys and other alloys made with corrosion resistant materials such as chromium, nickel, molybdenum, and zirconium, are more resistant to MIC.[6]

The elemental and mineral composition of the corrosion products and surface deposits are important to determine whether MIC is a likely failure mechanism. Various analytical methods, such as energy dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD), are available to analyze the composition of deposits and corrosion products. EDS gives the elemental composition of deposits, whereas XRD gives the phase-composition of deposits. Sulfides, oxides, and carbonates are the commonly observed corrosion products associated with MIC. This information on corrosion products and deposits should be collected from the location of failure along with other non-corroded locations. The comparison of the corrosion products and deposits between the failure location and the non-corroded location can provide meaningful insights on the uniqueness of the corrosion threat and the mechanism of failure.

Chemical Environment

Information on the chemical environment near the failure location is important to establish the likelihood of MIC as a failure mechanism. Analytical methods such as liquid chromatography mass spectrometry (LC-MS) and high-performance liquid chromatography (HPLC) are available to analyze the chemical composition of fluids. The chemical environment is characterized by the presence of dissolved gases (such as carbon dioxide, hydrogen sulfide, oxygen), organic acids (such as acetic acid, propionic acid, lactic acid), presence of electron acceptors and electron donors (such as sulfate, nitrate, hydrogen, acetate, lactate, iron, sulfur), availability of carbon sources, pH of the fluids, and the use of production chemicals (such as biocides, corrosion inhibitors, scale inhibitors) in the system. The impact of these parameters on MIC is discussed below.

Dissolved gases such as carbon dioxide, hydrogen sulfide, and oxygen not only promote abiotic corrosion, but can also be associated with MIC. Methanogens utilize carbon dioxide to produce methane and aerobic bacteria use oxygen as an electron acceptor, while sulfate reducers can convert sulfate to hydrogen sulfide. Organic acids such as acetic acid, propionic acid, and lactic acid can be generated as products of microbial metabolism and can indirectly promote corrosion. Microorganisms facilitate various redox reactions between electron acceptors and electron donors present in the environment as part of their metabolism. The presence of electron acceptors such as sulfate and nitrate promote the metabolism of sulfate and nitrate reducers in the presence of iron as the electron donor, increasing the likelihood of MIC. Other electron donors associated with metabolism of MIC causing microorganisms are acetate, lactate, hydrogen, and sulfur. Similarly, the availability of carbon source is crucial for microbial metabolism. Hydrocarbons and production chemicals present in the system are some of the common sources of carbon. The pH of the fluids determines the likelihood of microbial growth in the system. A pH value closer to neutral offers an ideal environment for microbial growth. Yet, it is not uncommon for MIC to occur in extreme pH conditions as acidophilic microorganisms that can tolerate low pH conditions have been detected in corroding systems. The concentration and frequency of the production chemicals added to the system should be examined to determine their effectiveness on abiotic corrosion and MIC. In some cases, these production chemicals can be utilized by microorganisms as organic carbon sources or electron acceptors or donors to facilitate their metabolism.

All of these parameters that are characteristic to the chemical environment impact microbial metabolism and their interactions with the corroding metal and corrosion products, subsequently resulting in MIC. Examining the chemical environment between the failure location and non-corroded locations, along with historical changes to these chemical parameters, can reveal valuable information to interpret the cause of failure.

Physical Environment

Information on the physical environment of the system such as temperature, possibility of water wetting, duration of no flow, and use of physical cleaning methods (such as pigging, scraping, jetting) near the failure location are important to investigate the existence of a MIC threat. The impact of these parameters on the likelihood MIC is discussed below.

Temperature of the system in the range of 25°C to 45°C provides an ideal environment for microbiological growth. However, MIC has frequently been reported in environments with temperatures outside of this range. Water is a fundamental factor required for corrosion in general. Hence, the possibility of water wetting and duration of water wetting near the failure location is essential to investigate the cause of failure. Further, the absence of flow in the system will promote water settlement, subsequently increasing the likelihood of corrosion. The use of physical methods such as pigging, scraping, and jetting to clean the surfaces is effective in removing corrosion products and biofilms. Therefore, the use of physical cleaning methods generally reduces the likelihood of internal corrosion.

All of these parameters that are characteristic to the physical environment impact the microbiology and corrosion products, along with their interactions with the chemical environment, and subsequently can lead to MIC. Comparing the physical environment between the failure location and non-corroded locations in the system along with historical changes to the physical environment can reveal valuable information to interpret the cause of failure.

INTEGRATING INFORMATION FOR FAILURE ANALYSIS

After collecting the appropriate data from a failure, the next step is to integrate this data to determine the corrosion mechanism and cause of failure. This step requires careful consideration along with comprehensive examination of all information available. Abiotic and biotic threats in the system should be considered based on the historical corrosion susceptibilities of the system and the data collected at the time of failure. As stated earlier, the evidence for MIC in the system should not be determined solely based on high microbial abundance or high microbial activity or presence of usual suspects of MIC like sulfate reducing bacteria (SRB), acid producing bacteria (APB) and methanogens. Rather, the evidence for MIC should be ascertained using multiple lines of evidence by investigating whether microbiology, materials and corrosion products, physical and chemical environment highlight the existence of a MIC threat. Once MIC has been identified as a possible failure mechanism, all the threats in the system (abiotic corrosion and MIC) should be weighed against each other based on the data collected at the time of failure to determine the corrosion mechanism(s). Abiotic corrosion mechanisms can be eliminated as a failure mechanism by performing modeling to determine abiotic corrosion rates and comparing against the observed corrosion rate that led to the failure. Corrosion rates in the system higher than probable abiotic corrosion rates, along with existence of a MIC threat, may reveal MIC as the failure mechanism.

In the next section, two case studies are provided to demonstrate the process of integrating data collected to determine the corrosion mechanism and the reason for failure.

Case study 1 - Offshore oil production

In 2012, carbon steel pipe spools from an oil production system on the Otter offshore platform in the North Sea were found during inspection to exhibit severe internal corrosion and an investigation was performed.[7] The facilities were commissioned in 2002. The average temperature of the produced fluids at the inlet of the Otter oil separator was 60°C. At the time of commissioning, the major internal corrosion threat was believed to be the high partial pressure of carbon dioxide in the system that was to be mitigated using corrosion inhibitors. Early microbiological monitoring of produced fluids using MPN methods showed very low numbers of bacteria and as a result, no mitigation of MIC was performed. In 2010 and 2011, samples from the Otter Inlet Oil Separator showed between 9.05×10^0 cells/mL and 2.5×10^1 cells/mL of SRB. In the water phase, dissolved sulfide levels of 15 ppm and a pH of 6-7 were reported. Liquid velocities were reported to be about 1 m/s.

Upon removal of the spools, samples of the surface deposits associated with the corrosion were collected for analysis using qPCR for total bacteria, total archaea, SRB and sulfate reducing archaea (SRA). High numbers of methanogenic archaea (1.1×10^8 cells/g), SRB (2.3×10^6 cells/g) and SRA (2.0×10^7 cells/g) were measured in the deposits; all of which have been associated with MIC in oil production. Since most SRA are not active below 60°C, the high numbers present in these samples were believed to reflect the system temperature. Since SRA and methanogens cannot be cultivated in commonly available commercial media used for MPN, this explains why they were not identified earlier in the operation of the platform.

Chemical composition analysis of the surface deposits was performed using XRD. The sample contained a mixture of siderite ($FeCO_3$, 68.1%), mackinawite ($Fe_{(1-x)}S$, 9.6%), quartz (SiO_2, 7.7%), akaganeite (β-FeOOH, 8.6%) and lepidocrocide (γ-FeOOH, 6%). The typical sources of these minerals/corrosion products in oil production are shown in **Table 1**.

Table 1. Sources of corrosion products

Mineral	Potential Sources in Oil Production
Siderite (iron carbonate)	CO_2 corrosion of steel
Mackinawite (a form of iron sulfide)	Often associated with corrosion from SRB activity, decomposes to lepidocrocite and sulfur with oxygen exposure
Akaganeite (a form of iron oxide)	Iron oxide formed in the presence of halides (salts)
Lepidocrocide (a form of iron oxide)	Iron oxide formed below 200°C, decomposition product of mackinawite
Quartz	Sand; formation fines

After the corroded spools were replaced, the platform was returned to service and intrusive coupons were used to collect biofilms for qPCR analysis and for monitoring general and localized corrosion rates. In 2015, analysis of the coupons showed the presence of biofilms with up to 4.6×10^6 gene abundance/cm^2 and methanogenic archaea as high as 4.2×10^{11} gene abundance/cm^2 with localized corrosion rates up to 0.49 mm/yr. This investigation concluded that, although CO2 was likely contributing to general corrosion under the deposits, MIC associated with the high numbers of methanogens and SRB was driving or contributing to high localized corrosion rates. A biocide treatment program was initiated to mitigate MIC going forward and monitoring of biofilms using qPCR and coupon analysis was used to regularly help evaluate biocide performance. Returning to the concept of multiple lines of evidence as depicted in **Figure 2**, **Table 2** summarizes the evidence in each data category that was used to assess MIC in this case.

Table 2. Summary of data from offshore oil production for MIC assessment

Microbiology	Materials/Corrosion Products
• Abundant SRB, SRA and methanogens in biofilm/ deposits • Reported synergistic MIC mechanism[8] • Methanogens use CO_2 in their metabolism • SRA are active above 60°C	• Mackinawite and its decomposition product present in deposits; associated with SRB • High siderite suggests CO_2 corrosion has a role • Akageneite reflects corrosion in presence of brine
Chemical Environment	**Physical Environment**
• pH range favors most microorganisms • CO_2 a significant influence • Corrosion inhibitor is used • Electron acceptors unknown (e.g. sulfate, nitrate) • Carbon sources (crude oil) provide energy for microorganisms • Origin of dissolved sulfide is not clear	• Operating temperature 60°C • Low velocity allows solids and water to accumulate • More severe corrosion at low points (6:00 position) • Topside piping cannot be pigged to remove solids

Case study 2 – Produced water treatment plant

A produced water treatment plant was built in 2010 to process water from offshore production. The piping in this plant was made of carbon steel and was not coated internally. Produced water flowed through the treatment plant at low velocity from the slug catcher through the filters and then to a primary separator to reach the produced water storage tank. The incoming pipeline to the plant was regularly pigged. Solid accumulation occurred in the slug catcher and the plant piping, but these were infrequently cleaned due to production demands. The ambient temperatures near the plant ranged from 35°F to 100°F with the operating pressure at 950 psig. These conditions are favorable for occurrence of MIC.

qPCR testing was conducted on the produced water collected from the plant and 106 cells/mL each of SRB, iron oxidizing bacteria (IOB), and general heterotrophs were identified. Further, approximately 105 cells/mL of APB, nitrate reducing bacteria (NRB), and sulfur oxidizing bacteria (SOB) were detected. qPCR tests of the produced water showed higher SRB and APB than culture-based tests. Presence of moderate abundance levels of SRB, IOB, APB, NRB, and SOB indicated the potential for the existence of MIC. Although sessile samples were not analyzed from the plant, the presence of abundant solids provided an enormous surface area and ideal conditions for microbiological growth.

The gas produced offshore had 7 vol% carbon dioxide. Further, the reported concentration of dissolved carbon dioxide in the water phase was 2500 mg/L and dissolved hydrogen sulfide was 5 ppm. The salinity of the produced water ranged between 3% to 3.5% and the pH was near 6. Water analysis showed 45,321 mg/L of total dissolved salts with 2800 mg/L of calcium, 900 mg/L of potassium, 310 mg/L of sulfate, 300 mg/L of acetate, 200 mg/L of iron and magnesium, and negligible nitrate. Quaternary amine-based corrosion inhibitor was used to mitigate corrosion at offshore production but the residual level onshore was low, 2-7 ppm. Glutaraldehyde was batch dosed three times a week at 500 ppm concentration for an hour at the inlet of the produced water treatment plant. Batch treatment of biocide (rather than continuous) suggests potentially limited kill effectiveness of bacteria under deposits and likely quick microbial regrowth to original abundance levels, since the chemicals added would have difficulty penetrating the solids. The headspace of the produced water tanks revealed high levels (800 ppm) of hydrogen sulfide.

XRD analysis of fine solids from the pig trap showed considerable amount of silica (SiO_2; 28%) and siderite ($FeCO_3$; 15-20%). Further, lepidocrocite (γ-FeOOH; 16%), akageneite (β-FeOOH; 10%), iron sulfide (FexSy; 7%), magnetite (Fe_3O_4; 5-8%), calcite ($CaCO_3$; 6%), and mackinawite (Fe_9S_8; 4-5%) were detected. The typical sources of these minerals/corrosion products are shown in **Table 1**. Some parts of the piping were inspected using non-destructive techniques and revealed general corrosion and deep pits at the bottom of the pipe. Further, visual inspection during maintenance also revealed pitting in the equipment. ILI of the incoming pipeline, which was routinely pigged, showed little corrosion and the produced water tank had not been inspected.

In this produced water plant, the information collected highlight CO_2 corrosion and MIC as potential threats. Analysis of the microbiology, materials and corrosion products, physical and chemical environments and location of the corrosion (under deposits on the bottom of the pipe) suggest a higher potential for MIC as compared to CO_2 corrosion. However, additional information on the microbial abundance, activity, and diversity of the sessile samples collected from the plant is necessary to conclude the existence of MIC threat. Using the concept of multiple lines of evidence as depicted in **Figure 2**, **Table 3** summarizes the evidence in each data category that was used to assess the MIC threat in this case.

SUMMARY

In summary, the following key points have been discussed and illustrated to establish a framework for investigating corrosion damage and failures, particularly those that could be the result of MIC:

- MIC can be reliably diagnosed using multiple lines of evidence.
- The microbiology, physical and chemical environment conditions, materials of construction, and corrosion products are essential information for a reliable corrosion failure analysis.
- When diagnosing MIC, the abiotic (i.e., non-biological) effects of the environment must also be considered.
- Microbiological testing should be conducted on sessile samples (such as surface swabs and corrosion deposits) as they provide substantially more valuable information than planktonic samples (liquid samples).
- DNA-based methods, such as qPCR, DNA sequencing, and ATP analysis, provide the most reliable information on the

Table 3. Summary of data from produced water treatment plant for MIC assessmentt

Microbiology	Materials/Corrosion Products
• Moderate levels of SRB, IOB, APB, NRB, SOB in produced water • No biofilm testing	• Mackinawite and its decomposition product, lepidocrocite present in deposits; associated with SRB • High siderite in slug catchers suggests CO_2 corrosion has a role but could be more prevalent in the pipeline that is regularly pigged. • Akageneite reflects corrosion in presence of brine • Solid accumulation in the piping and slug catcher rarely cleaned
Chemical Environment	**Physical Environment**
• pH favors most microorganisms • Presence of CO_2 and H_2S in gas and dissolved phase • Marginally high salinity • Low residual of corrosion inhibitor • Biocide used but likely fast microbial regrowth • High solids suggest inability of chemicals to penetrate and mitigate corrosion • Presence of electron acceptors sulfate and nitrate • Carbon sources in the condensate phase	• Favorable temperature 35°F -100°F • Low velocity allows solids and water to accumulate • General and pitting corrosion at the bottom of the pipe • Incoming pipeline regularly pigged (pushes solids into plant)

microbial abundance, composition of the microbial community, or activity, respectively.

- Microbiological testing and surface and deposit chemical analyses should be conducted both at corroded locations and non-corroded locations for comparison.
- Integration of various types of data (operational, physical, chemical, microbiological, etc.) is essential for determining whether MIC or abiotic corrosion is the predominant mechanism.

With a more reliable diagnosis of the corrosion mechanism, pipeline and plant operators have a better capacity to implement and optimize the appropriate mitigation and prevention measures, leading to cost savings, risk reduction, and extended asset life. ■

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Richard Eckert has over 37 years of experience with oil and gas industry corrosion/failure investigations, internal corrosion assessment, mitigation and management; materials selection, forensic corrosion engineering, litigation support and regulatory compliance. Mr. Eckert has specific expertise in the area of microbiologically influenced corrosion (MIC) and has authored numerous publications and research reports in this field. He is the author of 3 books on internal corrosion mitigation and monitoring, corrosion failure investigation and MIC. Mr. Eckert is a NACE certified Internal Corrosion Specialist, previously served on the NACE Board of Directors, and has chaired a number of technical committees that produced standards on corrosion management and internal MIC of pipelines. He works as Senior Principal Specialist in Corrosion Management and Materials Advisory Services for DNV GL - North America Oil & Gas, in Dublin, Ohio.

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Dr. Susmitha Purnima Kotu is an engineer in the corrosion management group with expertise in microbiologically influenced corrosion (MIC) at DNV GL in Columbus OH, USA. Dr. Kotu received her Ph.D. in Chemical Engineering along with Safety Engineering certification from Texas A&M University. Before joining DNV GL, Dr. Kotu worked as an intern in the corrosion labs at ExxonMobil Upstream Research Company and Baker Hughes, a GE company. In her current role, she supports internal corrosion projects with a focus on MIC and microbiology services such as MIC threat assessments and biocide screening and optimization. She has also instructed a course on "Introduction to MIC" at Appalachian Underground Short Course in May 2019.