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Review of Testing Methods and Standards for Oilfield Corrosion Inhibitors

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ABSTRACT

Laboratory methodologies to evaluate corrosion inhibitors for oilfield application are reviewed. The importance of establishing the factors that influence the hydrodynamic parameters in the laboratory methodologies is discussed. Currently available standards for evaluating and qualifying oilfield corrosion inhibitors are presented. Areas where development of new standards is needed are identified. A methodology is presented to predict the performance of corrosion inhibitors in the field, based on laboratory data.

KEYWORDS

Inhibitors, standards, rotating cage, jet impingement, prediction

INTRODUCTION

Because of the complexity involved in evaluating corrosion inhibitors, the availability of sophisticated methodologies to evaluate inhibitors, the cost associated with screening and using inhibitors to control internal corrosion of pipelines, the widespread use of inhibitors, and to manage risk where public safety is involved, it is important to standardize the methodologies that are used to evaluate and qualify inhibitors. Any laboratory methodology that is being considered for the evaluation of inhibitor performance in a particular system should itself be assessed for the effectiveness with which the significant variables can be simulated. In this paper, the laboratory methodologies to evaluate corrosion inhibitors for oil field application are reviewed. The importance of establishing the factors that influence the hydrodynamic parameters of the laboratory methodologies is presented. The need to develop standardized procedures for the laboratory methodologies to evaluate corrosion inhibitors is identified. A methodology is also presented to predict the performance of corrosion inhibitors in the field, based on laboratory data.

LABORATORY METHODOLOGIES

The Working Party on Inhibitors of the European Federation of Corrosion has produced a state-of-the-art report on laboratory methodologies for inhibitor evaluation **[1]**. The methodologies for evaluating inhibitors in oil and gas technologies, e.g., in production, transmission, and storage, often require the use of two phase oil-water fluids, in addition to the corrosive gas. The methodologies described in the report are based on rotating probes in autoclave, wheel, and bubble tests.

Recognizing that it may not be necessary to control all the parameters in laboratory tests that are intended for screening, evaluation and selection of inhibitors, it is reported that inhibitors selected based on the wheel test and kettle test (linear polarization resistance [LPR]) in the laboratory performed consistently well in pipeline and downhole tubular applications in the field **[2]**.

Based on the data presented by Garber et al. **[3]** the better methodologies for inhibitor evaluation are high-pressure (500 psi) linear polarization, flow loop test, and rotating electrode (higher speeds). In these tests, only a few inhibitors resulted in more than 90% inhibition efficiency, and many inhibitors resulted in less than 60% inhibition.

In the round-robin test of LPR (kettle test) and wheel tests for the performance ranking of three corrosion inhibitors under identical test conditions **[4],** the most repeated ranking of three inhibitors was obtained by 5 out of the 9 laboratories for the wheel test and by 6 out of the 8 laboratories for LPR (kettle test), indicating that better reproducibility is obtained in the kettle test (75% reproducibility) than the wheel test (55% reproducibility).

NACE has developed a technical committee report on the wheel test method for evaluating film persistent inhibitors for oil field applications **[5]**. The wheel test is described as versatile because the procedure may be adjusted to test a variety of inhibitors and may be performed on various types of test specimen. Unfortunately, the low level of reproducibility of the test results offsets the advantages of its versatility. Based on the experimental results, Hausler et al **[6]**. concluded that the wheel test does not differentiate inhibitors.

NACE has also developed a state-of-the-art report on controlled-flow laboratory corrosion tests **[7]**. This report (i) describes test methods (including rotating disk, rotating cylinder, jet impingement, and flow loop) that give quantitative results; (ii) provides information on selecting the most appropriate test method; and (iii) provides information on interpreting test results.

An ASTM Standard describes three methodologies, rotating cylinder electrode (RCE), rotating cage (RC), and jet impingement (JI), for inhibitor evaluation **[8]**. These methodologies are compact, inexpensive, hydrodynamically characterized, and scalable, i.e., they can be carried out under various flow conditions. The Standard also describes methodologies to evaluate several secondary inhibitor properties, including water/oil partitioning, solubility, emulsification tendency, foam tendency, thermal stability, toxicity, and compatibility with other additives/materials.

Liu et al. **[9, 10]** developed relationships between rotating disk, pipe flow and corrosion. Based on experiments using three hydrodynamic systems in a flow-through pipe channel, an annular flow channel, and a rotating cylinder system, Chen et al. **[11]** concluded that the corrosion rate measured in all hydrodynamic systems is independent of the geometry involved, implying that transfer of corrosion data from one geometry to another can be accomplished based on the mass transfer coefficients. On the other hand, in the absence of a surface film, transfer of corrosion data can be achieved based on mass transfer coefficient in the diffusion boundary layer.

Chesnut found that the kettle test is an effective tool for identifying inhibitors which are clearly unsuitable for use, but that it does not differentiate good inhibitors **[12]**. Chesnut et al. obtained good correlation between RCE and flow loop tests at a shear stress of about 40 Pa. They further showed that the ranking of inhibitors at shear stresses less than 40 Pa can be different from the ranking at higher shear stresses.

Carrying out flow loop and RCE experiments simultaneously, Nesic et al. **[13]** found that, in the absence of surface films, corrosion rates measured in flow loop and RCE experiments correlate under the same mass transfer conditions (at 2 m/s velocity) and at room temperature. For similar experiments at higher temperatures, corrosion rates in the RCE experiments were higher than those measured in the flow loop.

Denpo and Ogama **[14]** compared the corrosion rates of steel pipe with different RCE speeds. The diameter, test solution, temperature, and dissolved oxygen content were identical in both experiments. Based on the similarity of solutions obtained for mass transfer with pipe flow and the rotating electrode, the rotating velocity was converted to the equivalent velocity in the pipe. On that basis, the corrosion rate of the pipe was equal to the corrosion rate of the electrode. The corrosion rate of the rotating electrode obtained electrochemically was used to predict the corrosion rate of the pipe at the equivalent velocity. The predicted corrosion rate was in agreement with the measured corrosion rate.

A stirred autoclave has been successfully modified by Milin to an RCE autoclave to meet the requirement of oil field inhibitor assessment with consideration of flow-induced corrosion **[15]**. The corrosion rates were measured electrochemically.

Abayarathna et al. **[16]** studied the performance of corrosion inhibitors in the laboratory using RCE, rotating cylinder in autoclave (RCA), and flow loop and, in the field, using weight loss and electrical resistance probes. During the RCE tests, the chemicals were tested at low concentration, 5 ppm, since the conditions being simulated were relatively mild. The performance of the chemicals was difficult to differentiate if higher concentrations were used in this test. However, in the aggressive environments established in the RCA and flow loop tests that simulated the field conditions, the chemicals were evaluated at higher concentrations, 25 and 75 ppm, respectively. Although one- to-one correlation of the field and laboratory data was difficult, the field and laboratory data were consistent. The chemicals that performed well in the laboratory also performed well in the field.

Dawson et al. **[17]** obtained identical results from the rotating cylinder electrode and from the jet impingement for the same wall shear stress. Based on the results, they concluded that the shear stress can be used as a fundamental test parameter for inhibitor evaluation under turbulent flow conditions. They cautioned that the use of fluid velocity to describe the hydrodynamic conditions is inadequate unless the geometry or test apparatus dimensions are also specified. In addition, they recommended that the actual hydrodynamic conditions in the tests must be known (e.g., fully developed flow, developing flow or flow separation) in order to compare with other tests and to predict inhibitor performance in practical operating systems. The maximum wall shear stress achieved in RCE and jet impingement were 28 Pa; and 1300 Pa respectively.

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Based on the data from RCE, JI, and pipe of carbon steel in brines containing CO_2 under conditions where a protective scale was not formed, Efird et al. **[18]** concluded that flow-accelerated corrosion of jet impingement with a test ring at $r_{jet, radial}/r_{jet} = 3$ correlated with pipe flow when plotted as a function of wall shear stress, but not at $r_{jet, radial}/r_{jet} = 5$, where $r_{jet,radial}$ is the radial distance of jet measured in terms of radius of the jet and r_{jet} is radius of jet. On the other hand, RCE did not correlate with pipe flow as a function of wall shear stress or mass transfer for carbon steel in brines containing CO_2 under conditions where a protective scale was not formed.

Orazem et al. **[19,20]** studied the impinging jet system in the stagnation region. This stagnation region is observed directly beneath the jet. With appropriate experimental design, the critical shear for removal of a protective film can be obtained by measuring the profile of an electrode subjected to a given jet velocity. Schmitt et al. **[21]** developed a submerged jet for experiments up to 180°C and 100 bar pressure under sour gas conditions. Provisions were made to vibrate the probe if desired with frequencies up to 100 Hz and amplitudes up to 120 μm.

Both flow loop and jet impingement techniques provided similar trends of increasing corrosion rates with increasing wall shear stress **[22]**. Bartos **[23]** studied two inhibitors, one water-soluble and the other oil-soluble, in bubble, jet impingement and flow loop experiments. The water-soluble inhibitor performed better than the oil-soluble inhibitor in all three tests. The poorer performance of oil soluble inhibitor in the bubble test is explained as due to non-agitation, and consequently, lack of partitioning.

Using the rotating cage, Hausler, Stegmann et al. **[24-26]** have shown that the corrosion morphology of both mesa and pitting corrosion observed in the field can be reproduced. They claim that the predictive capability of the rotating cage lies in the measurement of local penetration rates. They found that the general corrosion rates increased sharply above about 93 to 121°C (200 to 250°F), but that localized corrosion depended to a large extent on the alloy composition of the steel.

Comparing the results from the rotating cage with those from the flow loop, Schmitt et al. **[27]** concluded that the rotating cage created more severe flow conditions, and hence, higher localized corrosion rates than flow loop tests. They concluded that, because of the easy fabrication of the rotating cage and because microturbulent flow is generated, the rotating cage can be recommended for screening corrosion inhibitors.

Based on the quantitative comparison of general and pitting corrosion rates obtained in the laboratory with those obtained in pipe in the operating fields, Papavinasam el al **[28]** concluded that

- Rotating cage **(Fig.1)** is the top-ranked methodology based on the severity of the corrosion conditions simulated. It is one of the methodologies that generated large and deep pits. The rotating cage provides a rigorous test for corrosion inhibitors to pass. The apparatus is simple, compact, has a well-established flow pattern, and its hydrodynamic flow is reasonably well characterized.

- Jet impingement, RCE, and RDE may be used to screen inhibitors. These methodologies have well-established flow patterns that are well-characterized hydrodynamically. However, they do not simulate the extreme field operating conditions, e.g., pitting corrosion of coupons at the bottom of the pipe.

- The wheel test, bubble test, and static test are the lowest-ranked methodologies; i.e., results obtained using these tests showed the poorest correlations with data obtained from operating pipelines.

Papavinasam **[29]** formulated empirical equations for determinating wall shear stresses at coupons in the rotating cage. Schmitt et al **[30]** determined the local wall shear stresses using microelectrodes and found that the wall shear stress increases with the rotation speed of the cage and that the local wall shear stresses are strongly influenced by the liquid viscosity.

Matsumura et al. **[31]** developed a jet-in-slit apparatus; Thomasan et al.**[32]** described a rotating cell consisting of a glass cylindrical vessel containing layers of sand, salt water, oil, and carbon dioxide (gas phase). The rotator is a grooved (to achieve greater shear) nylon cylinder. In a study of electroplating, the influence of additives, temperature variation, and current density on the properties of the deposit were investigated by using a rotating-cone electrode ($RC_{one}E$) **[33]**. This type of electrode was used because of the well-defined current distribution and the reproducible and controlled mass-transfer performance at fixed rotation speeds. Because electroplating (cathodic) and corrosion (anodic) processes are both electrochemical in nature, in theory, the $RC_{one}E$ could be used for inhibitor evaluation.

STANDARDS FOR LABORATORY METHODOLOGIES

Factors that make laboratory evaluation of corrosion inhibitors for application in oil fields difficult include the large number of laboratory methodologies that are available; the several correlations that can be used to convert corrosion rate (and hence, inhibitor efficiency) from one geometry to another; the vast variation of field operating conditions; and the impossibility of reproducing in the laboratory all field operating conditions. In order for pipeline operators to be able to use the appropriate laboratory methodologies for evaluating inhibitor performance in a particular system, uniform international standards should be developed by organizations such as NACE, ASTM, and ISO in tandem. These standards need to be based on round robin experiments that involve participation from corrosion inhibitor suppliers, users, oil field companies, R&D laboratories, government, and other stake-holders. The development and usage of such standards will benefit all those involved as a result of increased effectiveness of corrosion inhibitors, lower cost, fewer field failures, and increased safety.

PREDICTION OF FIELD PERFORMANCE FROM LABORATORY DATA

Users of corrosion inhibitors often face the task of selecting the best performing inhibitors for a particular application in a rapid and cost-effective manner **[34]**. In addition to corrosion protection, the inhibitor should be compatible with other chemicals, such as scale inhibitors and biocides, and should meet environmental and safety requirements. Laboratory tests are usually, but not exclusively, used to select inhibitors for field use. The ideal test should reproduce all the relevant parameters of the intended application, including pressure, temperature, compositions

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(steel, solids, liquid, and gases), and flow. Hydrodynamic relationships can be used to correlate inhibitor performance in the laboratory and in the field **[28]**.

Traditional ranking and selection of corrosion inhibitors is generally based on percentage inhibition calculated based on the formula shown in Eqn. 1

$$\% Inhibition = \frac{[C.R.]_{No\ Inhibitor} - [C.R.]_{Inhibitor}}{[C.R.]_{No\ Inhibitor}} x100 \qquad \text{Eqn.1}$$

Where $(C.R)_{No\ Inhibitor}$ is the corrosion rate in the absence of corrosion inhibitor and $(C.R)_{Inhibitor}$ is the corrosion rate in the presence of corrosion inhibitor. Inhibitors are generally ranked based on the values of % inhibition. The inhibitor with highest inhibition is usually selected.

On the other hand, the selection of inhibitors should also take into account the following:

1. Laboratory methodologies;
2. Extent to which field conditions are simulated in the laboratory; total pressure and partial pressures of acid gases, i.e., H_2S and CO_2;
3. Temperature;
4. Composition of gas, oil, and brine;
5. Cost; and
6. Concentration of corrosion inhibitor.

A four-step process can be used to predict the performance of corrosion inhibitors in the field, based on the laboratory data. This process can be used to evaluate corrosion inhibitors.

Step 1: Determination of Pipeline Operating Conditions: The hydrodynamic condition of a pipeline is based on: Production rates of oil, gas, and water; temperature; pressure; and pipe inclination.

Step 2: Selection of a Laboratory Methodology: It is assumed that the corrosion rates in the laboratory and in the field are similar when the hydrodynamic parameters are the same. To predict the corrosion rate, and hence, the inhibitor efficiency in the field, the laboratory experiments should be carried out under the hydrodynamic conditions of the pipe in the field. The laboratory methodology that is selected for evaluating corrosion inhibitors depends on the pipeline operating conditions, specifically the wall shear stress (WSS). Recommended methodologies are:

Jet impingement (JI)	(for WSS > 200 Pa);
Rotating cage (RC)	(for WSS between 200 and 20 Pa),
Rotating cylinder electrode (RCE)	(for WSS between 20 and 5 Pa) and
Rotating disk electrode (RDE)	(for wall shear stress <5 Pa).

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These four methodologies are called "high-shear laboratory methodologies". The methodologies can be hydrodynamically characterized and they can be operated under various hydrodynamic conditions.

Step 3: Determination of Operating Conditions for the Laboratory Methodologies: Simply carrying out laboratory methodologies under the hydrodynamic condition of the pipe in the field does not guarantee useful results. Several other parameters also influence the correlation of data between laboratory and pipe **[35]**

Step 4: Selection of Corrosion Inhibitors: The selection of corrosion inhibitor is based on Eqn. 2, which includes the influence of both direct and indirect effects.

$$IR = \frac{[C.R.]_{Mean} + [C.R.]_{std} + [Cost] + [Concn.]}{[A] + [B\} + [C] + [D] + [E] + [F]} \qquad \text{(Eqn.2)}$$

Where IR Individual ranking

$[C.R]_{Mean}$ is the average or mean corrosion rate

$[C.R]_{std.}$ is the standard deviation of corrosion rate (It is a measure of the reproducibility of the test data)

[Cost] is the cost of corrosion inhibitor per ppm

[Concn.] is the concentration of corrosion inhibitor in ppm

[A] is the effect of laboratory methodology

[B] is the effect of wall shear stress

[C] is the effect of temperature

[D] is the effect of total pressure

[E] is the effect of H_2S partial pressure

[F] is the effect of CO_2 partial pressure

Inhibitor selection is based on the individual rankings. The top-ranked inhibitor is the one that has the lowest individual ranking.

SUMMARY

Laboratory methodologies are critical to optimize inhibitor selection for pipeline applications. Several laboratory methodologies are available including the rotating cage, rotating cylinder, jet impingement, rotating disc, the wheel test, and the bubble test.

Development of international standards (after round robin experiments) is imperative to select and use laboratory methodologies effectively to evaluate oil field corrosion inhibitors.

To use laboratory methodologies effectively, the various parameters of the pipe under field operating conditions, such as production rate, water/oil ratio, pressure, and temperature, should be determined.

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To select corrosion inhibitors both cost-effectively and reliably from the laboratory experiments, both direct (compositions, pressure, and temperature) and indirect (flow) variables in the field should be simulated in the laboratory.

Several approximations are involved in the development of hydrodynamic relations to relate the corrosion rate measured in one geometry to that predicted in another geometry.

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Fig.1: Photograph of Rotating Cage