## EROSION-CORROSION IN DISTURBED LIQUID/PARTICLE FLOW

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## This thesis titled

## EROSION-CORROSION IN DISTURBED LIQUID/PARTICLE FLOW

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#### Abstract

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Erosion-corrosion occurs in pipelines that transport both corrosive liquids and erosive solid particles. This study has tested the erosion-corrosion behavior of mild carbon steel under conditions where there is no protective iron carbonate film. High and low salt concentrations were studied in order to determine the effect of salt concentration on the erosion-corrosion process.

The effect of erosion-corrosion on mild steel was tested under disturbed flow by using a specially designed test section consisting of three flow alterations: a flow constriction, protrusion, and expansion. Under the tested conditions it was found that there is no synergistic effect between erosion and corrosion and that for an unprotected base metal the rate of metal loss is equal to the sum of erosion loss and corrosion loss. The higher salt concentration led to a lower corrosion rate and erosion rate but did not affect the interaction between erosion and corrosion.

Approved:

Srdjan Nesic

Professor of Chemical and Biomolecular Engineering

## То

my parents, Tony and Kim Addis

my brother, Kyle Addis

my grandparents, Barton & Donna Addis and Wayne & Carol Harmon

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## **Chapter 1: Introduction**

Material degradation in industrial pipelines occurs through two main processes: *corrosion* and *erosion*. If both erosion and corrosion occur at the same time there is a chance that a synergistic effect may cause the amount of metal loss to be greater than the sum of metal loss due to erosion and corrosion. Corrosion is a chemical or electrochemical degradation process by which electrons from the target metal are lost to corrosive species in the process fluid and the remaining metal cation is left to be dissolved into the fluid or to form a protective film. Erosion occurs due to the impact of particles on the surface of pipe walls which mechanically removes material from the target metal surface. Industrial pipelines which transport corrosive species and solid particles are subject to *erosion-corrosion*. For highly used industrial materials such as carbon steel it is important to know how the material will wear in erosion-corrosion environments as there may be interactions between the two mechanisms leading to a synergistic effect<sup>1</sup>.

## **Chapter 2: Literature Review**

#### **2.1 Pure Erosion**

#### 2.1.1 The Mechanisms of Erosion: Brittle versus Ductile

Brittle erosion involves the impact of particles on the surface of the metal which cause cracks to propagate down through the metal surface<sup>2</sup>. These cracks propagate throughout the metal and lead to chunks of metal being chipped away by repeated impact of particles<sup>2</sup>. This type of erosion occurs because the metal has little tendency to strain which causes it to fracture<sup>2</sup>.

As opposed to brittle erosion, ductile erosion involves the impact of particles which plastically deform the surface of the metal<sup>2</sup>. The repeated impact of particles forges hardened platelets on the surface of the metal and this creates a transient response in the erosion process and this process is termed *the platelet formation theory of erosion*<sup>2</sup>. Work hardening of the metal subsurface occurs for ductile metals undergoing erosion due to the extensive plastic deformation at the metals surface<sup>2</sup>. This leads to a transient response in the erosion process for a ductile metal at which time the surface of the metal is being work hardened and platelets are being formed<sup>2</sup>. The steady-state response occurs after the platelets are formed<sup>2</sup>.

The process of erosion for a ductile metal, such as carbon steel, occurs by the followings steps. First a single particle is impinged upon the surface of the target metal<sup>2</sup>. This initial impingement creates a crater in the metal surface which removes little to no material<sup>2</sup>. Subsequent impacts on the metal surface forge platelets from the deformations caused by the extrusion process<sup>2</sup>. During this time there is also work-hardening of the

metal subsurface occurring<sup>2</sup>. The impact of the particle on the surface of the metal carries a much greater force than that required to form platelets<sup>2</sup>. This energy is transferred into the subsurface of the metal where the metal is cold worked, creating a less ductile subsurface below the platelet surface<sup>2</sup>. The result of platelet formation is a harder surface which now allows particles to impinge and chip away at the platelets<sup>2</sup>.

The mechanism of erosion is dependent on the type of material that is being eroded. Harder metals will tend to erode by brittle erosion mechanisms and softer metals will erode by ductile erosion mechanisms<sup>2</sup>. It should be noted that it is possible to have a metal where both mechanisms are occurring simultaneously<sup>2</sup>. One example of this would be in alloyed metals with very coarse grain structure<sup>2</sup>. It could be that one of the components of the alloy erodes in a ductile manner and one in a brittle manner.

#### 2.1.2 Particle Impact and Velocity

It would be expected that as the velocity of the particle increases the greater the erosion damage upon impact and much experimental evidence from researchers such as Levy<sup>2</sup>, Nesic and Postlethwaite<sup>3</sup>, and Salama<sup>4</sup> have shown this. The faster a particle moves the more kinetic energy it has. According to the equation for kinetic energy: kinetic energy =  $\frac{1}{2}$  MV<sup>2</sup>, where M = mass and V = velocity. Therefore, it would be expected that as the velocity increases the erosion rate would increase and most likely in a non-linear manner. If the erosion rate is proportional to the kinetic energy of the particle then it would be likely that erosion rate is proportional to the square of the velocity. However, only part of the velocity component is lost upon impact on the metal

surface because the particle is under constant movement due to flow and therefore not all of the particles energy is transferred to the metal surface<sup>2</sup>.

It should be noted that increasing the velocity increases the erosion rate for both brittle and ductile metals although they erode by different mechanisms<sup>2</sup>. Surface analysis for 1018 carbon steel shows that erosion tests using an impingement jet with velocities ranging from 15-130 m/s show that the mechanism of platelet formation occurs over this entire range of velocities<sup>2</sup>. This suggests that the mechanism of erosion does not change with increasing velocity for a ductile metal.

#### 2.1.3 Particle Size

Nesic and Postlethwaite<sup>3</sup> found that at a velocity of 20 m/s the erosion rate remains unchanged as long as erodent particles are in the range of 175 – 900 microns. The reason for this is because as the particle increases in size the surface area of the particle increases as well<sup>2</sup>. This means there is more particle surface which will contact the metal surface upon impact which will lead to the force upon impact being spread out over a wider area<sup>2</sup>. This will lead to a shallower depth of penetration and therefore the erosion damage will not significantly change even though the mass and size of the particle is greater<sup>2</sup>. Another reason for this phenomenon is that as particles increase in size there are more particle interactions which may inhibit certain particles from contacting the metal surface<sup>3</sup>. Larger particles at the metal surface may hinder other particles from coming in contact with the surface<sup>3</sup>. Particles less than 175 micron yielded lower erosion rates than those in the range of 175-900 microns given above<sup>3</sup>. This is because the smaller particles have less kinetic energy and therefore cause less erosion damage<sup>2</sup>.

#### 2.1.4 Particle Shape

Sharper particles tend to have higher erosion rates associated with them than duller, more spherical shaped particles<sup>2,3</sup>. This is because sharper particles can penetrate deeper into the metal surface<sup>2,3</sup>. Sharper particles also have a smaller contact area at the metal surface so the force generated is much larger<sup>2</sup>. A spherical shaped particle has little penetration power and the exposed area is greater which means there is the same amount of force exerted as a sharper particle but over a larger area of the metal surface<sup>3</sup>. Although smooth, spherical particles do erode metals, the erosion rates are significantly less than sharper particles such as sand<sup>3</sup>.

#### 2.1.5 Solids Concentration

The concentration of solids in the erodent significantly affects the erosion rate of a metal up to a point where more solids hinder the erosion rate due to particle interactions<sup>2</sup>. This is similar to the particle size affect where the particle can only erode the metal if it can reach the surface. Nesic and Postlethwaite<sup>3</sup> concluded that at higher concentrations only a fraction of the particles are actually contacting the metal surface. Therefore most erosion results show that increasing the solids concentration has a non-linear increasing trend up to a point where more solids will not significantly alter the erosion rate of the metal<sup>2,3</sup>.

#### 2.1.6 Measuring Erosion Rate

The simplest method for measuring the erosion rate of a material is by measuring the weight loss of a metal sample before and after being subjected to an erosive condition<sup>2</sup>. In this study the erosion rates will be calculated using the following equation<sup>7</sup>:

Metal Loss Rate = 
$$\frac{W}{\rho \cdot A \cdot T}$$
 Eq. 1

Where W is the weight loss,  $\rho$  is the density of the metal, A is the exposed area, and T is time. This calculation can be used for corrosion and erosion weight loss data to determine the metal loss rate.

#### 2.2 Pure Corrosion

#### 2.2.1 Basics of CO<sub>2</sub> Corrosion

Corrosion of mild steel in an aqueous carbon dioxide solution occurs through the anodic reaction of iron(III) going to iron(II) according to the following reaction<sup>7</sup>:

$$Fe \rightarrow Fe^{2+} + 2e^{-}$$
 Eq. 2

The electrons are consumed by the cathodic reactions, which for aqueous carbon dioxide are hydrogen reduction and carbonic acid reduction<sup>7</sup>:

$$2H^+ + 2e^- \rightarrow H_2$$
 Eq. 3

$$H_2CO_3 + e^- \rightarrow H + HCO_3^-$$
 Eq. 4

The amount of  $H_2CO_3$  in the solution is proportional to the partial pressure of  $CO_2$ . The  $CO_2$  present in the system hydrates to give carbonic acid ( $H_2CO_3$ ) according to the following equation<sup>5</sup>:

$$CO_2 + H_2O \Leftrightarrow H_2CO_3$$
 Eq. 5

For acidic solutions, the reduction of hydrogen is the most important cathodic reaction<sup>5</sup>. In CO<sub>2</sub> solutions, at a pH range of 4 to 6, the direct reduction of carbonic acid becomes significant<sup>5</sup>. Therefore, a solution with aqueous CO<sub>2</sub> is more corrosive than a solution without CO<sub>2</sub> at the same pH due to the additional cathodic reactions occurring at the metal surface<sup>5</sup>. For solutions without CO<sub>2</sub> the only cathodic reaction is hydrogen

reduction which can cause a rapid increase in the  $pH^5$ . The undissociated  $H_2CO_3$  in the solution acts as a buffer<sup>5</sup>.

If the concentration of  $Fe^{2+}$  in the solution reaches the solubility limit then iron carbonate (FeCO<sub>3</sub>) will precipitate on the surface of the metal<sup>5</sup>. The solubility limit (K<sub>sp</sub>) is a function of temperature and ionic strength<sup>5</sup>. The formation of FeCO<sub>3</sub> on the surface of the corroding metal acts as a diffusion barrier to corrosive species and therefore reduces the corrosion rate of the metal<sup>5</sup>. These films, however, are generally only protective above a pH of around 6.0 or larger and at lower pH the films formed on the metal surface are generally non-protective and show no reduction in the corrosion rate<sup>5</sup>. For this reason the pure corrosion experiments and the erosion-corrosion experiments are carried out at a pH of 4.0 to ensure that no protective films will form.

#### 2.2.2 Measuring Corrosion Rate

The corrosion process can be monitored in order to determine the actual rate of metal loss occurring on the target metal. Electrochemical measurements can be made in order to determine the actual current flowing through the target metal. This current is called the corrosion current and is directly proportional to the corrosion rate<sup>7</sup>. As the potential between the corroding metal and the cathode gets larger the corrosion current increases up to a point where the current no longer changes with increasing potential difference<sup>7</sup>. This point is called the limiting current and is limited due to the transfer of charges from the metal to the cathode<sup>7</sup>. When the limiting current is reached there are more electrons at the metal surface than the cathodic specie can consume<sup>7</sup>.

The corrosion rate can be obtained through the use of a potentiostat<sup>7</sup>. A potentiostat controls the voltage difference between a working electrode and a reference electrode<sup>7</sup>. The working electrode is the metal which is being tested and the potentiostat controls the potential of the metal and measures the current passing through the working electrode. The reference electrode is used to measure the potential of the working electrode<sup>7</sup>. Since potential is relative there needs to be a stable reference electrode that maintains the same potential throughout the experiment<sup>7</sup>. The electrons flow from the working electrode to the counter or auxiliary electrode which completes the circuit<sup>7</sup>.

In order to test the corrosion rate of a metal, the corrosion potential of the target metal is tested first. The corrosion potential is the potential of the non-polarized target metal in the corrosive solution<sup>7</sup>. A potentiodynamic sweep polarizes the target metal at a given voltage above and below the corrosion potential<sup>7</sup>. The current is monitored at each incremental change in the potential difference. At potentials lower than the corrosion potential (more negative) the sweep shows the current versus potential curve for the cathodic reactions and at potentials higher than the corrosion potential (more positive) the sweep shows the current versus potential (more positive) the sweep shows the current versus potential curve for the anodic reaction<sup>7</sup>. The corrosion current can be measured directly from the plot of the potentiodynamic sweep by extending the linear portions of the anodic and cathodic curves until they cross at the corrosion potential<sup>7</sup>. The current at which these two lines intersect is the corrosion current<sup>7</sup>.

When the potential of an electrode is plotted as a function of the logarithm of current density then this is called a Tafel plot<sup>7</sup>. The straight line portions of the curves which can

be extrapolated to determine the corrosion current are called the Tafel lines<sup>5</sup>. The slope of the Tafel lines is defined as the Tafel slope<sup>7</sup>. The equation for each Tafel line is given as<sup>7</sup>:

$$\eta = \frac{2.3RT}{\alpha nF} \log i_{o} - \frac{2.3RT}{\alpha nF} \log i$$
 Eq. 6

Where  $\eta$  is the overpotential, R is the ideal gas law constant, T is temperature,  $\alpha$  is the cathodic electron transfer coefficient, n is the number of equivalents exchanged, and F is Faraday's constant (96,500 coulombs/equivalent),  $i_0$  is the exchange current density, and i is the current at the given overpotential<sup>7</sup>.

As the overpotential is shifted more negatively then the cathodic reaction or reactions will be accelerated and the anodic reaction will be decreased<sup>7</sup>. The difference between the increase in the cathodic reduction rate and the decrease in the anodic oxidation rate is equal to the applied current<sup>7</sup>:

$$i_{app,c} = i_c - i_a$$
 Eq. 7

As the cathodic overpotential increases there is a point where the anodic current density becomes insignificant when compared to  $i_c$  and therefore the straight line portions of the Tafel plot are seen<sup>7</sup>. This linear behavior at high cathodic overpotentials is referred to as Tafel behavior<sup>7</sup>.

Using the polarization resistance method the corrosion current can be directly measured from polarization data<sup>7</sup>. For small deviations in the overpotential (up to 20 mV from the corrosion potential) the plot of overpotential versus applied current is linear<sup>7</sup>. The slope of this line is the polarization resistance for the electrode<sup>7</sup>:

$$Rp = \frac{\Delta E}{\Delta i_{app}}$$
 Eq. 8

The corrosion current can be measured directly from the polarization resistance using the proportionality constant. The proportionality constant is calculated from the anodic and cathodic Tafel slopes,  $\beta_a$  and  $\beta_c$ , from the following equation<sup>7</sup>:

$$B = \frac{\beta_a \beta_c}{2.3(\beta_a + \beta_c)}$$
Eq. 9

The corrosion current can then be calculated from the proportionality constant and the polarization resistance from the equation<sup>7</sup>:

$$i_{corr} = \frac{B}{Rp}$$
 Eq. 10

#### 2.2.3 Electrochemical Impedance Spectroscopy (EIS)

In an electrochemical cell there is a solution resistance that creates a voltage drop along the path of the current<sup>7</sup>. Therefore, the potential measured between the reference and working electrode has an error associated with it due to the resistance of the solution. This solution resistance is increased as the reference electrode is moved farther from the working electrode<sup>7</sup>. In corrosion measurement techniques there is always going to be some distance between the reference and working electrode and thus solution resistance will cause an inaccuracy in the corrosion rate measurement. All electrochemical cells have a solution resistance; however, for some testing this solution resistance may be insignificant when compared to the overall polarization resistance of the working electrode. If the solution resistance is significant then it is subtracted from the polarization resistance measured on the working electrode. To correct for solution resistance the reference and working electrode can be considered a capacitor with the solution between them acting as the dielectric<sup>7</sup>. When a direct current is passed through an electrochemical cell the resistance through the system is a sum of the solution resistance (capacitance) and the polarization resistance of the working electrode<sup>7</sup>. High frequency alternating currents passed through the electrochemical cell will directly measure the solution resistance of the electrochemical cell by measuring the resistance of the equivalent ohmic resistive element<sup>7</sup>. At very low alternating current frequencies the current is more like a direct current and the resistance measured is once again the sum of the ohmic solution resistance and the polarization resistance of the working electrode can be determined by taking the difference between the low end alternating frequency currents and the high frequency currents<sup>7</sup>. This procedure for measuring solution resistance is called electrochemical impedance spectroscopy (EIS).

#### 2.2.4 Weight Loss Calculation

The corrosion rate can also be calculated from weight loss data. The equation used to calculate the weight loss corrosion rate is the same as Equation 1 which can be used to calculate any metal loss rate from weight loss data.

#### 2.3 Erosion-Corrosion

Many studies have shown interaction between wear and corrosion<sup>6</sup>. The purpose of studying erosion and corrosion simultaneously is to determine in which situations there will be a synergistic effect. Excessive wear on pipelines due to erosion and corrosion interactions pose a major threat to the oil and gas industry as the amount of additional

weight loss to expect from synergy has not been well quantified<sup>6</sup>. The interaction between erosion and corrosion in pipelines containing a protective film or inhibitor is also of much importance to industrial pipelines as there can be even more synergistic effect in the erosion-corrosion process due to stripping of inhibitor or corrosion product by solid particles which would greatly increase the local corrosion rate and may induce localized corrosion<sup>10</sup>. Erosion-corrosion is also a term given to the wear of corrosive products (films) due to the shear stress of the fluid acting on the surface of the film<sup>2</sup>. It should be noted that at high enough velocities, liquid particles have enough energy to cause erosive wear of pipelines, especially at high impact angles where the direct impact of the liquid will cause a greater force to be applied to the pipe<sup>2</sup>.

#### 2.3.1 Determining Synergism between Erosion and Corrosion

The ASTM Standard G 119-04 was issued in 2004 as a standard procedure for determining the amount of synergy in an erosion-corrosion experiment<sup>9</sup>. The procedure calls for erosion-corrosion tests to be carried out to determine the total weight loss in erosion-corrosion<sup>9</sup>. The erosion-corrosion results account for wear and corrosion metal loss as well as the additional material loss due to any synergistic effect<sup>9</sup>.

The corrosion rate under erosion-corrosion should also be measured in order to determine the increment in corrosion during erosion-corrosion<sup>9</sup>. This measurement will be taken using an electrochemical technique such as potentiodynamic sweep<sup>9</sup>. Pure corrosion experiments should then be carried out to determine the corrosion rate with no wear<sup>9</sup>.

Pure erosion experiments should be carried out under cathodic protection to determine the pure amount of metal loss due to wear<sup>9</sup>. In this study cathodic protection was not done by increasing the electrical current density but by removing the corrosive species, H<sup>+</sup> and CO<sub>2</sub>, by adjusting the solution to a neutral pH of 7 and using nitrogen gas to purge the system. This did not completely eliminate the corrosion rate, but reduced it to an average corrosion rate of around 0.1 mm/yr. Due to the number of coupons used in this experiment it was not feasible to place all of them under cathodic protection by increasing the electric current density throughout the entire experiment. Electrochemical measurements were made during erosion tests and these results were subtracted from the weight loss erosion rate in order to obtain a pure erosion rate.

The total amount of synergy can be calculated according to the following procedure given by ASTM  $G119 - 041^9$ :

Determine the amount of synergy, S, using the results from erosion-corrosion total weight loss,  $W_{ec}$ , pure corrosion rate,  $C_o$ , and pure erosion rate or wear rate,  $W_o$ :

$$S = W_{ec} - W_o - C_o \qquad \qquad \text{Eq. 11}$$

This synergy is composed of the increment in corrosion due to erosion,  $\Delta C_w$ , and the increment in erosion due to corrosion,  $\Delta W_c$ :

$$S = \Delta C_w + \Delta W_c \qquad \qquad \text{Eq. 12}$$

The increment in corrosion due to erosion can be calculated by subtracting the pure corrosion rate loss from the electrochemically measured corrosion rate under erosioncorrosion conditions:

The increment in erosion due to corrosion can then be calculated using the following calculation:

#### 2.3.2 Previous Study in Erosion-Corrosion

A previous erosion-corrosion study was carried out by Ramakrishna Malka using a similar flow loop and the same operating conditions as current testing. Malka's results were from a study published in 2005 where the synergistic effect between erosion and corrosion for carbon steel in aqueous carbon dioxide was studied<sup>6</sup>. The goal of the current project is to carry on the testing procedure as Malka and to continue researching the erosion-corrosion process in a re-circulating flow loop. This work was proposed because there is no clear understanding of the interaction between erosion and corrosion under realistic flow conditions<sup>6</sup>. The unique design of the test section and testing procedures allows for the study of interactions in realistic disturbed pipe flow conditions through the use of in-situ, localized, electrochemical measurements and weight loss measurements in order to determine the effect of erosion on corrosion and corrosion on erosion<sup>6</sup>.

Malka's results showed a significant synergy existing between erosion and corrosion under disturbed liquid flow containing 2 wt% silica sand with an average particle size of 275 microns<sup>6</sup>. The test conditions for Malka's experiments are the same as the testing conditions used in this study with the exception that Malka only performed tests at 2 wt% sand and 1 wt% salt. Malka's results showed that erosion enhances corrosion and corrosion enhances erosion attributing to a significant overall synergy<sup>6</sup>. The results also showed enhancement of erosion by corrosion was the dominant process in the synergism under the conditions studied<sup>6</sup>.

In attempt to repeat Malka's results it was found that under the same conditions *there was no significant synergy* and the summation of pure erosion and pure corrosion weight loss rate was equal to the erosion-corrosion weight loss rate. The design of the test section and testing procedures are identical to those used by Malka and have proven to be successful in acquiring both in-situ electrochemical measurements as well as weight loss measurements<sup>6</sup>. The significant difference between Malka's results and the repeat experiments in this study suggests one of the studies has been flawed. It is believed that the flow loop used by Malka could have been tainted with a corrosion inhibiting chemical used from a previous test. Possibly the chemical was not fully removed from the system such that the testing coupons were always exposed to it during testing. The discrepancy between current results and Malka's lies in the pure corrosion results shown in Figure 1.



Figure 1: Comparison between current pure corrosion weight loss results and Malka's pure corrosion weight loss results.

The pure erosion and erosion-corrosion results are similar and on the same magnitude as Malka's. The pure corrosion results, however, are approximately five to ten times lower in Malka's results than in the current repeat results. This suggests that some type of inhibition was present in Malka's system. The current results have shown very good repeatability and are taken to be the correct analysis for erosion-corrosion under the tested conditions. Malka's results do suggest that under inhibited conditions there may be a significant synergy that develops between wear and corrosion. Erosion-corrosion under inhibited flow may be a direction that this research can take in the future.

#### 2.4 Corrosion Retardation Tests Using High Salt Concentration

Testing at the Ohio University Institute for Corrosion and Multiphase Technology has shown that concentrations of salt up to 10 wt% retards both the cathodic and anodic

corrosion reactions and lowers the corrosion rate up to five times the corrosion rate at 1 wt% salt<sup>11</sup>. This property was used in this study to lower the corrosion rate and study erosion-corrosion under slight inhibition.

Malka's study yielded corrosion rates that were approximately five times lower than the corrosion rates from current testing so using high salt concentrations reduces the corrosion rate to the same magnitude as Malka. This property was used to determine if the synergy that Malka observed is reproducible under similar conditions.

## **Chapter 3: Research Objectives and Test Matrices**

#### **3.1 Research Objectives**

The research objectives for this study are:

- 1) Determine the effect of disturbed flow on corrosion
- 2) Determine the effect of disturbed flow on erosion
- 3) Study any synergy between erosion and corrosion and quantify that effect
- 4) Test the effect of sand concentration on erosion and erosion-corrosion
- 5) Test the effect of salt concentration on erosion, corrosion, and erosion-corrosion
- 6) Determine if there is any localized corrosion due to the disturbed flow

#### **3.2 Test Matrices**

The following are the experimental test matrices used in this study.

Parameter	Conditions	
Solution	Water/Salt	
Temperature	35°C	
Flow Velocity	2 m/s in 4" (10.2 cm) pipe section	
рН	4	
CO <sub>2</sub> Partial Pressure	1 bar	
NaCl Concentration	1, 10 wt%	
Corrosion Rate Measurement	Weight loss	
	Linear Polarization Resistance	

Table 1: Pure Corrosion Test Matrix

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Parameter	Conditions
Solution	Water/Salt/Sand
Temperature	35°C
Flow Velocity	2 m/s in 4" (10.2 cm) pipe section
pH	7
N <sub>2</sub> Partial Pressure	1 bar
NaCl Concentration	1, 10 wt%
Sand Concentration	1,2 wt%
Corrosion Rate Measurement	Linear Polarization Resistance
Erosion Rate Measurement	Weight Loss

Parameter	Conditions
Solution	Water/Salt/Sand
Temperature	35°C
Flow Velocity	2 m/s in 4" (10.2 cm) pipe section
pH	4
CO <sub>2</sub> Partial Pressure	1 bar
NaCl Concentration	1, 10 wt%
Sand Concentration	1,2 wt%
Corrosion Rate Measurement	Linear Polarization Resistance
Erosion-Corrosion Rate	Weight Loss
Measurement	

Table 3: Erosion-Corrosion Test Matrix

### **Chapter 4: Experimental Procedure and Testing Equipment**

#### **4.1 Experimental Testing Equipment**

The approach used in this research is to monitor erosion and corrosion in a five foot acrylic test section with fifty-eight concentric coupons mounted in a re-circulating flow loop (Figure 2). The coupons are made from 4" (10.2 cm) and  $2\frac{1}{2}$ " (6.4 cm) diameter 1018 carbon steel pipe which has been cut into  $\frac{3}{4}$ " (1.9 cm) wide rings. Surface areas tested range from 5.98 in<sup>2</sup> (38.6 cm<sup>2</sup>) to 9.49 in<sup>2</sup> (61.2 cm<sup>2</sup>). These rings have been machined so that the outer diameter fits tightly into an acrylic tube. The test section consists of five individual acrylic tubes which are flanged so they can be bolted together once the rings are in place. The rings are isolated from each other using 1/8" (0.32 cm) Viton o-rings which also provide sealing to prevent the outer surface of the rings from contacting solution. The stress applied by bolting the acrylic flanges together creates the proper amount of compression on the o-rings. Stainless steel machine screws through helicoil inserts in the acrylic pipe wall make electrical contact with each individual ring for external measurements. The test section consists of a six inch section of 4" (10.2 cm) rings, a constriction from 4" (10.2 cm) to  $2\frac{1}{2}$ " (6.4 cm), halfway between the 25 inches of  $2\frac{1}{2}$  (6.4 cm) pipe section is a 2" (5.1 cm) protrusion, and then an expansion from 2  $\frac{1}{2}$ " to 4" with another 25 inches of 4" pipe after the expansion. Weight loss and electrochemical analysis can also be performed on the constriction because it is a flat circular plate of 1018 carbon steel which is made to fit in a recess on the 6" acrylic test section. A three-dimensional cutaway view of the test section is given as Figure 3.



Figure 2: Three-dimensional rendering of the erosion-corrosion flow loop



Figure 3: Three dimensional rendering showing the erosion-corrosion test section in a cutaway view

### **4.2 Experimental Procedure**

#### 4.2.1 Coupon Preparation

The coupon preparation procedures used in this study are derived from NACE Standard RP0775-2005<sup>8</sup>. Coupons are prepared by initially bead-blasting all of the coupons to remove any corrosion product that has formed on the surface. The outer parts of the coupons are also bead blasted to avoid rust on the outer surface. The bead-blasting process ensures a repeatable surface and makes hand polishing quicker and easier. The coupons are then polished by hand with 400 grit sandpaper to remove any dimples or irregularities caused by bead-blasting. Polishing with sandpaper provides a smoother more repeatable finish than just sandblasting. After polishing with sandpaper the coupons are rinsed with isopropyl alcohol and dried. Once the coupons are dry they are weighed to an accuracy of 0.1 mg. The coupons are then placed in the test section.

#### 4.2.2 Setting System Parameters

For every test, the flow loop is filled with approximately 180 gallons (680 liters) of water and then salt is added for the desired weight percent. For pure corrosion and erosion-corrosion tests the system is then purged with carbon dioxide overnight and for pure erosion the system is purged with nitrogen. For erosion and erosion-corrosion tests sand is added to the desired weight percent and the sand concentration is measured using the flow diversion tube (Figure 4). The concentration of oxygen is measured before the test to ensure the concentration of dissolved oxygen is less than 10 ppb. Before the test begins the pH of the system is checked and adjusted if necessary. The temperature is increased to the desired testing temperature using heating pads located on the tank. The test section is then placed in the bypass section (Figure 5) and purged with the appropriate gas to eliminate any oxygen in this area. At this point the test section has not yet contacted any liquid and the test section is only exposed to the gas used to purge the area. The bypass section is then opened to the test section and testing begins.



Figure 4: Three dimensional rendering of the flow diversion tube used to measure the insitu sand concentration



Figure 5: Three-dimensional rendering of the bypass section with the test section in place
#### 4.2.3 Monitoring During Testing

Throughout testing the pH is subject to drifting upwards due to the increase in  $Fe^{2+}$  concentration. The pH is monitored to an accuracy of 0.01 and hydrochloric acid is added if the measured pH is 0.05 pH units away from the desired pH. The system temperature is maintained using heating pads located on the tank to increase temperature or a heat exchanger using tap water to decrease the temperature. The pump adds heat to the system and therefore it is necessary to use the heat exchanger throughout the experiment to maintain the proper temperature.

During testing electrochemical measurements are made on each coupon to acquire the *in situ* corrosion rate using linear polarization resistance. The solution resistance is measured using electrochemical impedance spectroscopy.

#### 4.2.4 Shut Down

Once the experiment is finished, the pump is turned off and the bypass section is shut off to isolate the test section. The liquid is drained from the bypass and the test section is removed. The coupons are removed from the test section and rinsed with isopropyl alcohol and weighed. The coupons are then stored in an air tight container. The flow loop is drained and rinsed.

# **Chapter 5: Results and Discussion**

## 5.1 Analysis of Sand

Figure 6 shows the distribution of particle sizes for the sand used in this study. Each point shows the weight percent of sand that passed through the given sieve size. The mass average sand size is 275 microns.



Figure 6: Distribution of sand particle sizes

The effect of sand degradation with time was studied using a scanning electron microscope (SEM). It is known from previous studies that erosion rates will reduce with time in a re-circulating flow loop experiment due to sand degradation and edge rounding<sup>3</sup>. Nesic and Postlethwaite<sup>3</sup> reported that erosion coupons in a re-circulating flow loop had significantly lower erosion rates after ten hours exposure compared to two

hours. For this study it was important that sand degradation not play a factor so pure erosion and erosion-corrosion tests were held to four hours.

Figure 7 shows sand particles before pure erosion exposure at 2 wt% sand and 1 wt% NaCl. Figure 8 shows sand particles after four hours exposure at 2 wt% sand and 1 wt% NaCl. Figure 9 and Figure 10 show the before and after pictures, respectively, at a higher magnification. These images show that there is no significant rounding of sand particle edges after four hours exposure. The relative size of sand particles also appears to be the same which means there is insignificant degradation of the sand.



Figure 7: SEM image of the sand before the experiment



Figure 8: SEM image of sand after a four hour pure erosion test at 2 wt%



Figure 9: SEM image of the sand before exposure at higher magnification



Figure 10: SEM image of the sand after exposure at higher magnification

#### 5.2 Flow Analysis using Computational Fluid Dynamics

Figure 11 shows the velocity profile for the pure liquid solution of 1 wt% NaCl in water which was generated using FLUENT 6.2. This figure shows how the flow alterations change the velocity profile down the test section. This figure can be referred to in order to understand how the erosion and corrosion rates change down the test section as a function of the velocity.



Figure 11: Velocity profile down test section at 1 wt% NaCl.

Figure 12 shows the velocity profile for the pure liquid solution of 10 wt% NaCl in water which was generated using FLUENT 6.2. The solution parameters have been changed from those at 1 wt% NaCl in order to match the solution density and viscosity at 10 wt% salt. The fluid density is 1009 kg/m<sup>3</sup> at 1 wt% and 1113 kg/m<sup>3</sup> at 10 wt% and the fluid viscosity is 0.0009 kg/m-s at 1 wt% and 0.0011 kg/m-s at 10 wt%.

This velocity profile shows no significant difference from the profile obtained using the solution parameters at 1 wt% NaCl shown in Figure 11. This means that any differences in erosion or corrosion rates between 1 wt% and 10 wt% NaCl are not due to alterations in the velocity profile according to the FLUENT analysis. Using the values above the Reynolds number in a 4" (10.2 cm) pipe section at 2 m/s is 228,706 at 1 wt% NaCl and 206,400 at 10 wt% salt. The Reynolds number in a 2.5" (6.4 cm) pipe section at 4 m/s is 287,000 at 1 wt% NaCl and 259,000 at 10 wt% salt. For both the larger and smaller pipe sections there is roughly a 10% reduction in the Reynolds number when going from 1 wt% to 10 wt% salt. The significance of this change in turbulence will be discussed in greater detail in later sections. Figure 15 through Figure 18 show a more detailed analysis of the flow around the disturbances. The flow lines give more detail as to the change in turbulence around the flow due to the change in NaCl concentration according to the FLUENT analysis.



Figure 12: Velocity profile down test section at 10 wt% NaCl.



Figure 13: Flow analysis around constriction at 1wt% NaCl conditions showing the path of flow lines



Figure 14: Flow analysis around constriction at 10wt% NaCl conditions showing the path of flow lines



Figure 15: Flow analysis around protrusion at 1wt% NaCl conditions showing the path of flow lines







Figure 17: Flow analysis around expansion at 1wt% NaCl conditions showing the path of flow lines



Figure 18: Flow analysis around expansion at 10wt% NaCl conditions showing the path of flow lines

## 5.3 Results at 1 wt% NaCl

Each of the tests shown have been repeated once and the error bars given are the minimum and maximum metal loss rates for the two experiments. For the pure erosion results the average electrochemically measured corrosion rate has been subtracted from the weight loss rate in order to obtain the pure erosion rate. The magnitude of corrosion under the "pure erosion" conditions was always less than 0.2 mm/yr and this rate was consistent down the entire length of the test section.

### 5.3.1 Pure Erosion at 1 wt% NaCl

The pure erosion results at 1 wt% sand and 1 wt% NaCl show that there is very little erosion (Figure 19 and Figure 20) under these conditions. The erosion rate is less than 1 mm/yr at the most turbulent section just past the protrusion. There is an insignificant amount of erosion in the larger 4" (10.2 cm) diameter section. Due to the low erosion

rates over the length of test section it is not of any value to test at sand concentrations at or less than 1wt% sand in future tests.



Figure 19: Pure erosion rate at 1wt% sand. Under these conditions there is very little metal loss due to erosion. (2 m/s in 4" (10.2 cm) pipe section, pH 7, 1 wt% NaCl, 1 wt% sand, 1 bar  $N_2$  partial pressure)



Figure 20: Pure erosion rate at 1wt% sand on a smaller scale range

The pure erosion results at 2 wt% sand and 1 wt% NaCl show that there is significant erosion in the smaller 2.5" (6.5 cm) diameter section, but no significant erosion in the larger 4" (10.2 cm) sections (Figure 21 and Figure 22). There is also a significant increase in the erosion rate past the protrusion leading to erosion rates approximately twice those before the protrusion. The flow velocity in the 2.5" (6.4 cm) diameter section is around 3.5 m/s before the protrusion and around 5 m/s past the protrusion which can be seen from the velocity profile acquired from FLUENT (Figure 11). Under the tested conditions the minimum flow velocity and sand concentration to induce significant erosion is around 3.5 m/s and 2 wt% sand.



Figure 21: Pure erosion results at 2wt% sand. Average results from two repeat experiments. (2 m/s in 4" (10.2 cm) pipe section, pH 7, 1 wt\% NaCl, 2 wt\% sand, 1 bar N<sub>2</sub> partial pressure)



Figure 22: Pure erosion results at 2wt% sand on a smaller scale range

#### 5.3.2 Pure Corrosion Results at 1wt% NaCl

Figure 23 shows the average corrosion rate measured by weight loss. From this figure it can be seen that the corrosion rate measurement shows good repeatability. There is a slight increase in the corrosion rate past the constriction which is due to the increase in velocity and therefore an increase in the mass transfer rates of corrosive species to the metal surface. There is no increase in the corrosion rate past the protrusion which suggests the corrosion rate at this point is no longer affected by mass transfer. The corrosion rate of the constriction plate is significantly higher than the ring coupons and this can be attributed to a high corrosive wear at the edge of the plate. Figure 24 shows how the edge of the constriction plate suffered more corrosion damage than the area just 2 mm away from the edge. Figure 25 is the profile of the constriction coupon prior to experimentation which shows no edge rounding prior to corrosion exposure.

Figure 23 also shows that past the expansion the corrosion rate goes back to the same magnitude as seen in the first 6" (15.2 cm) of the test section. It can also be seen that the corrosion rate goes immediately back to the same magnitude as previously seen and remains constant.

Figure 26 shows the average weight loss corrosion rate plotted with the average corrosion rate obtained through electrochemical measurements. Both results show good agreement and therefore validate the corrosion rates obtained in these experiments.



Figure 23: Pure corrosion rate at 1wt% NaCl. These results come from the average of two repeat experiments. (2 m/s in 4" (10.2 cm) pipe section, pH 4, 1 wt% NaCl, 1 bar  $CO_2$  partial pressure)



Figure 24: Profile view attained from Infinite Focus Microscope showing the shape of the constriction edge after pure corrosion exposure for 24 hours.



Figure 25: Profile of constriction edge before corrosion exposure.



Figure 26: Average corrosion rate measured by linear polarization resistance and the average corrosion rate measured by weight loss. The electrochemical results show good agreement with the weight loss measurements.

## 5.3.3 Erosion-Corrosion Results at 1wt% NaCl

The erosion-corrosion results for 1 wt% sand and 1 wt% NaCl are shown in Figure 27. The experimental results show that the erosion-corrosion rate is not significantly different from the electrochemically measured corrosion rate except for the area of test section between the protrusion and the expansion. This is expected since the pure erosion results for 1 wt% sand and 1 wt% NaCl show that there is only significant erosion in the area past the protrusion and the other areas of the test section show no significant erosion.



Figure 27: Erosion-corrosion rate at 1 wt% sand. Under these conditions it can be seen that only past the protrusion is the erosion-corrosion rate significantly different from the electrochemically measured corrosion rate. (2 m/s in 4" pipe section, pH 4, 1 wt% NaCl, 1 wt% sand, 1 bar CO<sub>2</sub> partial pressure)

The erosion-corrosion results at 2 wt% sand and 1 wt% NaCl are shown in Figure 28. These results show that the erosion-corrosion rate is significantly different from the electrochemically measured corrosion rate only in the 2.5" (6.4 cm) area of the test section. These results are consistent with the pure erosion results obtained at 2 wt% sand and 1 wt% NaCl.



Figure 28: Erosion-corrosion rate at 2wt% sand. (2 m/s in 4" pipe section, pH 4, 1 wt% NaCl, 2 wt% sand, 1 bar CO<sub>2</sub> partial pressure)

#### 5.3.4 Combined Analysis of Results

Figure 29 shows the combined analysis of results at 1 wt% sand and 1 wt% NaCl. As discussed in previous sections under these conditions there is no significant erosion and only corrosion leads to any significant metal loss. This is obvious since the erosion-corrosion weight loss results overlay the pure corrosion results. The pure erosion results also show insignificant erosion down the entire length of the test section.



Figure 29: Combined analysis of results for 1 wt% sand and 1 wt% NaCl. At 1 wt% sand the corrosion rate is not significantly different from the erosion-corrosion rate suggesting that there is no erosion at or below this weight percent of sand. This can also be seen in the pure erosion plot where the erosion rate is less than 1 mm/yr.

Figure 30 shows the combined analysis for 2 wt% sand and 1 wt% NaCl. These results show that there is only significant erosion in the 2.5" (6.4 cm) area of the test section and the erosion rate approximately doubles in the area past the protrusion. The erosion-corrosion and pure corrosion results show no significant difference in the 4" (10.2 cm) areas of the test section which is also consistent with the pure erosion results which show no significant erosion in these areas of the test section.

Figure 31 shows the sum of pure erosion and pure corrosion plotted with the erosioncorrosion results for 2 wt% sand and 1 wt% NaCl. These plots show no significant difference meaning there is no synergistic effect under these conditions.



Figure 30: Combined analysis of results for 2 wt% sand and 1 wt% NaCl.



Figure 31: Pure erosion and pure corrosion plotted with the erosion-corrosion rate. It can be seen that under these conditions there is no synergistic effect between erosion and corrosion.

#### 5.4 Results 10 wt% NaCl

## 5.4.1 Comparison between Pure Corrosion Results at 1 wt% and 10 wt % NaCl

Figure 32 shows the comparison between pure corrosion at 1 wt% and 10 wt% NaCl. The corrosion rates decrease by one half when going from 1 wt% to 10 wt% NaCl. The reason for this comes from changes in both the chemical and mass transfer reactions as well as changes in the charge transfer reactions as postulated by Fang et al<sup>11</sup>. High concentrations of NaCl affect the chemical and charge transfer reactions by increasing the fluid density (1009 kg/m<sup>3</sup> at 1 wt% and 1113 kg/m<sup>3</sup> at 10 wt%) and the fluid viscosity (0.0009 kg/m-s at 1 wt% and 0.0011 kg/m-s at 10 wt%). This affects the mass transfer limiting current by increasing the boundary layer through which the cathodic reaction occurs. The high NaCl concentration also increases the ionic strength which reduces the amount of dissolved CO<sub>2</sub> present in the system at the same partial pressure. Fang et al.<sup>11</sup> found that these changes alone did not add up to the reduction in corrosion rate and postulated that there is also chemical adsorption of Cl<sup>-</sup> onto the metal surface which affects the charge transfer reactions.



Figure 32: Comparison between pure corrosion weight loss results at 1 wt% and 10 wt% NaCl. The corrosion rate is decreased by approximately half when going from 1 wt% to 10 wt% NaCl.

#### 5.4.2 Comparison between Pure Erosion Results at 1 wt% and 10 wt % NaCl

Figure 33 and Figure 34 show the comparison between pure erosion results at 1 wt% sand and 1 and 10 wt% NaCl. The results show that there is no change in the erosion rate profile when going from 1 to 10 wt% salt. Under these conditions there is still only a significant amount of erosion just past the protrusion leading to erosion rates around 1 mm/yr.



Figure 33: Comparison between pure erosion weight loss results at 1 and 10 wt% NaCl at 1 wt% sand. The erosion rate is unchanged when going from 1wt% to 10 wt% NaCl. Under these conditions there is still no significant erosion except just past the protrusion but still remains less than 1 mm/yr.



Figure 34: Comparison between pure erosion weight loss results at 1 and 10 wt% NaCl at 1 wt% sand on a smaller scale range.

Figure 35 and Figure 36 show the comparison between pure erosion results at 2 wt% sand and 1 and 10 wt% NaCl. The results show that the erosion rates are significantly lower past the constriction and the protrusion at 10 wt% NaCl. There is no significant difference in the erosion rates in the larger 4" (10.2 cm) sections due to the low amount of erosion loss in these areas. The reason the erosion rate is lower in the smaller 2.5" (6.4 cm) section is due to the increase in fluid density and viscosity. As discussed earlier there is a reduction in the turbulence of the flow when going from 1 wt% to 10 wt% NaCl. Another factor leading to the lower erosion rates is due to the decrease in the amount of sand that can be entrained in the flow at higher solution densities<sup>4</sup>. Higher density flows have less capacity to entrain sand as shown by the following model given by Salama<sup>4</sup>:

$$Ve = S \frac{D\sqrt{p_m}}{\sqrt{W}}$$
 Eq. 15

Where Ve is the erosional velocity limit (m/s), S is a correction factor depending on pipe geometry,  $p_m$  is the density of the fluid mixture (kg/m<sup>3</sup>), D is the pipe diameter (mm), and W is sand production (kg/day). From this equation it can be seen that if the density increases then the velocity required to induce erosion also increases. This means that at the same velocity if the density increases the amount of sand entrained is reduced. If the amount of sand entrained is reduced, then the portion which was entrained would drop out and be dragged along the bottom. This is true if the operating velocity is near the erosional velocity limit. It is known by visual observation as well as sampling technique that the tests performed in this study were near the erosional velocity limit. It could be observed through clear acrylic pipe sections located before the test section that there was sand being dragged along the bottom of the flow for both 1 and 10 wt% salt. Sampling ports were placed on the top and bottom of one of the 4" (10.2 cm) pipe sections located on the flow loop. Sampling showed that at 1 wt% salt and 2 wt % sand the top sampling port yielded a sample with a sand concentration of 1 wt% sand and the bottom sample had a sand concentration of 3 wt% which shows that there was more sand flowing along the bottom of the pipe than at the top. The average sand concentration as measured by the flow diversion tube was 2 wt% which makes sense since this was the average sand concentration of the entire flow. At 10 wt% salt and 2 wt% sand the top sample port yielded a sample with 0.75 wt% sand and the bottom sand concentration was 3.5 wt%. Once again the average sand concentration measured by the flow diversion tube was 2 wt% which is close to the average of the top and bottom sand concentration measurements. This shows that at 10 wt% NaCl there is less sand entrained since the sand concentration at the top of the pipe was lower and at the bottom of the pipe was higher.

The combination of the reduction in turbulence as well as the reduction in sand entrainment can be used to explain the difference between the erosion rates between 1 and 10 wt% NaCl at 2 wt% sand. This reduction in sand entrainment comes from the change in fluid density and viscosity which cause more drag force on the sand and less turbulence to entrain the sand.



Figure 35: Comparison between pure erosion weight loss results at 1 and 10 wt% NaCl at 2 wt% sand. The erosion rate is significantly lower at 10 wt% NaCl past the constriction and protrusion. The difference in the erosion rate is due to the changes in fluid viscosity and density.



Figure 36: Comparison between pure erosion weight loss results at 1 and 10 wt% NaCl at 2 wt% sand on a smaller scale

Figure 37 and Figure 38 shows the comparison between pure erosion results at 1 and 2 wt% sand and 10 wt% NaCl. The results show that the magnitudes of erosion rates are similar under both conditions. The sampling port at 1 wt% sand yielded a sand concentration at the top of the pipe of 0.5 wt% and at the bottom the sand concentration was 1.5 wt% which gives an average sand concentration of 1 wt% which is what was measured by the flow diversion tube. Comparing these results with the results at 2 wt% sand it can be seen that the sand concentration at the top of the pipe is the same for both 1 and 2 wt% sand and this suggests that the amount of entrained sand is also equal. This explains why these results are of similar magnitudes.



Figure 37: Comparison between pure erosion results at 1 and 2 wt% sand at 10 wt% NaCl.



Figure 38: Comparison between pure erosion results at 1 and 2 wt% sand at 10 wt% NaCl.

## 5.4.3 Erosion-Corrosion Results at 1 and 2 wt% Sand and 10 wt% NaCl

Figure 39 shows the comparison between erosion-corrosion results at 1 and 2 wt% sand. As expected from the previous pure erosion results these two trends are not significantly different since the magnitude of erosion is similar for both 1 and 2 wt% sand at 10 wt% NaCl.



Figure 39: Erosion-corrosion results comparison at 1 and 2 wt% sand at 10 wt% NaCl.

#### 5.4.4 Results Analysis at 10wt% NaCl

Increasing the concentration of NaCl from 1 wt% to 10 wt% salt not only affected the pure corrosion rate, but also caused the pure erosion rate to decrease. At 10 wt% salt the pure erosion rates are similar for both 1 wt% and 2 wt% sand. This is due to the increase in density and viscosity which causes the solution to retain the same erosivity despite the increase in the sand concentration. Figure 40 and Figure 41 show the combined analysis of results for 1 wt% and 2 wt% sand at 10 wt% NaCl. It can be seen from these graphs that the erosion-corrosion rates are significantly lower or equal to the pure corrosion rates. Under these conditions there is essentially a purely corrosive environment but there does seem to be some negative synergistic effect when sand is added which is discussed later in this section.



Figure 40: Combined analysis for 1 wt% sand and 10 wt% NaCl.



Figure 41: Combined analysis for 2 wt% sand and 10 wt% NaCl.

Figure 42 and Figure 43 show that the sum of pure erosion and pure corrosion are significantly higher than the erosion-corrosion results in some areas of the test section for both 1 wt% and 2 wt% sand. It is important to remember that under these conditions for both 1 and 2 wt% sand, erosion does not lead to significant metal loss. There may be some affect of the addition of sand on the pure corrosion rates since it has been shown that additional sand above 1 wt% simply increases the amount of sand flowing along the bottom of the pipe and does not increase the amount of entrained sand. The sand flowing along the bottom could be interfering with the pure corrosion by acting as another barrier for the corrosive species to travel through. Any sand barrier along the bottom of the pipe would also interfere with the erosion rates since sand particles cannot impact the surface where un-entrained sand is flowing.



Figure 42: Sum of erosion and corrosion rates plotted with the erosion-corrosion rate for 1 wt% sand and 10 wt% NaCl.



Figure 43: Sum of erosion and corrosion rates plotted with the erosion-corrosion rate for 2 wt% sand and 10 wt% NaCl.

#### 5.5 Microscopy Analysis of Coupons

Select coupons from experiments at 10 wt% salt were analyzed using an Infinite Focus Microscope (IFM). Erosion, corrosion, and erosion-corrosion coupons from tests at 10 wt% salt were analyzed in order to determine if there is localized corrosion under these conditions. Although the testing periods are relatively short (< 24 hours) there may be some visible pits or pit formation on the metal surface. These experiments and others<sup>11</sup> have shown that salt will reduce the corrosion rate at high concentrations. Fang<sup>11</sup> concluded that there must be some chloride adsorption onto the metal surface to fully explain the magnitude of reduction. If chloride does adsorb onto the surface this could possibly lead to localized corrosion attack.

#### 5.5.1 Pure Corrosion 10 wt% NaCl

Figure 44 shows a three dimensional IFM image of the bottom of the 2.5" (6.4 cm) corrosion coupon located three inches past the protrusion. The coupon was chosen because it was in the highest velocity range according to the FLUENT analysis. Figure 45 shows the same image but in an uncolored view which shows only the surface texture. From these two pictures no discernable pits or possible pit initiation can be seen. Only general corrosion is observed on this area of the coupon. Figure 46 shows the texture view of the top of the same corrosion coupon also showing no indication of pitting.

Figure 47 shows the textured view of constriction plate from the same experiment. There is no visible pit formation seen in this image and the striations that are seen are polishing marks. Figure 48 shows the profile view of the constriction plate. From this image it can be seen that the edge has corroded more than the rest of the plate. The edge wear at 10 wt% NaCl appears to be less than that shown in Figure 24 at 1 wt% NaCl.



Figure 44: IFM image of the bottom of the pure corrosion coupon three inches past the protrusion. (2 m/s in 4" (10.2 cm) pipe section, pH 4, 10 wt% NaCl, 1 bar  $CO_2$  partial pressure)



Figure 45: IFM image of the bottom of the pure corrosion coupon three inches past the protrusion showing the texture of the surface.  $(2 \text{ m/s in 4"}(10.2 \text{ cm}) \text{ pipe section, pH 4}, 10 \text{ wt\% NaCl}, 1 \text{ bar CO}_2 \text{ partial pressure})$ 



Figure 46: IFM image of the top of the pure corrosion coupon three inches past the protrusion showing the texture of the surface.  $(2 \text{ m/s in 4"} (10.2 \text{ cm}) \text{ pipe section, pH 4}, 10 \text{ wt\% NaCl, 1 bar CO}_2 \text{ partial pressure})$ 



Figure 47: Edge of the constriction plate from pure corrosion experiment. (2 m/s in 4" (10.2 cm) pipe section, pH 4, 10 wt% NaCl, 1 bar CO<sub>2</sub> partial pressure)



Figure 48: Edge profile of constriction plate from pure corrosion experiment. (2 m/s in 4" (10.2 cm) pipe section, pH 4, 10 wt% NaCl, 1 bar CO<sub>2</sub> partial pressure)

#### 5.5.2 Pure Erosion 10 wt% NaCl

The following images are from an erosion coupon exposed to 2 wt% sand and 10 wt% NaCl for four hours. Since there was no significant difference between 2 wt% and 1 wt% sand at 10 wt% NaCl only the pure erosion at 2 wt% sand has been shown. Figure 49 shows the bottom of the 2 ½" (6.4 cm) pure erosion coupon that was located three inches past the protrusion. Figure 50 shows the top of the same coupon. Neither of these images show any visible signs of localized attack.

Figure 51 shows an image of the constriction plate taken from the pure erosion experiment. There is no visible localized attack on the constriction plate. The striations are from polishing and show that the amount of erosion attack has not been sufficient to remove these striations which are not as visible in the pure corrosion image (Figure 47).
Figure 52 shows the profile of the constriction edge. There is significant rounding of the coupon edge but the magnitude is less than that seen under pure corrosion conditions (Figure 48).



Figure 49: Textured view of bottom of small pure erosion coupon three inches past the protrusion. (2 m/s in 4" (10.2 cm) pipe section, pH 7, 10 wt% NaCl, 1 bar  $N_2$  partial pressure, 2wt% sand)



Figure 50: Textured view of top of small pure erosion coupon three inches past the protrusion. (2 m/s in 4" (10.2 cm) pipe section, pH 7, 10 wt% NaCl, 1 bar  $N_2$  partial pressure, 2wt% sand)



Figure 51: Textured view of constriction plate from pure erosion experiment. (2 m/s in 4" (10.2 cm) pipe section, pH 7, 10 wt% NaCl, 1 bar  $N_2$  partial pressure, 2wt% sand)



Figure 52: Edge profile of constriction plate from pure erosion experiment. (2 m/s in 4" (10.2 cm) pipe section, pH 7, 10 wt% NaCl, 1 bar N<sub>2</sub> partial pressure, 2wt% sand)

### 5.5.3 Erosion-Corrosion 10 wt% NaCl

The following images are from an erosion-corrosion coupon exposed to 2 wt% sand and 10 wt% NaCl for four hours. Figure 53 shows the bottom of the 2 ½" (6.4 cm) erosion-corrosion coupon that was located three inches past the protrusion. Figure 54 shows the top of the same coupon. Neither of these images show any visible signs of localized attack.

Figure 55 shows an image of the constriction plate from the erosion-corrosion experiment. Once again no visible localized attack can be seen. Figure 56 shows the edge profile for the constriction plate. The edge has been significantly worn as seen with the other exposed coupons.



Figure 53: Textured view of bottom of small erosion-corrosion coupon three inches past the protrusion. (2 m/s in 4" (10.2 cm) pipe section, pH 4, 10 wt% NaCl, 1 bar CO<sub>2</sub> partial pressure, 2wt% sand)



Figure 54: Textured view of top of small erosion-corrosion coupon three inches past the protrusion. (2 m/s in 4" (10.2 cm) pipe section, pH 4, 10 wt% NaCl, 1 bar  $CO_2$  partial pressure, 2wt% sand)



Figure 55: Textured view of constriction plate from erosion-corrosion experiment. (2 m/s in 4" (10.2 cm) pipe section, pH 4, 10 wt% NaCl, 1 bar  $CO_2$  partial pressure, 2wt% sand)



Figure 56: Edge profile of constriction plate from erosion-corrosion experiment. (2 m/s in 4" (10.2 cm) pipe section, pH 4, 10 wt% NaCl, 1 bar CO<sub>2</sub> partial pressure, 2wt% sand)

# **Chapter 6: Conclusions and Future Work**

#### **6.1 Conclusions**

- No synergistic effect between erosion and corrosion under tested conditions
- No significant erosion at 1 wt% NaCl and 1 wt% sand
- Significant erosion was seen at 2 wt% sand and 1 wt% NaCl in the constriction and protrusion sections but not in the 4" diameter sections
- No significant erosion at 10 wt% NaCl for either 1 or 2 wt% sand
- Erosion rates decrease from 1 wt% to 10 wt% NaCl which was attributed to an increase in fluid density and viscosity which reduced the amount of entrained sand
- Higher density and viscosity make the sand entrained move more in line with the fluid and therefore does not let it deviate and strike the surface of the metal
- The amount of sand entrained at 10 wt% NaCl and 2 wt% sand was significantly less than that entrained at 1 wt% NaCl and 2 wt% sand
- Corrosion rates reduced by approximately one half when the concentration of NaCl was increased from 1 wt% to 10 wt% NaCl
- No formation of localized attack was observed at 10 wt% NaCl

#### **6.2 Summary of Conclusions**

This study has shown a significant difference in results compared to the previous study by Malka<sup>6</sup>. The pure corrosion results obtained in Malka's study were approximately ten times lower than the pure corrosion results obtained in this study.

This suggests that there was some type of inhibitor present in Malka's system which lead to the synergistic effect he found.

The results from the current study has shown salt reduces the magnitude of corrosion when going from 1 wt% to 10 wt% NaCl. This effect has been found in other studies<sup>11</sup> and it has been concluded the reduction in the corrosion rates at higher concentrations of NaCl comes from the changes in density and viscosity which affect the mass transfer reactions, from an increase in ionic strength which reduces the amount of dissolved CO<sub>2</sub>, and from chloride ion adsorption onto the metal surface.

Furthermore this study has found that increasing the concentration of NaCl also affects the erosion rates. The erosion rates at 1 and 2 wt% sand and 10 wt% NaCl are not significantly different. At 1 wt% NaCl, however, erosion rates at 2 wt% sand are significantly higher than at 1 wt% sand. This reduction in the erosion rate comes from the increase in fluid density and viscosity. Increasing the density and viscosity causes more drag force on the particle and also reduces the turbulence of the flow. These changes decrease the amount of sand entrained. Only 1 wt% sand can be entrained at 10 wt% NaCl and any additional sand added rolls along the bottom of the pipe and does not lead to erosion. Therefore the erosion rates at 1 wt% sand are the maximum erosion rates that can be obtained under the tested conditions at 10 wt% NaCl.

#### **6.3 Suggestions for Future Work**

In order to determine if localized corrosion occurs at high concentrations of salt longer testing times need to be used. Extending pure corrosion tests to one week would determine if localized attack will occur due to the high salt concentration. Longer test times would also determine if the reduction in corrosion rate with an increase in salt concentration occurs over longer periods of time or if it only prolongs the transient corrosion response.

It was assumed that the reduction in the corrosion rate at higher salt concentrations was due to changes in ionic strength, changes in mass transfer effects due to increasing viscosity and density, as well as adsorption of the chloride ion onto the metal surface. The ionic strength change could be tested by determining the dissolved  $CO_2$  at 1 wt% and 10 wt% NaCl. This will determine the actual reduction in dissolved  $CO_2$  experimentally.

The assumption that chloride ions adsorb onto the metal surface could be tested by using a higher concentration of sand which will ensure there is significant erosion at 10 wt% NaCl. If a synergistic effect is seen under more erosive conditions then the chloride adsorption assumption would be supported.

In order to test for localized corrosion under erosion-corrosion conditions some form of sand separation will need to be used in order to increase the test time. Currently the sand is re-circulated and increasing test times would cause a reduction in the erosion rates due to sand degradation. A new method would need to be devised in order to constantly or periodically remove old sand while replenishing the loop with fresh sand. This would be easier if a small scale flow loop was used in order to reduce the amount of sand required for a test.

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