EROSION-CORROSION STUDY OF CARBON STEEL AND DUPLEX STAINLESS STEEL ELBOWS IN POTASH BRINE-SAND SLURRY

A Thesis Submitted to the College of Graduate and Postdoctoral Studies In Partial Fulfillment of the Requirements For the Degree of Doctor of Philosophy In the Department of Mechanical Engineering University of Saskatchewan Saskatoon

> By Raheem Elemuren

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Abstract

Carbon and low alloy steel pipes are widely used to transport slurries in the mining and mineral processing industry because of their good mechanical properties and relative affordability. However, the exposed internal walls of these pipes are prone to erosion-corrosion damage, which can lead to product leakage and eventual failure of the piping system. The purpose of this research is to evaluate the performance of AISI 1018 carbon steel and AISI 2205 duplex stainless steel elbows to erosion-corrosion damage in a saturated potash brine-sand slurry.

Erosion-corrosion tests were conducted in a flow loop using a slurry consisting of saturated potash brine-sand slurry. Flow velocity and particle concentration were varied, but the slurry temperature was kept constant at 30 ± 1 °C. The synergistic effect of corrosion and erosion during erosion-corrosion was evaluated by conducting corrosion and erosion tests using particle-free saturated potash brine and deaerated slurry, respectively. The elbow surfaces before and after exposure to different test conditions were characterized using a scanning electron microscope, optical profilometer, Vickers micro-hardness tester, X-ray photoelectron spectroscopy, Raman spectroscopy, X-ray diffraction, and electron-backscattered diffraction.

The results of these study revealed that the synergy between corrosion and erosion accelerated erosion-corrosion rates for the exposed carbon steel elbows. Microstructural examination showed that mechanical wear, in conjunction with pitting corrosion, played a substantial role in removing materials from the exposed surfaces of carbon steel elbows, especially at low slurry flow velocity. Raman and X-ray spectroscopic studies indicated that the corrosion products found inside the pits of the exposed carbon steel elbow are FeOOH, Fe₂O₃ and Fe₃O₄. This confirms the oxidation of iron during erosion-corrosion in saturated potash brine slurry.

Furthermore, mechanical wear was the sole material removal mechanism for the duplex stainless steel elbows, which indicate that the combined effects of corrosion and erosion did not influence erosion-corrosion rates. X-ray and electron-backscattered diffraction techniques revealed that some austenite phase of the duplex stainless steel transformed to martensite after particle impacts at high velocity. The exceptionally high hardness and brittleness of martensite accelerated the removal of materials from the duplex stainless steel surface by brittle chipping. Plastic deformation due to particle impingement occurred up to about 4 μ m below the exposed elbow surface.

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Dedication

To God,

"The seven heavens, the earth and everything in them glorify Him. There is not a thing that does not praise Him, but we do not understand their ways of praising Him. Indeed, He is ever forbearing and forgiving."

To my dad,

who passed away exactly four months into my Ph.D. program (January 1, 2017), may God in his infinite mercy forgive all your shortcomings.

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List of abbreviations and symbols

Abbreviations

AISI	American iron and steel institute
ASTM	American society for testing and materials
DSS	Duplex stainless steel
EBSD	Electron backscattered diffraction
EDS	Energy-dispersive X-ray spectroscopy
EPDM	Ethylene propylene diene terpolymer
GFRP	Glass fiber reinforced polymer composites
JIT	Jet impingement testing machine
MLRA	Multiple linear regression analysis
PVC	Polyvinyl chloride
SEM	Scanning electron microscopy
XRD	X-ray diffraction
XPS	X-ray photoelectron spectroscopy
A_{ws}	Area of the material
CR	Corrosion rate
$C_{b_{O_2}}$	Bulk concentration of oxygen
Co	Material loss due to pure corrosion
C _w	Contribution of corrosion to the total material loss
D	Diffusivity of oxygen
Ε	Erosion rate
E-C	Erosion-corrosion
F	Faraday constant (96,496 Coulombs/mole)
F_{ε}	Deformation wear factor
Fρ	Cutting wear factor
Κ	Maximum velocity of the impacting particles
KE	Kinetic energy

L_P	Length of the particle
M_{Fe}	Molar mass of iron
N _{SP}	Number of particles impacting on a surface per hour
Р	Perimeter of the particle
P _c	Particle concentration
PS	Percentage contribution of the synergistic component
Q	Volume of material removed
R _e	Reynolds number
S	Synergistic component
Sa	Arithmetic mean height
S _c	Schmidt number
S_{ku}	Kurtosis
S _p	Maximum peak height
$\mathbf{S}_{\mathbf{q}}$	Root-mean-square height
\mathbf{S}_{sk}	Skewness
$\mathbf{S}_{\mathbf{v}}$	Maximum pit depth
Sz	Maximum height
Т	Total material loss due to erosion-corrosion
V	Velocity
V _m	Material volume
V _{mc}	Core material volume
\mathbf{V}_{v}	Void volume
V _{vc}	Core void volume
\mathbf{V}_{vv}	Pit void volume
W _c	Contribution of erosion to the overall material loss
W_o	Material loss due to pure erosion
W_P	Particle width
Y	Response variable
a_o	Estimated constant

a_1 , a_2 and a_n	Regression coefficients	
d_p	Particle diameter	
е	Residual error	
i _{lim}	Limiting current density	
k	Constant	
k _d	Mass transfer coefficient for a fully developed flow	
k_m	Mass transfer coefficient for a turbulent flow	
m	Mass	
m_l	Mass of potash brine	
m_s	Mass of dry silica sand particles	
n	Velocity exponent	
p	Constant plastic flow stress	
W _C	Cutting wear parameter	
W _D	Deformation wear parameter	
x_1 , x_2 and x_n	Independent variables	

Symbols

θ	Impingement angle
γ	Austenite
α	Ferrite
σ	Standard deviation
μ	Mean
$ ho_w$	Density of water
$ ho_p$	Particle density
$ ho_{Fe}$	Density of iron
ΔW_c	Change in erosion rate as a result of corrosion
ΔC_w	Change in corrosion rate as a result of erosive wear

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Chapter One Introduction

1.1 Overview

Potash is an essential mineral to Saskatchewan's economy, with the annual sales of potash in 2018 increasing to \$5.7 billion, following a \$4.8 billion in sales in 2017 [1]. The potash sales in 2018 contributed a gross domestic product (GDP) of 6.6% to the economy of Saskatchewan due to the increase in production. Potash is widely used in combination with phosphorus and nitrogen as fertilizer; it serves as a regulator to enhance the circulation of fluid in plants, protect against stress, photosynthesis and protein activation. Production of potash in Saskatchewan accounts for about one-third of the world potash production, and it is mined either by conventional or solution mining methods. Unlike solution mining, which is used in regions where the potash reserves are more than 1500 m deep, the conventional mining method is utilized in areas where the potash reserves are about 900 to 1100 m beneath the earth's surface. For efficient transfer during processing in a conventional mine, potash is commonly transported in carbon steel pipes as a slurry at a high flow rate to avoid caking. However, these steel pipes are highly prone to slurry erosion-corrosion damage. Damage to materials as a result of erosion-corrosion often leads to substantial economic loss emanating from increased maintenance costs, downtime and premature failure of parts [2]. The overall annual corrosion cost (direct and indirect) in Canada is estimated to be approximately \$46.4 billion in 2003 [3], which accounts for about 2.5% of the GDP. Furthermore, the global cost of corrosion is reported to be \$2.5 trillion in 2013 [4], representing a global GDP of 3.4%.

Erosion corrosion is one of the major causes of material damage in the mineral processing industries. For example, steel pipes conveying oil sands slurry, which contains NaCl, NaHCO₃, bitumen, oxygen and sand, are prone to erosion-corrosion attacks, leading to high maintenance costs. In potash processing plants, the presence of oxygen, saturated potash brine and solid potash ore impinging on pipe walls lead to damage and leakage of potash brine. The associated maintenance cost in potash processing plants is also very high. Erosion-corrosion is the rapid removal of material from a metallic surface by the combined actions of mechanical erosion and electrochemical corrosion processes. The synergistic effect between corrosion and erosion that occurs during erosion-corrosion could accelerate the total material removal rate through the enhancement of corrosion by erosion or the enhancement of erosion by corrosion [2,5,6]. Erosion-

corrosion is prominent in fluid flow systems, especially at elbows, in valves and pumps, where turbulent flow or a sudden change in fluid flow direction causes severe material loss [7]. It is influenced by factors such as flow conditions, properties of the exposed material and those of the particles. Thus, different research studies have been carried out to understand the effects of flow velocity, impact angle, particle concentration, temperature, material hardness, particle size and shape on the erosion-corrosion behaviour of metallic materials [7–26].

Although several investigations have been conducted to study the erosion-corrosion behaviour of materials in different environments; however, little is known about their behaviour in potash slurry. In 1974, Postlethwaite *et al.* [6] reported that there was no substantial effect of particle concentration and flow rate on the corrosion process that occurs during the erosion-corrosion attack of a carbon steel straight pipe carrying potash slurry. They suggest that the observed low corrosion rate was a result of scaling because most of their experimental studies were conducted at flow rates below 1.7 m/s, which is not representative of the flow rate commonly used in the potash processing industry. Huang [27] studied the erosion-corrosion behaviour of different materials in potash brine slurry at a flow rate of 1.47 m/s and an impingement angle of 33°; he reported that glass fiber reinforced polymer composites (GFRP) exhibited excellent resistance to erosion-corrosion damage, but the propensity of GFRP to absorb potash brine could limit its application in potash processing plants.

1.2 Motivation

In spite of the advances that have been made to understand this problem, erosion-corrosion attack continues to be a major challenge in the Saskatchewan's potash processing facilities. Therefore, it is necessary to mitigate erosion-corrosion damage to carbon steel pipelines to reduce plant maintenance costs and workplace hazards posed by damaged or leaking pipes. Erosion-corrosion damage in pipelines can be mitigated by controlling the slurry flow rate, using chemical inhibitors, reducing solid particle content or changing pipe shape and geometry. However, reducing the slurry flow rate during operation can cause caking/scaling, chemical inhibitors can alter the composition of the final product and changing the shape and geometry of pipes can be expensive, since it involves stopping production. Thus, this thesis report aims to use materials selection method to alleviate the problem of erosion-corrosion damage in potash processing facilities. In this study, the erosion-corrosion behaviour of carbon steel and a duplex stainless steel elbow in a saturated potash

brine-sand slurry was investigated using a laboratory flow loop apparatus. The effects of concentrations of solid particles and flow velocities on erosion-corrosion of the materials were determined.

1.3 Research objectives

The overall goal of this research investigation is to reduce the erosion-corrosion damage to potash slurry pipelines and the associated maintenance cost in Saskatchewan potash processing facilities. This goal will be achieved through the following specific objectives:

- 1) Determine the effects of velocity, particle concentration and the synergistic erosioncorrosion behaviour of AISI 1018 carbon steel elbows in saturated potash brine-sand slurry.
- Determine the effects of velocity, particle concentration and the synergistic erosioncorrosion behaviour of AISI 2205 duplex stainless steel elbows in saturated potash brinesand slurry.
- Develop an empirical statistical model using full factorial analysis to determine the contributions of velocity, particle concentration and their interaction to the total erosioncorrosion rate of both alloys.

1.4 Research contribution

The outcome of this research will add to the currently limited information on the performance of the selected materials when exposed to erosion-corrosion attack in saturated potash brine-sand slurry. The results of this investigation would be beneficial in understanding the synergistic effect of erosion and corrosion that occur during erosion-corrosion and the associated damage mechanisms. Furthermore, the results obtained from this research and subsequent ones by our research group on other alloys will provide a guide for selecting slurry pipeline materials in the minerals processing industry. This will help reduce premature pipe leakage, maintenance costs and shutdowns. Finally, since several operating conditions can influence the extent of damage to metallic materials during an erosion-corrosion attack, the generated data can be used to bridge the knowledge gap in research areas related to erosion-corrosion and to predict erosion-corrosion rate using the identified flow parameters.

1.5 Thesis arrangement

This thesis report consists of eight chapters. The research overview, motivation, objectives, and expected contributions are presented in chapter one. Chapter two contains the review of literature in subject areas about erosion-corrosion, while the detailed information about the materials and methodology used in this study are presented in chapter three. The results of the experimental studies to realize the first objective of this research are discussed in chapters four and five. Chapters six and seven contains the experimental results that address the second and third objectives.

In chapters four and five, the experimental results and discussion for the influence of particle concentration, slurry flow velocity, and the synergistic effect of corrosion and erosion on the erosion-corrosion behaviour of AISI 1018 carbon steel elbows are presented. These chapters aim to determine the effects of different test conditions on the materials removal rates for the carbon steel elbows exposed to erosion-corrosion in saturated potash brine-sand slurry. Both chapters are published in *Wear*. In Chapter Six, the development of an empirical model using full factorial statistical analysis is presented and discussed. The purpose of this empirical model is to predict the erosion-corrosion rates of the exposed AISI 1018 carbon steel elbows and to determine the test parameter with the most significant influence on erosion-corrosion rate. The results of the statistical, microscopic and spectroscopic investigations have been published in *Tribology International*.

In Chapter Seven, the results of the synergistic effects of erosion-corrosion, the influence of the operating parameters and statistical analysis for the AISI 2205 duplex stainless steel elbows are presented and discussed. This chapter investigated the influence of the different test parameters on the material removal rates for the duplex stainless elbows exposed to erosion-corrosion in saturated potash brine-sand slurry. The results presented in this chapter are discussed in a paper currently under consideration for publication by the *Journal of Materials Engineering and Performance*. Finally, the summary and conclusions drawn from this study, along with some recommendations for future work, are provided in Chapter 8.

Chapter Two Literature review

2.1 Forms of corrosion

Corrosion in the mining and mineral processing industry commonly occurs as a result of the electrochemical reaction of metallic components with their environments. Corrosion modes can be classified based on their appearance. The major forms of corrosion that commonly occur in metallic alloys are presented in Fig. 2.1. Erosion-corrosion is one of the most common corrosion problems encountered in mining and mineral processing facilities. Erosion-corrosion damage and its associated mechanisms in metallic alloys are reviewed in this chapter. The different types of erosion-corrosion test devices, the factors influencing erosion-corrosion, models for predicting erosion-corrosion, and erosion-corrosion mitigation processes are also reviewed.



Figure 2.1. Schematic diagrams of the different forms of corrosion failure. (Reproduced from Handbook of Corrosion Engineering [28]).

2.1.1 Erosion-corrosion

Erosion-corrosion is a complex phenomenon because it involves the conjoint actions of corrosion and erosion processes. It is the principal cause of leakage in pipes, especially when the liquid flowing through the pipe contains entrained solid particles or gas bubbles. Erosion-corrosion damage is usually aggravated by the impingement of particles (or bubbles) on the inner wall of the pipe. It occurs in regions where the fluid flow is turbulent as in heat exchanger tubes and internal combustion engines [29]. It also occurs in elbows/bends where there is an abrupt change in the direction of fluid flow. Figure 2.2 shows some of the modes of material removal from an exposed surface during an erosion-corrosion attack. The continuous impingement of solid particles, gas bubbles or liquid in a multiphase flow enhances material removal from the exposed surface. Progressive thinning of the pipe wall occurs during erosion-corrosion by the development of craters and metal cutting in the direction of flow (Figs. 2.2a and 2.2b).



Figure 2.2. Modes of material removal from an exposed surface due to erosion-corrosion damage [7].

2.2 Test apparatus used for evaluating erosion-corrosion

It is important to conduct either a pilot-scale or laboratory-scale testing of engineering materials for erosion-corrosion resistance before using them in applications where they will be exposed to moving fluid containing solid particles or gas bubbles. Most laboratory-scale erosion-corrosion studies are usually accelerated and scaled-down to determine the erosion-corrosion resistance of structural materials. However, the modes of erosion-corrosion damage in pipes based on flowconditions that simulate industrial practice are not well understood due to the complexity in the hydrodynamics of slurry flow in pipes. The complexities associated with this form of corrosion has led to the development of several laboratory equipment used to estimate erosion-corrosion rate. The jet impingement and slurry pot erosion apparatus are common due to their relatively low maintenance costs and ease of usage.

2.2.1 Slurry pot

Tsai *et al.* [30] designed and constructed a slurry pot apparatus for erosion test in 1981. The sketch of a typical slurry pot is provided in Fig. 2.3. The slurry pot consists of a cylindrical tank, a regulator for controlling the velocity and a central shaft, which is spun by an electric motor. The shaft supports two or more vertically positioned test materials (Fig. 2.3a). Baffles are fixed to the internal walls of the tank to prevent particles from settling at the bottom of the tank. A heat exchanger coils placed within the cylindrical container is used to maintain a steady slurry temperature. Over the years, several modifications to the slurry pot have been done [31–35]. Rajahram *et al.* [32] improved the slurry pot tester to simultaneously conduct erosion and in-situ electrochemical corrosion measurements of materials using a three-cell electrode, as shown in Fig. 2.3b.

The main problem associated with the slurry pot is the wide variation in particle concentration at different regions within the apparatus. Thus, leading to slurry inhomogeneity and non-uniform flow pattern [36]. Since the flow field in this apparatus is a function of the particle size [37,38], changes to the particles must be known to fully understand the effect of particle size on the erosion or erosion-corrosion behaviour of materials. Researchers should take adequate precautions when using data generated from the slurry pot to predict material removal rate due to a substantial difference in the hydrodynamic intensities between slurry pot apparatus and that of an actual pipe flow.



Figure 2.3. (a) Schematic diagram of a slurry pot [39], (b) modified slurry pot used to measure insitu corrosion [32].

2.2.2 Jet impingement

The jet impingement apparatus is used to study the erosion or erosion-corrosion behaviour of flat materials at high flow rates. Jet impingement can be categorized as either dry or wet. The dry jet impingement apparatus was developed to evaluate the behaviour of materials exposed to solid-gaseous mixture [40–43]. The main components of the earliest known dry jet impingement testing machine (JIT) consisted of a reservoir, a compressor, a sample holder and a nozzle. High air pressure from the compressor is used to circulate solid particles which are fed from the tank at a constant speed. The particles are accelerated through the nozzle by the flow of inert gas and made to impinge on the target surface at a specified angle. Modification of the dry JIT was done so that the investigation of materials' performance in a slurry containing suspended particles could be done. The mode of operation remains the same, while the only modification to the first developed JIT machine is the swapping of the compressor with a pump. Figure 2.4 shows the schematic diagram of a typical wet jet impingement testing machine. The main advantage of the dry JIT over the wet JIT is the avoidance of wear damage to the pump. Jet impingement testing does not simulate the typical slurry flow field in pipes. Thus, it is best suited for materials ranking and selection.



Figure 2.4. A 2D schematic diagram of a wet jet impingement apparatus [44].

2.2.3 Pipe flow loop

The pipe flow loop offers a test condition that is closest to the flow condition in typical industrial applications and has the advantage of providing the best estimate for the erosion or erosion-corrosion rates of pipeline materials that are often used. The flow loop is not widely used as a result of its high construction and maintenance costs. Based on the Scopus citation database, one of the earliest use of a flow loop to investigate the slurry erosion behaviour of a pipe was by Karabelas [45] in 1978. His flow loop consisted of a rotary pump, a reservoir, an impeller, a heat exchanger coil, and a 16-meter long schedule 40S 304 stainless steel pipe. The particles in the slurry are kept suspended within the reservoir by an impeller, and the rotary pump is used to move the homogenous slurry through the stainless steel pipe. The temperature and flow velocity of the slurry in the system is controlled by a heat exchanger coil and a Fisher-Porter magnetic flow meter, respectively. The schematic diagram of a typical flow loop apparatus is presented in Fig. 2.5.



Figure 2.5. A schematic diagram of a flow loop used for investigating flow-assisted corrosion, slurry erosion, or erosion-corrosion [46].

Over the years, several flow loop modifications and experimental investigations have been conducted on pipeline materials [47–50]. Malka *et al.* [51] designed and developed a pipe flow loop that allows for the simultaneous study of ring-like steel in disturbed flow geometries that simulates pipe protrusion, expansion and contraction. In their study, a positive displacement pump circulated the fluids through a 30 ft long, 4-in internal diameter polyvinyl chloride (PVC). The test materials for the contraction and expansion studies are installed between 4-in and a 2.47-in internal diameter pipes, as depicted in Fig. 2.6, while that of the protrusion was fitted in the 2.47-in internal diameter straight pipe.



Figure 2.6. Schematic diagrams showing the cross-sectional view of the pipe contraction, protrusion and expansion in a flow loop [51].

No.	Apparatus	Advantages	Disadvantages
1.	Flow loop	 It simulates the actual flow conditions in pipeline steel used in industrial applications [52]. Simultaneous testing of several materials at different locations. It allows for the homogeneous mixing of slurry. 	 Limitation of laboratory space [53]. High construction cost [54]. Long test duration is required to acquire detectable wear data. Repeated impingement causes fragmentation and dulling of particles [37]. The impingement angle of particles is difficult to control.
2.	Jet impingement	 Impingement angle and flow velocity can easily be varied. Easy removal of test material. Best used for materials ranking. Economical, easy to construct and operate [55]. 	 Operating velocities are usually higher than those found in industrial conditions. Particle concentration is difficult to control. Rapid damage of the nozzle leads to the constant calibration of flow velocity [56].
3.	Slurry pot	 Easy to manufacture. Fast and suited for materials ranking. Simultaneous testing of multiple materials 	 Rapid fragmentation of solid particles. Impact velocity and angle are difficult to measure and control. The concentration of particles varies in different regions within the apparatus.

Table 2.1. Summary of some of the different apparatus used to investigate slurry erosion-corrosion.

2.3 Factors affecting slurry erosion-corrosion

Slurry erosion-corrosion is a complex phenomenon due to the wide range of operating parameters that can influence the erosion-corrosion behaviour of metallic materials. Such parameters include flow condition, test apparatus, slurry properties, target material and particle properties. Interactions between these parameters make the estimation of erosion-corrosion rates difficult. Some of the factors affecting the erosion-corrosion behaviour of metals are discussed below.

2.3.1 Flow velocity

Velocity is an essential operating parameter that influences erosion-corrosion because the rate of material removal from a surface is dependent on the speed of the impacting particles. Several researchers have reported that material loss increases with increasing velocity [57–60], which is due to the increased kinetic energy of the impacting particles. The permanent damage to the surface of materials only occurs when the yield stress is exceeded [61]. The particle kinetic energy (*KE*) is given by Eqn. 2.1. Erosion rate (*E*) is proportional to flow velocity based on the power law equation, as provided in Eqn. 2.2 [62,63]. Lopez *et al.* [64] investigated the effect of particle velocity on the erosion-corrosion of AISI 304 and 420 stainless steels. They reported that the rate of material loss increased with increasing flow velocity. The dependence of material loss on velocity for AISI 304 stainless steel and a Fe-based amorphous metallic coating is presented in Fig. 2.7.

$$KE = \frac{1}{2}mV^2 \tag{2.1}$$

$$E = kV^n 2.2$$

where k is a constant, m is the mass of the particles moving at a velocity, V and n is the velocity exponent. The velocity exponent for metals has been reported to be within the range of 1 and 3 for erosion studies [65–69] and from 1 to 5 for erosion-corrosion studies [29,70,71]. Zhou and Bahadur [72] reported a velocity exponent value of 1.89 after a dry erosion study of Ti-6Al-4V alloy at 500 °C, with silicon carbide particles impacting the alloy at an angle of 30°. However, Yerramareddy and Bahadur [73] reported an exponential value of 2.35 when they investigated the dry erosion behaviour of Ti-6Al-4V alloy at room temperature using silicon carbide particles that impinge on the surface at an of 30°. The substantial variation in the velocity exponents obtained
by different researchers is attributed to factors such as slurry conditions and properties of the target material.



Figure 2.7. Material removal rate versus flow velocity obtained for AISI 304 stainless steel and a Fe-based amorphous metallic coating after erosion-corrosion in 3.5 wt.% NaCl solution containing 2 wt.% silica sand [74].

2.3.2 Particle concentration

The influence of particle concentration on material loss has been extensively studied [74–77]. The presence of solid particles at high concentration in a two-phase flow enhances the rate of material loss due to an increased number of particles impacting the metal surface. Figure 2.8 shows the effect of particle concentration on material loss for grey cast iron in a 3.5% NaCl-silica sand slurry. The materials removed due to mechanical erosion increased rapidly with particle concentration at low particle loadings, but the volume loss increased progressively at a much slower rate as the concentration of particles in the slurry was increased further. This could be attributed to the degradation of solid particles as a result of particle-to-particle or particle-to-wall interactions.



Figure 2.8. The variation of material loss with particle concentration for grey cast iron in a slurry containing 3.5% NaCl and silica sand [78].

The impingement of solid particles on metals can cause the protective layers of film on the surface to breakdown, which consequently exposes the bare metal surface to further corrosion and erosion attack. Neville and Hodgkiess [79] investigated the erosion-corrosion behaviour of UNS S32760 super duplex stainless steel and Inconel 625 in 3.5 wt.% NaCl solution containing silica particles. They reported that these alloys exhibited passive corrosion behaviour during pure corrosion study but noted a rapid increase in materials removal rate when 1000 ppm of solid particles was added to the solution. The authors attributed the reported effect of particle addition to the fluid to the breakdown of the adhering passive oxide film.

2.3.3 Impact angle

The severity of material removal from a surface is a function of the angle at which solid particles impinge on the target surface. The influence of impingement angle on materials damage has been widely studied using jet impingement apparatus [9,24,26,80–82]. It has been reported that the maximum erosion-corrosion rate for ductile materials occurred at impingement angles ranging from 15 to 50° [81,83,84]. Azarian *et al.* [85] investigated the effect of impact angles on the synergistic erosion-corrosion behaviour of AA5052 aluminum alloy in a 3.5 wt.% NaCl solution

containing 90 g/l silica sand. They conducted their tests at a constant velocity of 3 m/s and showed that the optimum material loss for pure erosion and erosion-corrosion in a jet impingement apparatus occurred when the particles impinge the alloy's surface at an angle of 30° . Similarly, Andrews *et al.* [82] studied the erosion-corrosion behaviour of a 316 austenitic stainless steel and Stellite 6 alloy at different impingement angles. Tests were conducted using a 3.5 wt.% NaCl solution containing 1.177 g/l SiO₂ at a flow velocity of 19 m/s. They reported that the maximum erosion-corrosion rate for the AISI 316 stainless steel and Stellite 6 alloy occurred for impingement angles of 45° and 60° , respectively.

Based on the above results, slurry condition and materials property influence the particle impact angle at which the target material exhibits the maximum mass loss. The variation of erosion rate with impingement angle for a ductile and brittle material is presented in Fig. 2.9. Ductile materials such as aluminum 1100 exhibit maximum erosion rate at impact angles between 15 to 30° , which is often as a result of severe plastic deformation on the surface. In contrast, the maximum material loss rate for brittle materials such as glass and ceramic occurs at 90° [86].



Figure 2.9. The variation of erosion rate with impingement angle for ductile and brittle materials [26].

2.3.4 Temperature

It is important to develop materials that can withstand high-temperature applications in the aerospace, automobile and minerals processing industries. However, subjecting these materials to elevated temperature applications could consequently alter their wear resistance. The influence of operating temperature on the wear behaviour of different materials has been studied [13,87–89]⁻ Mesa *et al.* [90] investigated the effect of temperature on the erosion-corrosion behaviour of AISI 410 and 420 martensitic stainless steel in a slurry containing sea water and 20 wt.% quartz particles, flowing at 3.5 m/s. They reported that the erosion-corrosion rates of the alloys increased with increasing temperature due to an increase in the corrosion process at higher temperatures. High temperature can increase the ductility of metals and enhance the formation of oxide films. An increase in metal ductility and localized shear deformation at elevated temperature promotes the formation of deep impact craters with lips from which the extruded lips break off during subsequent particle impacts [91,92].

On the contrary, Stack *et al.* [93] suggested that an increase in the corrosion process at high temperature causes thick oxide films to form, which can consequently prevent the particles from impacting directly on the metal surface. Furthermore, Young and Ruff [94] investigated the dry erosion behaviour of a 304 austenitic stainless steel in a gas jet apparatus containing 5 μ m and 50 μ m alumina particles using a jet velocity of 30 m/s. They reported that the material removal rate for the alloy at 25 °C is greater than at 500 °C because thick oxide films protected the metal against particle erosion at 500 °C. Therefore, the effect of temperature on erosion-corrosion can vary depending on the properties of the particles and the materials.

2.3.5 Particle size and shape

The effects of particle size and shape have been investigated by many researchers [15,95–98]. Larger particles with rough/angular edges enhance material damage when compared to smaller particles with a round/dull edge [99]. The effects of particle attrition and impact angle must be considered to fully understand the influence of particle size on the rate of material loss. Kasem [100] investigated the effect of particle size on the erosion behaviour of AISI 5117 steel in a slurry using a whirling arm apparatus. He conducted his erosion tests using a water-sand slurry flowing at a velocity of 15 m/s and reported that mass loss rate increased with an increase in particle size. The variation in the erosion rate and the number of particles striking the surface of aluminum 6063

alloy per hour with particle size, as reported by Desale *et al.* is presented in Fig. 2.10 [95]. The number of particles impacting the surface of the aluminum alloy reduces with an increase in particle size, whereas erosion rate of the aluminum alloy increased. Bree *et al.* [101] proposed that the number of particles impacting on a target surface per hour (N_{SP}) in a slurry pot can be estimated using Eqn. 2.3. This equation was derived based on the assumptions that all particles impinge on the surface, and that they are spherical in shape and are evenly distributed in the slurry.

$$N_{SP} = \frac{\pi D_r A_{WS} \sin \theta N \rho_W P_c 60}{\left(\frac{\pi d_p^3}{6}\right) \rho_p}$$
2.3

where *D* is the rotational diameter of the specimen, A_{ws} is the area of the material, α is the impingement angle, *N* is the rotational speed of the specimen, P_c is the particle concentration, d_p is the particle diameter, ρ_w is the density of water and ρ_p is the density of the particle.



Figure 2.10. Variation in mass loss and the number of particles impacting the surface of AA 6063 at 30 and 90° using different particle sizes [95].

In 1927, Cox [102] proposed that the circularity of sand particles can be estimated using Eqn. 2.4, where circularity is the degree of roundness of a particle. The circularity of a perfectly rounded particle is 1, whereas those with rough or sharp edges are lower than 1. The perimeter of the particle (P) can either be calculated using Eqn. 2.5 or determined using image processing software.

$$Circularity = \frac{4\pi A_p}{P^2}$$
 2.4

$$P = \frac{\pi}{2} \left[\frac{3}{2} (L_P + W_P) - (L_P / W_P)^{1/2} \right]$$
 2.5

where A_p , L_p and W_p are the area, length and width of the particle, respectively. The material damage caused by solid particles with irregular shapes is four times greater than that caused by particles with round shapes [103]. Walker and Hambe [104] reported that the erosion rate of cast iron increased with decreasing particle circularity in slurries containing different concentrations of particles (i.e., alumina, silicon carbide and silica sand), but noted that erosion rate is also a function of particle properties such as density and size.

2.4 Erosion-corrosion models

Mathematical models are commonly used to predict the erosion and erosion-corrosion rates of materials. However, it is challenging to develop a universally accepted predictive model because of the several factors that influence materials behaviour. Mathematical models can be categorized into three: (a) empirical models, (b) semi-empirical models and (c) mechanistic models. Empirical models are best-fit parameters to experimental data and have no theoretical support. In contrast, mechanistic models describe the theory or mechanisms behind a result, whereas semi-empirical models depend on a combination of a theoretical framework and the observed relationship between parameters [105].

2.4.1 Flow-assisted corrosion

Flow-assisted corrosion is the rapid damage to metallic materials due to electrochemical corrosion reactions in a fast-flowing aqueous solution. This form of corrosion is distinct from erosion-corrosion because the corrosive electrolyte contains no solid particles which cause mechanical erosion. The increased damage to a target surface during flow-assisted corrosion is due to the enhanced mass transfer of reacting species and the chemical dissolution of the material. The dissolution rate of metals during flow-assisted corrosion is determined by the electrolyte condition (i.e., the concentration of dissolved oxygen, temperature and pH), hydrodynamic intensity, chemical composition and geometry of the material.

The earlier proposed corrosion models [106,107] are based on the assumption that corrosion rate increased linearly with exposure time, but a linear corrosion growth rate does not occur. The corrosion rate of metallic materials increases non-linearly with increasing exposure time but becomes stable after some time due to the adhering corrosion products on the surface [108,109]. Non-linear corrosion growth model can be categorized into three stages [110]:

- a. the formation of protective oxide layers, which prevents the surface from corroding,
- b. the linear increase in corrosion rate due to the breakdown of the oxide layer,
- c. the slow non-linear increase in corrosion rate until a constant corrosion rate is achieved.

The main drawback of the earlier corrosion models is that they overestimate the corrosion rate and do not apply to flow-assisted corrosion. Keating and Nešic [111] proposed that the corrosion rate (*CR*) of metals can be estimated using Eqn. 2.6.

$$CR = \frac{2k_m C_{b_{O_2}} M_{Fe}}{\rho_{Fe}}$$
 2.6

where M_{Fe} is the molar mass of iron, ρ_{Fe} is the density of iron, C_{bo_2} is the bulk concentration of oxygen and k_m is the mass transfer coefficient for a turbulent flow, which can be calculated using Eqn. 2.7. In contrast, the mass transfer coefficient for a fully developed flow (k_d) can be determined using Eqn. 2.8. [112].

$$k_m = \frac{D C_o}{\Delta y C_b}$$
 2.7

$$k_d = \frac{i_{lim}}{nFAC_b}$$
 2.8

where *D* is the diffusivity of oxygen, Δy is the distance from the wall to the first grid point, C_o is the concentration at that point, C_b is the bulk concentration of reacting species, *n* is the number of moles of electrons, *F* is the Faraday constant, *A* is the surface area of the material and i_{lim} is the limiting current density. The limiting current density can be expressed as [113]:

$$i_{lim} = \alpha n F\left(\frac{D}{d}\right) (R_e)^{\beta} (S_C)^{\gamma} C_b$$
2.9

where while *d* and *D* are the pipe diameter and the diffusion coefficient, respectively. α , β and γ are constants, *S*_{*C*} is the Schmidt number and *R*_{*e*} is the Reynolds number.

2.4.2 Erosion

Erosion models have been developed [41,66,114–121], but they have several limitations, and full information about the assumptions and factors used to generate the models are not known [122]. In 1960, Finnie proposed the earliest known erosion model based on the assumptions that the particle obeys Newton's law of motion, and the target surface was a plastic material with constant flow stress [42]. Finnie used the expressions in Eqns. 2.10 and 2.11 to estimate the volume of materials removed (Q) by a single particle impacting on a ductile surface at a specific angle (θ).

$$Q = C \frac{MV^2}{8p} f(\theta)$$
 2.10

$$f(\theta) = \begin{cases} (\sin 2\theta - 3\sin^2 \theta) & \text{for } \theta \le 18.5^{\circ} \\ \frac{1}{3}\cos^2 \theta & \text{for } \theta > 18.5^{\circ} \end{cases}$$
2.11

where p is the constant plastic flow stress, M is the mass of the impacting particle at a velocity, V and C is a constant.

Bitter [61,123] proposed a modification to Finnie's deformation model for ductile materials by assuming that both deformation (w_D) and cutting (w_C) mechanisms occur concurrently during erosion. Thus, suggesting that the total erosion rate (*E*) of a material can be estimated using Eqn. 2.12. Unlike Finnie's model, which assumed that the impacting particles obeyed the law of motion during deformation wear, Bitter derived his expression (Eqn. 2.13) for deformation wear of ductile materials based on the energy needed to detach a unit volume of material from the target surface. Furthermore, He proposed two cutting wear models (Eqns. 2.14 and 2.15) to estimate the volume loss during erosion of brittle materials. Equation 2.14 is used to estimate volume loss if the horizontal velocity of the particle is not zero after impacting the surface, while Eqn. 2.15 is used when the particle comes to a complete stop after impact.

$$E = w_D + w_C \tag{2.12}$$

$$W_D = \frac{\frac{1}{2}M(V\sin\theta - K)^2}{F_{\varepsilon}}$$
 2.13

$$W_{C1} = \frac{2MC(V\sin\theta - K)^2}{\sqrt{V\sin\theta}} \left[V\cos\theta - \frac{C(V\sin\theta - K)^2}{\sqrt{V\sin\theta}}\rho \right] \quad V \neq 0$$
 2.14

$$W_{C2} = \frac{\frac{1}{2}M(V^2\cos^2\theta - K_1(V\sin\theta - K)^{\frac{3}{2}})}{F_{\rho}} \qquad V = 0$$
 2.15

where *C* is a constant, *V* is the velocity of the particle, *K* is the maximum velocity of the particles when the impact is still elastic, F_{ρ} is the cutting wear factor, and F_{ε} is the deformation wear factor, which is the energy required to remove a unit volume of material.

2.5 Erosion mechanisms

The removal of materials from a surface during erosion is a result of either cutting or deformation wear. Deformation wear occurs when the angle of an impacting particle is normal to the surface of the target material, whereas cutting wear is attributed to the impingement of solid particles on a surface at low angles (i.e., below 90°) as presented in Fig. 2.11. The influence of each mechanism to material loss is a function of either the ductility or brittleness of the material. Material removal on a ductile surface during deformation occurs when the extruded lips around the impact crater fall off due to repeated particle impacts, while it involves the detachment of materials by the cutting action of the impacting particles at low angles. In contrast, materials are removed from a brittle surface by the initiation and propagation of cracks after particle impacts.



Figure 2.11. Schematic diagrams showing the modes of deformation that occur during erosion at low and high impacts: (a) brittle and (b) ductile materials [119].

2.6 Synergistic effect of erosion and corrosion

When both corrosion and erosion simultaneously occur in a multiphase flow system, the synergistic effect of both processes can either accelerate or reduce material damage. Previous investigations on erosion-corrosion have shown that the accelerated removal of materials from a surface occurs as a result of either the enhancement of corrosion rate by the erosion process or the enhancement of erosion rate by the corrosion process [124,125]. The total material loss during erosion-corrosion can be calculated according to the ASTM G119-09 standard using the following equations [126]:

$$T = S + (W_o + C_o) \tag{2.16}$$

where *T* is the total material loss due to erosion-corrosion, W_o is the material loss due to pure erosion, C_o is the material loss due to pure corrosion and *S* is the additional material loss due to the interactions between erosion and corrosion processes. The synergistic component (*S*) is given by Eqn. 2.17.

$$S = \Delta W_c + \Delta C_w$$
 2.17

$$\Delta W_c = W_c - W_o \tag{2.18}$$

$$\Delta C_w = C_w - C_o \tag{2.19}$$

where ΔW_c is the change in erosion rate as a result of corrosion, ΔC_w is the change in corrosion rate due to erosive wear, C_w is the contribution of corrosion to the total material loss and W_c is the contribution of erosion to the overall material loss. Thus, Eqn. 2.16 can be re-written as follow:

$$T = W_o + \Delta W_c + C_o + \Delta C_w$$
 2.20

Rajaram *et al.* [53] investigated the erosion-corrosion behaviour of 316L stainless steel, 1020 steel and a nickel-aluminum-bronze alloy (NAB) in a slurry containing 3.5 wt.% NaCl and silica sand slurry. They determined the contribution of synergy to the total material loss of the tested carbon steel, austenitic stainless steel and NAB to be 5.5%, -11.5% and -63.9%, respectively.

2.7 Mitigation of erosion-corrosion

The repair costs associated with the erosion-corrosion damage of pipelines transporting slurries can be extremely high. Thus, there is little to no chance for components redesign or to change the geometry of the pipes since the main deciding factors in the mineral processing industry are production cost and delivery time. In the absence of an efficient predictive model, routine monitoring of the pipe walls is necessary to determine the extent of damage and warn of an imminent loss of containment. In this section, some of the methods used for mitigating erosioncorrosion damage are reviewed.

2.7.1 Corrosion inhibitor

The use of corrosion inhibitors can effectively hinder the corrosion of metal pipes in the mineral processing industry. Inhibitors are chemical substances that decrease the corrosion rate of metallic materials by forming an adhering film on the corroding metal, increasing the cathodic or anodic polarization of the metal and enhancing the electrical resistance of the metal surface. Thus, protecting the metal surface from the corrosive environment. The effectiveness of an inhibitor is drastically reduced when used in fluids containing solid particles or in systems with high hydrodynamic intensities due to the breakdown of the passive inhibiting film. The efficiency of an inhibitor is a function of the flow regime, fluid temperature, inhibitor concentration and composition. The inhibition efficiency can be estimated using Eqn. 2.21 [127].

Efficiency (%) =
$$\left(\frac{CR_{\text{uninhibited}} - CR_{\text{inhibited}}}{CR_{\text{uninhibited}}}\right) 100$$
 2.21

where *CR* is the material's corrosion rate. It is not clear if the use of chemical inhibitors is an effective approach to mitigating erosion-corrosion since it can potentially alter the composition and grade of the final product. Several erosion-corrosion investigations have been conducted to understand the performance of inhibitors [128–130]. Neville and Wang used a jet impingement rig to evaluate the effectiveness of a chemical inhibitor on the erosion-corrosion behaviour of API-5L-X65 and UNS S41000 in a simulated Forties brine containing 100 and 500 ppm of silica sand [129]. They reported that the inhibitor substantially reduced the material loss for the tested API-5L-X65 and UNS S41000 by 50% and 45%, respectively. Figure 2.12 shows the eroded surface of carbon steel after erosion-corrosion in a water-sand slurry with and without a chemical inhibitor. Plastic deformation and pitting corrosion are the main materials removal processes in the absence

of a chemical inhibitor, while plastic deformation was the sole material removal process when an inhibitor was added to the slurry.



Figure 2.12. Backscattered SEM micrographs obtained for carbon steel after erosion-corrosion in a water-sand slurry: (a) without an inhibitor and (b) with 100 ppm inhibitor [131].

2.7.2 Coatings

Coatings can be used to reduce erosion-corrosion damage since they provide a strong barrier between a material surface and the environments. Coating provides an adhering layer of film that protects the surface of the material against corrosion and erosion damage. It is important to consider operating conditions and the underlying modes of material damage before specifying a suitable coating to prevent premature failure of coatings and the base material. Furthermore, adequate care must be taken to avoid defects (i.e., pores and cracks) during the application of coatings to a target surface to prevent localized corrosion/erosion. Several researchers have investigated the erosion-corrosion behaviour of wear-resistant coatings applied using thermal spraying [132–137], physical vapour deposition (PVD) [138–141] and plasma-transferred arc [76,77,142,143] coating processes.

WC-Co-Cr thermally sprayed coating has been reported to provide excellent protection against slurry erosion-corrosion damage when compared to bare metal surfaces such as austenitic and super duplex stainless steels [133]. However, the extent to which coatings protect a material surface against erosion-corrosion is a function of the operating conditions in which they are being used. Flores *et al.* [142] investigated the erosion-corrosion behaviour of plasma-sprayed tungsten

carbide (WC) coating in a chloride solution containing silica sand and flowing at a velocity of 14 m/s. They reported that the WC coating protected against erosion-corrosion damage by about 70% at 20 °C, but increased damage to the WC coating occurred at 65 °C due to an increase in the corrosion process.

Furthermore, electrochemical method such as electroless plating offers an effective way of protecting against erosion-corrosion. Lin *et al.* [144] investigated the dry erosion behaviour of electroless nickel (EN) and TiAlN/ZrN-PVD coatings on ductile iron, using 5 g of Al₂O₃ particles impacting the surface at 90°. The average velocity of the impacting particles was 83.2 m/s. They reported that both EN and TiAl/ZrN coatings protected the ductile iron against erosion damage, but the application of a duplex TiAlZrN/EN coating on the ductile iron offered the best protection against erosion damage. This is because the EN interlayer between the ductile iron and the TiAl/ZrN overlay enhanced the adhesion strength of the coating. Figure 2.13 shows the images of the worn surface after the dry particle erosion tests. The surface morphology of the coatings shows the occurrence of cracks and impact craters on the eroded surfaces, but the coatings successfully protected the base metal from erosion damage.



Figure 2.13. SEM images for the coated ductile iron after dry erosion test: (a) EN, (b) TiAl/ZrN and (c) TiAlZrN/EN coatings [144].

2.7.3 Materials selection

The proper selection of materials for a specific application is one of the essential ways to prevent the sudden failure of engineering components due to erosion-corrosion. It involves finding the right balance between factors such as corrosion and wear resistance, weldability, life expectancy, availability, and cost. Materials selection is usually the best and most economical method used to mitigate erosion-corrosion damage when compared to other preventative methods. Selecting the best material for an application generally involves three stages [28]:

- a. specifying the application requirements
- b. choosing and assessing the potential materials
- c. final selection of the most cost-effective material.

Several researchers have shown that duplex stainless steels are more resistant to erosion-corrosion damage in comparison to austenitic stainless steel and carbon steels [20,145,146]. Meng *et al.* [20] investigated the erosion-corrosion behaviour of a super duplex (UNS S32760) and an austenitic (UNS S31603) stainless steel in a jet impingement apparatus. They conducted erosion-corrosion tests using a 3.5 wt.% NaCl solution containing silica sand at flow velocities of 7 and 20 m/s. It was reported that UNS S32760 steel exhibited better resistant to erosion-corrosion damage than the UNS S31603 steel. However, they proposed that mechanical erosion was the dominant material removal process for both alloys. Similarly, Neville *et al.* [145] showed that carbon steel experienced the highest material loss, followed by austenitic stainless steel and then duplex stainless steel during erosion-corrosion attack in a slurry containing 3.5 wt.% NaCl solution and silica sand particles flowing at a velocity of 25 m/s.

2.8 Knowledge gap

While the erosion-corrosion behaviour of carbon steel and austenitic stainless steel elbows in NaCl environments have been reported, to the best of the author's knowledge, the literature contains no information on the performance of steel pipes exposed to flow conditions that simulate potash processing. To address this gap, this study used a flow loop to investigate the erosion-corrosion performance of carbon steel and duplex stainless steel elbows in saturated potash brine-sand slurry. The effect of flow velocity and concentration of particles in the slurry was investigated.

Chapter Three Materials and methodology

In this chapter, the methods used for testing and evaluating the erosion-corrosion behaviour of the selected materials are provided. Macrostructural and microstructural characterization techniques such as optical and scanning electron microscopy (SEM), Raman spectroscopy, Surface profilometry, X-ray photoelectron spectroscopy (XPS), energy dispersive spectroscopy (EDS), X-ray diffraction (XRD) and electron backscattered diffraction (EBSD) are discussed. The procedure for the hardness testing method is also covered.

3.1 Test materials

The test materials investigated in this study are schedule 40, buttweld 90° long radius AISI 1018 carbon steel and AISI 2205 duplex stainless steel elbows. The carbon steel and duplex stainless steel elbows were sourced from Apex distribution incorporation and Pinacle stainless steel incorporation, respectively. The schematic diagrams of the elbows showing their dimensions are presented in Fig. 3.1. The chemical compositions of the elbows provided by the suppliers are presented in Tables 3.1 and 3.2. The packing list and certified inspection certificates for the elbows can be found in Appendices A and B. Steel elbows were used as the test materials because severe slurry erosion-corrosion attack in conventional potash processing facilities occurs at regions where there is an abrupt change in slurry flow direction, especially at the elbows and bends.



Figure. 3.1. Schematic diagrams of a schedule 40, 90° long radius elbow used in this study.

С	Mn	S	Si	Р	Mo	Fe
0.19	0.42	0.005	0.22	0.012	0.001	Balance

Table 3.1. Chemical composition (in wt.%) of the AISI 1018 carbon steel elbow.

Table 3.2. Chemical composition (in wt.%) of the AISI 2205 duplex stainless steel elbow.

Cr	Ni	С	Мо	Mn	Si	Р	S	Ν	Fe
22.7	5.25	0.012	3.1	1.03	0.28	0.022	0.0004	0.1855	Balance

3.1.1 Silica sand

The silica particles used in the study was obtained from Target Products Limited, Canada. A Malvern Mastersizer S (long bench) laser diffraction particle-size analyzer was used to determine the distribution of particle size according to ISO 13320:2009 standard [147]. The average size of the silica sand particles was determined to be 1.56 mm. This corresponds to NSF/ANSI 61 standard mesh size of 10 - 20. It is worth mentioning that the size distribution generated by this particle analyzer is based on the volume distribution of the measured particles. The physical and chemical properties of the sand provided by the supplier are presented in Tables 3.3 and 3.4, respectively (see Appendix C). The circularity of the particles before and after erosion-corrosion was determined from a 2D SEM micrograph using ImageJ, an image analysis software [148]. Circularity is a dimensionless shape factor that estimates the curvature of the corners on particles, and it is calculated using Eqn. 2.4. A typical micrograph of the silica sand particles acquired using a JEOL JSM-6010LA scanning electron microscope is presented in Fig. 3.2.

Bulk density	$1474 - 1522 \text{ kg/m}^3$
Hardness	6.5 – 7 Moh
Specific gravity	2650 kg/m ³
Melting point	1610 °C
Boiling point	2230 °C
Shape	Sub-rounded

Table 3.3. Physical properties of the silica sand.

SiO ₂	Al ₂ O ₃	Fe	CaO	MgO	Na ₂ O
93.2 - 93.6	3.6-4.6	0.30 - 0.35	0.25 - 0.65	0.08 - 0.15	0.75 - 0.85

Table 3.4. Chemical composition (in wt.%) of the silica sand.



Figure 3.2. Micrograph of the as-received silica sand particles used in this study.

The concentration of sand particles in a slurry is the ratio of the mass of the particles to the total mass of the slurry. The particle concentration used in this study was estimated using Eqn. 3.1.

$$P_c = \frac{m_s}{m_s + m_l} \tag{3.1}$$

where P_c is the particle concentration (in wt.%), m_s and m_l are the masses for dry silica sand particles and potash brine, respectively.

3.1.2 Potash brine

The potash used to prepare the saturated brine in this study was supplied by Nutrien Ltd., Canada. The saturated potash brine was prepared by dissolving potash ore in reverse osmosis water at 30 °C. Tables 3.5 and 3.6 show the chemical analyses of the potash used in this study, by inductively coupled plasma optical emission spectrometry.

Composition	KCl	NaCl	Insoluble
Mass (wt.%)	95.6	3.2	1.2

Table 3.5. Chemical composition (in wt.%) of the potash used in this study.

Table 3.6. Chemical analysis (in wt.%) of the potash used in this study.

Oxides	wt.%	Anions	wt.%
K ₂ O	60.4	Cl	47.6
Na ₂ O	1.69	Br	0.07
CaO	0.06		
MgO	0.03		
Fe ₂ O ₃	< 0.01		

3.2 Experimental methods

Corrosion, erosion and erosion-corrosion tests were conducted under controlled operating conditions to determine the synergistic effect of erosion and corrosion on the elbow. All the experiments were conducted in a flow loop apparatus. The mode of operation of the flow loop is described in Section 3.2.1. Corrosion tests were conducted using particle-free saturated potash brines. Erosion tests were performed in de-aerated saturated potash brine-sand slurry. The flow loop was continuously deoxygenated by purging with high-purity nitrogen gas (N₂) at a flow rate of 0.5 L/min, to eliminate corrosion during erosion tests. The slurry tank was properly sealed during pure erosion tests to prevent air intake, and the obtained material losses during erosion tests were taken to be as a result of pure erosion damage. However, it is worth mentioning that corrosion could still occur at a reduced rate in the absence of oxygen. Hence, the best way to completely eliminate the corrosion process is by cathodically protecting the elbows.

Erosion-corrosion tests were conducted in aerated slurry consisting of saturated potash brine and silica sand particles, to allow for the combined effects of corrosion and erosion attack on the exposed inner wall of the elbows. The slurry temperature was maintained at 30 ± 1 °C using a Van Waters and Rogers (VWR) circulating bath connected with a heat exchanger coil. Table 3.7 shows a summary of the operating conditions used during experimental investigations.

Test parameters	Corrosion	Erosion	Erosion-corrosion
Temperature (°C)	30	30	30
Velocity (m/s)	2.5 and 4	2.5 and 4	2.5, 3, 3.5 and 4
Particle concentration (wt.%)	-	10, 20, 30, 40 and 50	10, 20, 30, 40 and 50
Test duration (hours)	48 and 120	48 and 120	48 and 120
Dissolved oxygen (mg/l)	5.87 - 6.23	0.02 - 0.04	5.45 - 6.41
Solution	Saturated potash	Deoxygenated saturated potash	Saturated potash

Table 3.7. Operating parameters for the corrosion, erosion and erosion-corrosion tests.

3.3 Test device

The flow loop used in this study was designed and constructed in-house by the groups' research engineers. The schematic diagram of the laboratory-scale flow loop is presented in Fig. 3.3. It consists of six main parts: a peristaltic pump, a pulsation dampener, a flow calibration tank, a slurry tank, a heat exchanger and polyvinyl chloride (PVC) pipes. Each of the test material was embedded in a silicone elbow (not shown in the illustration) and held in place to the adjoining straight pipes at both ends of the elbow with metal clamps.



Figure 3.3. A 3D schematic diagram of the flow loop apparatus used in this study.

The main disadvantage of the recirculating flow loop shown in Fig. 3.3 is that the erosion corrosion aggressiveness of the slurry reduces over time due to particle fragmentation and wear. In an attempt to reduce the degree at which particle degradation occurred, the test duration was reduced from the initially planned 120 hours to 48 hours.

3.3.1 Peristaltic pump

A Verderflex Dura 55 peristaltic hose pump, which is a form of displacement pump consisting of a cast iron casing and a 5 HP motor, was used in this study. The slurry is circulated through the pipes by compressing a flexible ethylene propylene diene terpolymer (EPDM) hose fitted inside the pump with the aid of three sliding shoes/rollers that are attached to the motor. A glycerine-based lubricant is used to reduce the heat generated due to the continuous compression and expansion of the EPDM hose caused by the rollers.

3.3.2 Dampener

A Blacoh pulsation dampener, with a maximum operating pressure rating of 150 psi at 20 °C is installed vertically at the pump discharge manifold to dissipate high-pressure pulsations generated by the pump. The dampener was constructed using polypropylene and consists of an EPDM bladder. The function of the dampener is to reduce system shocks, pipe vibration, pressure fluctuations and fluid hammer.

3.3.3 Temperature controller

The excess heat transferred to the slurry as a result of the energy output from the pump is eliminated to maintain a constant fluid temperature. This was done by pumping water through heat exchanger coils that are fitted around the inner walls of the slurry tank using a 1400 W VWR heated/refrigerated circulating bath. Figure 3.4 shows the image of the temperature controller used in this study.



Figure 3.4. The Van Waters and Rogers heated/refrigerated circulating bath used in this study.

3.3.4 Piping

The pipes are made of a 1-inch nominal diameter, schedule 40 PVC pipe, with a pressure rating of 3100 kPa at 23 °C. The length of the pipes was made to be about 2 m long to achieve a fully developed flow before the slurry impact the elbow surfaces. A transparent PVC pipe was installed in between elbow locations 1 and 2 to ascertain the slurry flow regime within the pipe. Visual inspection confirmed that a fully developed flow was achieved in the straight pipes.

3.3.5 Velocity controller

The flow rate of the slurry was determined by diverting the slurry into the calibration tank at different pump speed, and the time it takes to fill up 4 gallons of the slurry was estimated using a stopwatch. The volumetric flow rate of the fluid flowing through the PVC tubing was calculated using Eqn. 3.2. The full calculations of the actual volumetric flow rate (in GPM) and the pump speed (in Hz) which corresponds to a flow velocity of 2.5 m/s are presented in Appendix D. Figure 3.5 shows the 5 HP pump control box used to regulate the pump speed in this study.



Figure 3.5. The 5 HP pump control box used in this study.

$$F_r = V\pi \left[\frac{d^2}{4}\right] \tag{3.2}$$

where the volumetric flow rate (in m³/s) is F_r , V is the velocity (in m/s) and d is the pipe diameter (in m²).

3.4 Gravimetric analysis

The mass of each elbow was measured before and after exposure to corrosion, erosion or erosioncorrosion conditions to determine the mass loss due to each of these phenomena. After the test, each of the tested elbows was removed from the loop and then cleaned by first immersing in diluted Micro-90[®] cleaning solution for 20 seconds, followed by soft brushing. The Micro-90[®] solution was used to dissolve slimes containing potash, clay or metallic particles from the surface. Lastly, the elbows were thoroughly rinsed with reverse osmosis water, ethanol, and then dried with a Powerfist 8259210 heat gun before measurement. The measurements were done using an OHAUS Adventurer[®] electronic balance, with an accuracy of ± 0.1 mg. Finally, the material removal rate for each test (i.e., corrosion, erosion and erosion-corrosion) was calculated in terms of the mass loss per unit area per unit time.

3.5 Synergistic effect between erosion and corrosion

Determination of the contribution of synergy to the total mass loss of the eroded elbow was done according to the ASTM G119-09 standard [126]. The synergistic effect of corrosion and erosion simultaneously occurring can be estimated as follows:

$$S = T - (W_o + C_o) \tag{3.3}$$

The synergistic component is the increased (or decrease in some cases) material loss resulting from the combined attack of erosion and corrosion processes during the erosion-corrosion test. The synergistic wear rate is the difference between the overall erosion-corrosion rate and the summation of the individual wear rate of erosion and corrosion. The percentage contribution of the synergistic component (*PS*) during erosion-corrosion can be calculated using equation 3.4.

$$PS = \left(\frac{s}{T}\right) 100\%$$
 3.4

3.6 Full factorial statistical analysis

A multiple linear regression analysis (MLRA) method was used to model the relationship between two independent variables (velocity and particle concentration) and a dependent variable (erosioncorrosion rate) by fitting a linear regression equation to the experimental data. A multiple linear regression model is often represented by the following equation:

$$Y = a_0 + a_1 x_1 + a_2 x_2 + a_{12} x_1 x_2 + e 3.5$$

where *Y* is the response variable; whereas x_1 , x_2 and x_n are the independent variables (a measure of descriptive variables that are used in predicting *Y*). The coefficient a_o is the estimated constant; a_1 , a_2 and a_n are constants known as regression coefficients, while *e* is the residual error. The estimated constant is the value at which the fitted regression line intercepts the *Y*-axis. A 5x4 and 5x2 full factorial design matrix were used in this study (see Tables 3.8 and 3.9). A total of 20 and 10 runs were conducted for the duplex stainless steel and carbon steel elbows, respectively, to ensure that all possible combinations of the factors across all levels are considered. The experimental data obtained were analyzed with JMP[®] statistical software (version 14.2.0) and empirical models were generated for both alloys. The statistical significance of the variables was determined using the analysis of variance, with a 95% confidence level.

Run	Pattern	Velocity (m/s)	Particle concentration (wt.%)
1	43	4.0	30
2	41	4.0	10
3	11	2.5	10
4	23	3.0	30
5	34	3.5	40
6	35	3.5	50
7	14	2.5	40
8	25	3.0	50
9	33	3.5	30
10	15	2.5	50
11	44	4.0	40
12	21	3.0	10
13	22	3.0	20
14	32	3.5	20
15	31	3.5	10
16	12	2.5	20
17	45	4.0	50
18	42	4.0	20
19	24	3.0	40
20	13	2.5	30

Table 3.8. The 5x4 full factorial design of experiment for the AISI 1018 steel elbows.

Table 3.9. The 5x2 full factorial design of experiment for the AISI 2205 stainless steel elbows.

Run	Pattern	Velocity (m/s)	Particle concentration (wt.%)
1	11	2.5	10
2	21	4.0	10
3	22	4.0	20
4	13	2.5	30
5	14	2.5	40
6	24	4.0	40
7	12	2.5	20
8	25	4.0	50
9	15	2.5	50
10	23	4.0	30

The significance of the effects of flow velocity, particle concentration, and their interaction on erosion-corrosion rates of the steel elbows was determined using a 95% confidence interval. The null hypothesis is that velocity, particle concentration, and their interaction will not significantly influence the erosion-corrosion rate of the investigated AISI 1018 and 2205 steel elbows. If the *p*-

value in this study is less than 0.05, then the null hypothesis (H₀) would be rejected. Rejecting H₀ implies that the flow parameters and the interaction between them will have a statistically significant effect on erosion-corrosion rate. However, null hypothesis would be accepted if the *p*-value is greater than 0.05. Thus, indicating that the parameters will have no significant influence on the erosion-corrosion rates of the elbows.

3.7 Repeatability tests

The uncertainty associated with the results at different elbow locations was determined by conducting erosion-corrosion tests three times using a freshly prepared slurry and a new set of elbows for each test. The slurry used for the repeatability tests contained 30 wt.% sand particles flowing at 2.5 and 4 m/s. The full reproducibility data obtained for the tested alloys using different flow velocities are presented in Appendix E. The uncertainty was obtained by multiplying the t-distribution value at 95% confidence level with the standard error of the mean. The error associated with the test could be due to the difference in wear mechanisms during particle impacts on the target material or changes in oxygen mass-transfer rate as a result of any variations in the hydrodynamic intensity of the fluid.

3.8 Materials characterization

Several techniques were used to characterize the tested elbows after erosion-corrosion, corrosion and erosion damage during exposure to the potash/silica slurry in the flow loop. Some of the techniques used in this study are X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD) and Electron backscattered diffraction (EBSD).

3.8.1 Optical microscopy and Electron backscattered diffraction

Microstructural examinations of the carbon steel and duplex stainless steel elbows were conducted before and after erosion-corrosion tests. Figure 3.6 shows the image of the optical microscope and the microstructure for the AISI 1018 steel elbows used in this study. The specimens (with an area of 1 cm²) for metallographic observation were sectioned from the elbows using a Buehler IsoMet[®] low-speed precision cutter. The carbon steel specimens were cold-mounted in acrylic resin made from a mixture of VersoCit-2 powder and liquid hardener. This was then pre-grinded using different grades of SiC emery papers (320, 400, 600, 800, 1200 grit sizes), followed by mechanical

polishing with 3 µm MD-Dac and 1 µm MD-Nap cloths using 3 µm MD-Dac and 1 µm MD-Nap polishing suspension, respectively, to achieve a mirror-like surface. The carbon steel surface was finally etched for 20 seconds using a 2 % Nital solution. Microstructural evaluation of the carbon steel elbow was conducted using a Nikon Eclipse MA-100 inverted optical microscope (Fig. 3.6a) interfaced with a PAX-it! image analysis software. The typical micrograph for the as-received carbon steel elbows is presented in Fig. 3.6b. The micrograph revealed a microstructure consisting of 82% ferrite (light area) and 18% pearlite (dark area), which is as expected for an AISI 1018 carbon steel.



Figure 3.6. (a) Nikon Eclipse MA-100 optical microscope and (b) optical micrograph for the asreceived carbon steel elbows.

The sectioned duplex stainless steel elbow was electrolytically polished to prevent the formation of strain-induced martensite on the elbow surface, which can be introduced by both mechanical grinding and polishing. Electropolishing process involves the application of a direct current (DC) to an electrolyte [149]. The duplex stainless steel elbow, which acts as the anode was connected to the positive terminal of a DC power supply, while the cathode (platinum wire) was connected to the negative terminal. The electrodes were then immersed in an electrolyte consisting of 70 ml sulfuric acid, 90 ml phosphoric acid and 40 ml distilled water for 30 seconds, followed by applying a potential of 32 V using the DC power supply. EBSD scans were performed using a SU 6600 Hitachi field emission scanning electron microscope that is equipped with an Oxford Instruments Nordlys Nano EBSD detector at an accelerating voltage of 20 kV. A step size of 1.5 μ m was used for wide-area scans, whereas high-resolution scans were conducted using a step size of 0.15 μ m.

using Oxford Instrument's Channel 5 software. The volume fraction of the fiber components was determined using a 20° tolerance. The EBSD phase map obtained for the as-received duplex stainless steel elbows is presented in Fig. 3.7. The phase map revealed a microstructure consisting of about 50% ferrite and 50% austenite.



250 µm

Figure 3.7. EBSD phase map for the as-received duplex stainless steel elbows.

3.8.2 X-ray photoelectron spectroscopy, Raman spectroscopy and X-ray diffraction

Corrosion products found inside the pits that formed on the inner surface of the elbows were analyzed using XPS and Raman spectroscopy. Both XPS and Raman analyses were conducted at the Saskatchewan Structural Sciences Centre, Saskatoon. XPS was done using a Kratos (Manchester, UK) AXIS Supra spectrometer. A 500 mm Rowland circle monochromated Al K- α (1486.6 eV) radiation source at an accelerating voltage of 15 keV and an emission current of 15 mA was used for the measurement. A test spot size of 55 µm was used to acquire the data.

The XPS survey scan was conducted using 1 eV steps with a pass energy of 160 eV to identify all the species. The survey scan was performed using a binding energy range of -5 to 1200 eV. High-resolution scans of C, O and Fe were also conducted using 0.05 eV steps with a pass energy of 20 eV. Quantitation and deconvolution of the acquired XPS spectra were done with a CasaXPS software (version 2.3.19) using a non-linear least-squares algorithm with Shirley background subtraction and Gaussian-Lorentzian combination.

Raman measurements were performed with a Renishaw InVia Reflex Raman spectroscopy that is coupled with a Peltier cooled CCD detector to determine the phases in the corrosion products. The Raman spectra were acquired with a 20X objective lenses, an argon ion laser operating at a wavelength of 514.5 nm and a laser power of 0.35 mW. The focused laser spot size on the elbow surface is approximately 2µm.

XRD analyses of the eroded and as-received surfaces were performed at the Saskatchewan Structural Sciences Centre, Saskatoon, using a Rigaku Ultima IV diffractometer equipped with a Copper X-ray tube. The X-ray radiation source was set to an accelerating voltage of 40 kV and an emission current of 44 mA before measurement. XRD data were acquired over a 2 θ range of 20 - 120°, with a step of 0.01°. Finally, phase identification of the acquired XRD diffractogram was done using X'Pert HighScore software (version 1.0d) [150].

3.8.3 Scanning electron microscopy and Energy-dispersive X-ray spectroscopy

Macrostructural evaluation of the selected elbows before and after erosion-corrosion was performed using a JEOL JSM-6010LV SEM at an accelerating voltage ranging from 9 to 20 kV. To examine the surface of the internal walls of the elbows, specimens were cut from the elbow entry and exit sections using a Buehler Abrasimet 2 abrasive cutter. Figure 3.8 shows the surface morphologies for the internal walls of the sandblasted carbon steel and the as-received duplex stainless steel elbows used in this study. The observed cracks on the surfaces of the carbon and duplex stainless steel elbows (Figs. 3.8a and 3.8b) could have been caused by the sandblasting and manufacturing processes, respectively.



Figure 3.8. The SEM surface morphologies for the internal walls of the elbows used in this study: (a) sandblasted carbon steel and (b) as-received duplex stainless steel.

EDS analysis was conducted using a SU 6600 Hitachi field emission scanning electron microscope combined with an EDS detector. The EDS phase maps were acquired using an accelerating voltage of 20 keV. EDS analysis was carried out at the Department of Mechanical Engineering, University of Saskatchewan, Saskatoon.

3.8.4 Optical profilometer

Profilometry scans were conducted using a Nanovea® PS50 3D non-contact optical profilometer to obtain 3D surface topographies and to determine the surface roughness of the elbows. Specimens for the surface characterization were cut from the entry, middle and exit regions of the worn elbows. The most important surface roughness parameters are arithmetic mean height and root-mean-square height [29–33]. The arithmetic mean height is the mean surface roughness of the assessed 3D surface topography while the root-mean-square height gives information about the standard deviation of the height distribution. All profilometry scans were conducted according to ISO 25178-3:2012 standard [34]. Surface roughness values presented in this paper are the averages of three scans acquired from different locations on the elbow surface, while the error represent the standard deviation of the mean.

3.8.5 Micro-hardness test

The hardness of the elbow surfaces before and after erosion-corrosion tests were measured using a Mitutoyo MVK-H1 Vickers microhardness testing machine. The elbows were subjected to a load of 1kgf for 10 seconds. The microhardness test on each elbow was repeated eight times, and the mean hardness values of the eight readings are reported in this study. The hardness values were calculated using the correction factors for the Vickers hardness test made on cylindrical surfaces as specified in the ASTM E92-16 standard [151].

Chapter Four

Slurry erosion-corrosion of 90° AISI 1018 steel elbows in saturated potash brine containing abrasive silica particles

In this chapter, the results of experimental investigations on the effect of synergistic interaction between erosion and corrosion on a schedule 40, 90° long radius AISI 1018 carbon steel elbow are discussed. The influence of flow velocity and particle concentration on the behaviour of the elbows in a flow loop containing saturated potash brine-sand slurry are also discussed. The microstructural examination for the exposed internal walls of the elbows was done using SEM, and the results are discussed in this chapter. This chapter has been published in "*Wear*" as follows:

Elemuren R, Evitts R, Oguocha INA, Kennell G, Gerspacher R and Odeshi A, "Slurry erosioncorrosion of 90° AISI 1018 steel elbows in saturated potash brine containing abrasive silica particles". *Wear* 410 (2018) 149-155.

My contributions to this research paper include a review of relevant literature, design and conduction of experiments, analysis of experimental results and preparation of the manuscript. My supervisor and the co-authors reviewed the draft manuscript before it was submitted for publication. The reviewed draft was submitted for publication in *Wear* after implementing their suggestions. The manuscript presented in this chapter is a modified version of the published paper. The experimental procedure of this paper has been removed to avoid repetition, and detailed information about materials and methods are provided in Chapter Three. The copyright permission for manuscript reuse is provided in Appendix F.

Abstract

Erosion-corrosion damage to pipeline materials used in potash processing plants may cause rapid material degradation if the flow velocity and solid particle concentration are high. In this study, the influence of the interaction between erosion and corrosion on AISI 1018 carbon steel elbows was investigated in a flow loop. Experiments were conducted using a slurry containing saturated potash brine and silica sand particles. The effects of particle concentration and slurry flow velocity on the erosion-corrosion rates for the elbows were investigated. Results indicated that increasing flow velocity and particle concentration increased the impacting effect of the solid particles on the

elbow surface, and consequently increased the erosion-corrosion rates of the exposed elbows. The elbows exhibited positive synergy at all elbow locations, where the synergistic components at low velocity are greater than those at high velocity.

Keywords: *Erosion-corrosion; Potash brine; Pitting corrosion; Carbon steel; Wear; Synergy.*

4.1 Introduction

Severe degradation of pipelines frequently occurs in the mineral processing industry due to erosion-corrosion. Failure of pipelines results in rapid plant degradation, leading to environmental pollution, decreased productivity, and increased maintenance costs. Erosion-corrosion damage is more pronounced at fittings where there is a sudden change in fluid flow direction such as pumps, bends, joints, valves and elbows [53,75]. Several researchers have reported that the total material loss due to the synergistic effect of corrosion and erosion during erosion-corrosion is greater than the sum of the material losses experienced when mechanical erosion and corrosion act separately [81,152–157].

Postlethwaite [158] and Hu *et al.* [159] reported that the erosion rate of metals is enhanced by corrosion during erosion-corrosion through roughening of the metal surface which degrades the exposed surface. Similarly, Aminul *et al.* [160] investigated the synergistic effect of erosion and corrosion on API X-70 pipeline steel and found that erosion degraded thin layers of protective corrosion products and created subsurface cracks on the steel surface, thereby increasing the surface roughness of the specimen. Malka *et al.* [51] studied the synergistic erosion-corrosion performance of AISI 1018 carbon steel in 1 wt.% sodium chloride solution containing silica sand, which was sparged with CO₂. They reported that erosion and corrosion by erosion or enhancement of erosion by corrosion.

Despite the numerous erosion-corrosion research investigations carried out in the past, the problem of erosion-corrosion continues to plague the potash processing plants where damage to pipelines transporting potash slurries stands out very clearly amongst other forms of corrosion attack. Therefore, the main goal of the work reported herein is to investigate the erosion-corrosion behaviour of AISI 1018 steel elbows in saturated potash brine-sand slurries using a flow loop.

4.2 Material and methods

4.2.1 Test material

The material used in this study is a schedule 40, 90° long radius AISI 1018 carbon steel elbows described in Section 3.1.

4.2.2 Mass loss

The material removal rate reported in this study is calculated based on the mass loss per unit area per unit time after a test period of 120 hours. Tests were conducted in saturated potash brine-sand slurry containing 10, 20 and 30 wt.% particles. The flow velocities used in this chapter are 2.5, 3.0, 3.5 and 4.0 m/s.

4.2.3 Synergistic effect of erosion and corrosion

The contribution of electrochemical corrosion and mechanical erosion to the total mass loss after erosion-corrosion damage of the AISI 1018 carbon steel elbows was quantified using the synergy equations described in Section 2.6.

4.2.4 Material characterization

The material characterization technique used in this chapter has been described in Section 3.8.3 (SEM). All microstructural evaluation in this chapter was conducted on the internal walls of the exposed elbows.

4.3 Results and discussion

4.3.1 Erosion-corrosion

The material removal rates for the AISI 1018 steel elbows subjected to erosion-corrosion under different test conditions are presented in Fig. 4.1. Erosion-corrosion rates of the elbows increased with increasing particle concentration and flow velocity, which is consistent with the findings of other authors [58,96,161,162]. The increase in erosion-corrosion rate is due to the fact that increasing flow velocity and particle concentration increased the number of solid particles impinging on the elbow surface.



Figure 4.1. Variations in erosion-corrosion rates for AISI 1018 steel elbows with flow velocity and particle concentration. The errors are based on the standard deviations of the mean for all four elbow locations.

Furthermore, it was found that erosion-corrosion rates varied with the locations of where the elbows are installed. Figure 4.2 shows the erosion-corrosion rates of the elbows installed at different locations. The erosion-corrosion rates at elbow locations 1 and 3 are greater than those of locations 2 and 4 under the same test condition. Elbow location 3 consistently had the highest erosion-corrosion rate at all flow conditions, followed by elbow locations 1 and 4, while elbow 2 experienced the lowest erosion-corrosion rate. The high erosion-corrosion rate at elbow location 3 can be attributed to gravitational effect. This is consistent with the data on gravity effect reported by McLaury *et al.* [163] in a study that investigated the effect of sand particles distribution in horizontal and vertical annular multiphase flow in pipes. They reported that the number of solid particles impacting the wall of a horizontally oriented elbow are fewer than those impinging on a vertically oriented elbow.



Figure 4.2. Variations in erosion-corrosion rates obtained for AISI 1018 steel elbows installed at different locations in the flow loop. Erosion-corrosion tests were performed in saturated potash brine-sand slurry containing 30 wt.% particles.

Figure 4.3 shows the surface morphologies of the entry and exit sections for the elbows tested in saturated potash brine-sand slurry containing 10 wt.% particles and flowing at velocities of 2.5 and 4 m/s. At 2.5 m/s, the elbow entry showed that most of the exposed area had been eroded leaving island structures (Fig. 4.3a), while that of the exit section is characterized by both pitting corrosion and plastic deformation (Fig. 4.3c). The surface features of the elbow entry at 4.0 m/s is similar to that of Fig. 4.3a, but the surface morphology of the exit section showed that plastic deformation is the main material removal process at 4 m/s (Fig. 4.3d). This is because high slurry flow velocity increased the kinetic energy of the impacting particles. Thus, enhancing the severity of surface damage at the exit section. The apparent absence of corrosion pits on the exposed surface of the elbow exit suggests that mechanical erosion is the sole material removal process [75], [164].



Figure 4.3. SEM micrographs obtained for AISI 1018 steel elbows exposed to erosion-corrosion conditions using 10 wt.% particles: (a) entry section at 2.5 m/s, (b) exit section at 2.5 m/s, (c) entry section at 4.0 m/s and (d) exit section at 4.0 m/s.

The surface morphologies of the entry and exit sections for the elbows tested in saturated potash brine-sand slurry containing 30 wt.% particles and flowing at velocities of 2.5 and 4 m/s are shown in Fig. 4.4. The surface morphology of the elbow entries (Figs. 4.4a and 4.4b) are similar to those tested in a slurry containing 10 wt.% particles. However, macro-scaled ridges and valleys are formed on the exit section of the elbows as a result of mechanical erosion (Figs. 4.4c and 4.4d). Although the presence of more particles generally increased erosion damage at the exit sections, a few corrosion pits were found on the exposed elbow surface at a velocity of 2.5 m/s. It can be deduced that electrochemical corrosion played a prominent role in material removal during erosion-corrosion at low velocity by attacking weakened layers of the eroded surface and subsequently resulting in the removal of the weakened materials.


Figure 4.4. SEM micrographs obtained for AISI 1018 steel elbows exposed to erosion-corrosion condition using 30 wt.% particles: (a) entry section at 2.5 m/s, (b) exit section at 2.5 m/s, (c) entry section at 4.0 m/s and (d) exit section at 4.0 m/s.

Generally, SEM morphologies of all eroded surfaces showed that erosion-corrosion damage is severe at the exit section of the elbows when compared to the elbow entry, which is attributed to the continuous impingement of solid particles on the internal walls of the elbow. Besides, the impingement of solid particles due to a sudden change in the direction of slurry flow, coupled with the high turbulence associated with the abrupt change in flow direction could cause increased mechanical erosion at the exit section.

4.3.2 Corrosion

The effect of corrosion was investigated by exposing the steel elbows to aerated saturated potash brine flowing at velocities of 2.5 and 4 m/s. Figure 4.5 shows the corrosion rates for the elbows installed at different locations. It is evident that corrosion rates increased with increasing flow velocity (i.e., from 1.64 g/m² h to 3.69 g/m² h at elbow location 3). This can be attributed to the increased diffusion of oxygen from the bulk solution to the elbow surface. The increase in corrosion rate is consistent with the findings reported by Wharton *et al.* [165], they investigated the effect of flow velocities on the corrosion rate of AISI 304L stainless steel in 3.5 wt.% NaCl solution. The SEM images showing the surface morphologies of the exposed elbows are presented in Fig. 4.6. There is clear evidence of pitting corrosion on all the elbow surfaces after exposure to saturated potash brine flowing at velocities of 2.5 and 4.0 m/s. Non-protective films are observed on the elbows tested at a velocity of 2.5 m/s. However, these films were scoured away at 4.0 m/s due to the increased hydrodynamic intensity of the fluid.



Figure 4.5. Variations in corrosion rates obtained for AISI 1018 steel elbows installed at different locations in the flow loop using flow velocities of 2.5 and 4.0 m/s.



Figure 4.6. SEM micrographs obtained for AISI 1018 steel elbows exposed to saturated potash brine for 120 h: (a) entry section at 2.5 m/s, (b) exit section at 2.5 m/s, (c) entry section at 4.0 m/s and (d) exit section at 4.0 m/s.

4.3.3 Erosion

Pure erosion tests were conducted in deoxygenated slurries containing saturated potash brine and 10 wt.% particles. Figure 4.7 shows the material removal rates for the steel elbows installed at different locations after erosion tests in deoxygenated potash brine-sand slurry containing 10 wt.% particles. It is evident that erosion rates at locations 1 and 3 are greater than those of locations 2 and 4 at both flow velocities. This trend is consistent with the erosion-corrosion test results obtained for the elbows installed at different locations (Fig. 4.2). The erosion rate of the exposed elbows increased with increasing flow velocity. For instance, that of elbow location 3 increased from 3.27 g/m^2 h to 16.36 g/m^2 h, when flow velocity was increased from 2.5 m/s to 4.0 m/s. This is due to a greater kinetic energy of the impacting particles at 4.0 m/s.



Figure 4.7. Variations in erosion rates obtained for AISI 1018 steel elbows installed at different locations in the flow loop. Erosion tests were performed in deoxygenated saturated potash brine-sand slurry containing 10 wt.% particles.

4.3.4 Synergistic effect of erosion and corrosion

It is well known that the material loss during erosion-corrosion of a material exposed to a moving corrosive fluid containing solid particles is caused by both erosion and corrosion [14,166–171]. The synergistic effect between corrosion and erosion for the elbows subjected to erosion-corrosion in a slurry containing 10 wt.% particles at flow velocities of 2.5 m/s and 4.0 m/s was calculated using Eqn. 2.16. Table 4.1 shows the influence of synergy on the total material loss rate for slurry flowing at 2.5 and 4.0 m/s in the flow loop.

Figure 4.7 shows the mass-loss rates for the elbows exposed to corrosion, pure erosion and erosioncorrosion at different elbow locations. The interaction between corrosion and erosion resulted in greater material removal rates during erosion-corrosion when compared to the summation of the individual material loss rates caused by corrosion and pure erosion. The exposed elbows showed positive synergy at all locations and flow velocities, which is an indication that the carbon steel elbows are susceptible to erosion-corrosion damage. It is apparent that the percentage of the synergistic effect is greater at 2.5 m/s than at 4.0 m/s for all elbow locations (see Table 4.1). For instance, the percentages of the synergistic component at elbow location 1 are 49% and 12% for flow velocities of 2.5 and 4.0 m/s, respectively. The decrease in the synergistic component at high flow velocity indicates that the influence of the interaction between corrosion and erosion during erosion-corrosion is more pronounced at 2.5 m/s.

Elbow locations	Velocity (m/s)	<i>T</i> (g/m ² h)	<i>C</i> _o (g/m ² h)	<i>W</i> _o (g/m ² h)	$S = T - (W_o + C_o)$ (g/m ² h)	(<i>S/T</i>) x 100 (%)
1	2.5	9.67	1.80	3.15	4.72	49
1	4.0	22.44	3.83	15.96	2.65	12
2	2.5	7.76	1.69	1.14	4.93	64
2	4.0	18.12	3.59	14.43	0.1	1
3	2.5	13.18	1.64	3.27	8.27	63
3	4.0	24.57	3.69	16.36	4.52	18
4	2.5	8.21	1.61	2.64	3.96	48
4	4.0	21.72	3.55	15.51	2.66	12

Table 4.1. Synergistic effect of corrosion and erosion for AISI 1018 steel elbows exposed to a saturated potash brine-sand slurry flowing at velocities of 2.5 and 4.0 m/s.

Furthermore, it is evident that the material removal rates for the exposed elbows after corrosion tests at 2.5 and 4.0 m/s is low. It could be inferred that corrosion plays a substantial role during erosion-corrosion by influencing mechanical erosion, and consequently increased the overall material removal rates at both velocities (see Figs. 4.8a-d).



Figure 4.8. Synergistic effect of erosion and corrosion in potash brine-silica sand slurry containing 10 wt.% particles.

4.4 Conclusions

A continuous flow loop was used to evaluate the erosion-corrosion of 90° AISI 1018 steel elbows in saturated potash brine-sand slurry containing 10, 20 and 30 wt.% particles. The flow velocities of the slurry are 2.5 and 4.0 m/s. The following conclusions are drawn from this study:

- 1. Erosion-corrosion rates for all the exposed AISI 1018 steel elbows increased with increasing particle concentration and slurry flow velocity.
- The synergistic effect between erosion and corrosion is greater at low velocity than at high velocity. The percentage of the synergistic components for elbow location 3 are 63% and 18% at 2.5 and 4.0 m/s, respectively.

- 3. The highest material loss occurred at the exit section compared to the elbow entry. Ridges and valleys formed at the exit of the elbow exposed to slurry containing 30 wt.% particles due to increased severity of mechanical erosion damage.
- 4. Corrosion increased the material removal rates during erosion-corrosion by enhancing the dissolution of the work-hardened layers on the exposed elbow surface during erosion-corrosion.

Chapter Five

Erosion-corrosion of 90° carbon steel elbows in saturated potash slurry: effect of particle concentration on surface roughness

In this chapter, the results of experimental investigations on the effect of particle concentration on the exposed surface of a 90°, schedule 40 AISI 1018 steel elbows are discussed. Erosion-corrosion tests were conducted in a flow loop containing saturated potash brine-sand slurry flowing at a velocity of 4 m/s. Microstructural examination of the exposed elbow surface was investigated using optical profilometer and SEM, while the corrosion products found inside pits after the erosion-corrosion test were analyzed using Energy Dispersive X-ray Spectroscopy (EDS). The influence of different particle concentrations on the hardness of the exposed elbow surface and particle degradation are also discussed in this chapter. This chapter has been published in "*Wear*" as follows:

Elemuren R, Tamsaki A, Evitts R, Oguocha INA, Kennell G, Gerspacher R and Odeshi A, "Erosion-corrosion behaviour of 90° AISI 1018 steel elbows in potash slurries: Effect of particle concentration on surface roughness". *Wear* 430 (2019) 37-49.

My contributions to this paper include a review of relevant literature, design of experiment, conducting all investigations, analysis of experimental results and preparation of the manuscript. My supervisor and the other contributors reviewed the draft manuscript before it was submitted for publication. The final draft document was submitted for publication in *Wear* after implementing their suggestions. The manuscript presented in this chapter is a modified version of the published paper. The experimental procedure of this paper has been removed to avoid repetition, and detailed information about materials and method are provided in Chapter three. The copyright permission for the manuscript's reuse is provided in Appendix F.

Abstract

A 5-day erosion-corrosion experiment was performed to evaluate the effect of particle concentration on the surface roughness of AISI 1018 steel elbows. Erosion-corrosion tests were performed in a flow loop containing 10, 20 or 30 wt.% particles and saturated potash brine. All erosion-corrosion tests were conducted using a slurry flow velocity of 4 m/s in this study. The results obtained from optical profilometry analysis showed that surface roughness is greater at the

elbow entry than that at the exit section of the elbows. SEM examination of the exposed surfaces revealed the presence of gullies and islands on the entry sections, but ridges and valleys formed at the middle and exit regions for high particle concentrations in the slurry. Overall, the results of this investigation showed that surface roughness, mass loss and surface hardness at the elbow exit increased with increasing particle concentration.

Keywords: Erosion-corrosion; Surface roughness; EDS; Wear; Hardness; Particle degradation

5.1 Introduction

With continuous demand for improvement in agricultural productivity due to the world's growing population, there is an increased pressure on potash mining and processing facilities to meet the increasing global need for fertilizer. About 95% of global potash production is used as fertilizer [172]. Low alloy steel is the most widely used engineering material for transporting potash slurries via pipes in potash processing plants. However, the presence of entrained particles in a fast-flowing corrosive liquid gives rise to erosion-corrosion damage, which impairs the integrity of the pipelines. This has consistently led to high maintenance costs in the potash mining and processing plants.

The effect of solid particle concentration on the mechanism of slurry erosion-corrosion damage of pipeline materials is not fully understood. This is because of the difficulties involved in predicting the number of particles impacting the material surface, changes in solid particle size (due to particle degradation), impact velocity and impingement angle, as well as the hydrodynamic intensity of the slurry. Erosion by solid particles has been positively utilized in some engineering processes, such as abrasive deburring and sandblasting. However, the presence of particles in fluids is detrimental to pipeline integrity in the mineral processing industries. Several types of testing equipment are available to investigate the effects of solid particle concentration on slurry erosion or erosion-corrosion performance of metallic materials. These include slurry pot [34,98,173–176], jet impingement [21,79,177,178], and a flow loop [51,179]. A number of researchers have studied the effect of particle concentration on the behaviour of different steel grades when exposed to erosion-corrosion attack [6,19–22,53,145,180]. Telfer *et al.* [96] investigated the effect of particle concentration on the erosion-corrosion of iron, and they found that erosion rate increased within the active region (anodic sites on the metal) due to an increase in particle concentration. However,

increased material removal at high particle concentrations only occurred before the formation of oxide film on the surface at the passivation potential. Rajahram *et al.* [32] reported that increasing particle concentration hindered re-passivation during erosion-corrosion of AISI 316L austenitic stainless steel and subsequently contributed to the enhancement of corrosion by erosion. This is because there is a continuous removal of the passive film due to a large number of particles impinging on the surface. The concentration of particles in a slurry affects the time between particle impingement as well as the composition and thickness of the film formed on metallic materials in a corrosive environment [96].

The surface roughness of a material is an important design parameter because it affects its susceptibility to wear, fatigue and corrosion damage. The impact of entrained particles in a fluid flowing within a pipe can damage the exposed inner walls of the pipe and increase the surface roughness. The resistance to corrosion, erosion and wear is strongly dependent on the surface properties of a material as the surface is in contact with the environment [181]. The formation of multiple micro-galvanic sites as a result of increased surface roughness would further enhance the corrosion rate of a metallic material [32]. Sasaki *et al.* [182] reported that an increase in surface roughness due to erosion reduced the pitting potential of 304L austenitic stainless steel. This could be the primary reason for the enhanced pitting of 304L austenitic stainless steel when exposed to slurry erosion-corrosion. In another research investigation, Aminul *et al.* [160] observed that an increase in the surface roughness of API X-70 pipeline steel enhanced the corrosion rate during erosion-corrosion due to the higher surface area of the eroded surfaces.

Less progress has been made in predicting the slurry erosion-corrosion behaviour of steel elbows in pipelines. This is because of the challenges involved in terms of the different modes of material damage imposed by varying hydrodynamic intensities and the fact that most laboratory-scale equipment does not accurately simulate the actual material loss in mineral processing plants. Thus, the objective of this research investigation was to examine how particle concentration affects the surface roughness of AISI 1018 steel elbows during slurry erosion-corrosion. The surface roughness at the entry, middle and exit sections of the investigated steel elbows was characterized using an optical profilometer and a scanning electron microscope.

5.2 Material and methods

5.2.1 Test material

The material used in this study is a schedule 40, 90° long radius AISI 1018 carbon steel elbows described in Section 3.1.

5.2.2 Mass loss

Erosion-corrosion values reported in this study are calculated based on the mass loss per unit area after a test period of 120 hours. The concentration of particles in the slurry used in this study are 10 wt.%, 20 wt.% and 30 wt.%. All tests were conducted using a flow velocity of 4 m/s.

5.2.3 Material characterization

The material characterization techniques used in this study are described in Sections 3.8.3 (SEM and EDS), 3.8.4 (Optical profilometer) and 3.8.5 (Micro-hardness machine). All characterizations in this study were conducted on the internal walls of the exposed elbows.

5.3 Results and discussion

5.3.1 Surface roughness

The surfaces of the elbows before and after erosion-corrosion were analyzed using a non-contact optical profilometer to obtain 3D surface topographies and surface parameters. The 3D surface roughness values presented in this section for all the test conditions are the means of three scans taken at different locations on the elbow surface. The typical 3D surface topography obtained for the sandblasted elbow before the erosion-corrosion test is shown in Fig. 5.1. The sandblasted surface shows traces of cutting marks caused by the impact of the abrasive particles used in the process [183].

Figures 5.2 - 5.4 show the typical 3D surface topographies obtained from worn surfaces of the exposed elbows tested at location 3 under the different particle loading conditions. The arrows indicate the direction of slurry flow across the surface of the elbows. It can be seen that the entry sections of the exposed elbows (Figs. 5.2a, 5.2b and 5.2c) are similar; they show uneven surfaces with gullies and several islands. However, the extent of erosion-corrosion damage at the elbow entry is severe at 10 wt.% particles, suggesting that corrosion could be the dominant degradation process at low particle concentration. The impact of particles on a ductile surface at low particle

concentration could lead to the generation of a rougher surface and, consequently, result in the nucleation of metastable pits on the surface of the exposed elbows. Therefore, the increased roughness on the elbow surface at low particle concentration could be as a result of the enhanced pitting corrosion during erosion-corrosion, especially in a corrosive chloride environment such as potash. The transformation of a metastable pit into a stable pit is discussed later in this paper. Another possible reason for the uneven surfaces at the elbow entry could be because of pipe misalignment. Pipe misalignment could cause turbulent flow and consequently result in severe erosion damage of the elbows.

For the slurry containing 10 wt.% particles, corrosion pits were observed on the middle section of the exposed elbow (Fig. 5.3a), while that of the exit section (Fig. 5.4a) appear to be smooth. In contrast, ridges and valleys are formed on the middle and exit sections of the elbows subjected to erosion-corrosion conditions using 20 and 30 wt.% particles (Figs. 5.3b, 5.3c, 5.4b and 5.4c). The peaks and valleys on the middle and exit surfaces of the exposed elbows at 30 wt.% particle loading (Figs. 5.3c and 5.4c) are higher than those of the elbows tested using slurry containing 20 wt.% particles (Figs. 5.3b and 5.4c). This is because the presence of more erodents in the slurry could alter the material's damage mechanism as well as the extent of material removal from the surface. The damage on the surface at higher particle loadings occur as a result of severe plastic deformation, which is probably due to the abrasive/sliding action of the impacting particles. Generally, the difference in the damage mechanism at the entry and exit regions of the elbows can be ascribed to the fact that slurry flow condition at the entry region is fully developed. In contrast, flow condition at the elbow exit is not in a steady-state and under the influence of turbulent due to the abrupt change in the direction of slurry flow.



Figure 5.1. A 3D surface topography for the sandblasted AISI 1018 steel elbow used in this study.



Figure 5.2. 3D surface topographies obtained for the entry section of the AISI 1018 steel elbows exposed to saturated potash brine-sand slurry containing: (a) 10 wt.%, (b) 20 wt.% and (c) 30 wt.% particles.



Figure 5.3. 3D surface topographies obtained for the middle section of the AISI 1018 steel elbows exposed to saturated potash brine-sand slurry containing: (a) 10 wt.%, (b) 20 wt.% and (c) 30 wt.% particles.



Figure 5.4. 3D surface topographies obtained for the exit section of the AISI 1018 steel elbows exposed to saturated potash brine-sand slurry containing: (a) 10 wt.%, (b) 20 wt.% and (c) 30 wt.% particles.

To quantitatively compare the surfaces of the AISI 1018 steel elbows before and after erosioncorrosion tests in slurries containing 10, 20 and 30 wt.% particles, a Mountain[®] expert 3D version X software was used to extract pertinent surface height and volume parameters. The surface parameters are summarized in Table 5.1. Figure 5.5 shows the variation in surface roughness (S_a) with particle concentration before and after erosion-corrosion. The surface roughness at the elbow entry after erosion-corrosion test is consistently greater than that of the sandblasted elbow (before erosion-corrosion) and those of the middle and exit sections at all test conditions, except for 10 wt.% particles, where the presence of a few large corrosion pits on the exposed elbow surface increased its surface roughness. This could be due to the increased contribution of electrochemical corrosion to the overall material loss at low particle loading.

Furthermore, it was observed that the surface roughness at the elbow entry after erosion-corrosion decreased from $68.5 \pm 0.26 \ \mu\text{m}$ to $52.0 \pm 1.69 \ \mu\text{m}$ as the particles increased from 10 wt.% to 20 wt.%. It further reduced to $47.3 \pm 0.06 \ \mu\text{m}$ at 30 wt.% particles. The decrease in surface roughness could be due to an increase in erosive damage at higher particle loadings, which probably reduced the effect of electrochemical corrosion attack. However, the surface roughness at the elbow exit after erosion-corrosion increased from $4.0 \pm 0.17 \ \mu\text{m}$ to $6.7 \pm 0.20 \ \mu\text{m}$ on increasing the particles from 10 wt.% to 20 wt.%, respectively. It further increased to $19.7 \pm 1.04 \ \mu\text{m}$ when particle concentration was increased to 30 wt.%. As can be seen in Table 5.1, a similar trend was exhibited by other surface roughness parameters, such as root-mean-square height (Sq) and skewness (Ssk).

In general, increasing the concentration of solid particles during slurry erosion-corrosion could lead to an increase in surface roughness and materials degradation. Abedini *et al.* [60] investigated the effect of particle concentration on the erosion and erosion-corrosion behaviour of Al-brass alloy using a jet impingement rig. They reported that the surface roughness of the eroded Al-brass alloy increased with increasing particle concentration for both erosion and erosion-corrosion study. It is worth mentioning that increasing the concentration of particles in slurries beyond a particle loading threshold could decrease the overall surface roughness of materials. This is because the quantity of particles impacting concurrently on the surface is sufficient to restrict plastic deformation or limit the extension of raised lips around impact craters on the material.



Figure 5.5. Surface roughness obtained for AISI 1018 steel elbows before and after exposure to erosion-corrosion condition at a flow velocity of 4 m/s. The standard deviations are represented by the error bars.

The variation in void volume (V_v) with particle concentration before and after erosion-corrosion is provided in Fig. 5.6. The void volume at the elbow entry after erosion-corrosion tests decreased from 123.0 ± 0.60 µm to 81.5 ± 3.14 µm and 62.4 ± 0.74 µm as particles increased from 10 to 20 and 30 wt.%, respectively. However, the void volume at the elbow exit after erosion-corrosion test increased from 6.4 ± 0.25 µm to 11.7 ± 0.40 µm at 10 wt.% and 20 wt.% particles, respectively. It further increased to 33.2 ± 1.66 µm when the concentration of particle in the slurry was increased to 30 wt.%. This is consistent with other volume parameters, such as Material volume (V_m) and void volume (V_v) presented in Table 5.1. Increasing volume parameters is an indication that more void/pit will be subjected to the aggressive erosion-corrosion condition, consequently increasing materials degradation either by mechanical erosion due to particle impact at normal/oblique angles or by electrochemical corrosion attack due to the retention of corrosive liquid in the void.



Figure 5.6. Void volume obtained for AISI 1018 steel elbows before and after exposure to erosioncorrosion condition at a flow velocity of 4 m/s. The standard deviations are represented by the error bars.

Surface	Before	After erosion-corrosion								
parameters	$\mu \pm \sigma$	10 wt.% particles		20 wt.% particles			30 wt.% particles			
		Entry	Middle	Exit	Entry	Middle	Exit	Entry	Middle	Exit
		$\mu\pm\sigma$	$\mu\pm\sigma$	$\mu\pm\sigma$	$\mu\pm\sigma$	$\mu\pm\sigma$	$\mu\pm\sigma$	$\mu\pm\sigma$	$\mu\pm\sigma$	$\mu\pm\sigma$
Height parameters (in µm)										
Sp	134.8 ±	$248.7 \pm$	342.71 ±	60.1 ±	145.1 ±	81.93 ±	62.6 ±	273.5 ±	$75.87 \pm$	$68.6 \pm$
	5.84	3.03	35.49	35.37	0.48	31.68	15.49	184.73	9.67	2.93
Sq	16.0 ±	82.7 ±	136.98 ±	5.0 ±	62.0 ±	12.13 ±	8.3 ±	57.2 ±	23.12 ±	24.0 ±
	0.27	0.01	19.48	0.12	2.04	1.22	0.31	0.48	5.56	1.09
$\mathbf{S}_{\mathbf{v}}$	$81.2 \pm$	$230.1 \pm$	$601.84 \pm$	$43.7 \pm$	$213.9 \pm$	$58.30 \pm$	$46.4 \pm$	$282.9 \pm$	$80.57 \pm$	$88.8 \pm$
	16.62	56.77	14.55	6.74	4.36	4.09	9.07	140.16	17.87	8.87
Sz	$216.0 \pm$	$478.8 \pm$	$944.54 \pm$	$103.7 \pm$	$359.0 \pm$	$140.24~\pm$	$109.0 \pm$	$556.4 \pm$	$156.45 \pm$	$157.4 \pm$
	19.51	59.80	50.04	35.01	4.29	34.07	24.53	324.88	8.26	6.10
S_{sk}	-0.2 ±	$0.3 \pm$	-1.51 ±	-0.04 \pm	-0.2 ±	-0.11 ±	$0.23 \pm$	-0.7 \pm	$-0.29 \pm$	-0.2 ±
	0.05	0.03	0.10	0.08	0.004	0.09	0.02	0.10	0.17	0.1
$\mathbf{S}_{\mathbf{ku}}$	3.2 ±	$2.4 \pm$	$6.40 \pm$	$4.9 \pm$	$2.3 \pm$	$2.88 \pm$	$2.8 \pm$	3.2 ±	$2.78 \pm$	$2.9 \pm$
	0.24	0.05	0.37	2.27	0.02	0.17	0.23	0.80	0.71	0.19
Volume parameters (in μ m ³ / μ m ²)										
V _{mc}	14.3 ±	$75.4 \pm$	85.97 ±	$4.4 \pm$	62.2 ±	11.03 ±	$7.5 \pm$	62.1 ±	21.52 ±	21.5 ±
	0.52	0.43	3.98	0.31	2.56	0.97	0.09	0.33	7.03	0.89
V _{vc}	18.6 ±	116.2 ±	134.61 ±	$5.8 \pm$	$75.2 \pm$	14.54 ±	10.9 ±	$54.9 \pm$	31.29 ±	$30.8 \pm$
	0.93	0.67	7.20	0.29	2.99	0.8	0.34	0.61	11.26	1.65
V_{vv}	$2.0 \pm$	$6.8 \pm$	$30.37 \pm$	$0.6 \pm$	6.3 ±	$1.47 \pm$	$0.8 \pm$	$7.5 \pm$	$1.88 \pm$	$2.5 \pm$
	0.05	0.08	3.88	0.04	0.16	0.27	0.06	0.15	0.74	0.01
Vm	0.7 ±	3.2 ±	3.79 ±	0.2 ±	1.8 ±	0.51 ±	0.4 ±	1.1 ±	0.75 ±	$0.8 \pm$
	0.07	0.16	0.14	0.01	0.01	0.09	0.03	0.09	0.11	0.12

Table 5.1. 3D surface roughness parameters obtained for AISI 1018 steel elbows before and after erosion-corrosion test at 4 m/s. The uncertainties in the reported measurement represent the standard deviation of the mean.

The SEM micrographs showing the morphologies of the internal wall surfaces of the entry region of the elbow after exposure to erosion-corrosion in a slurry containing 10, 20 and 30 wt.% particles are provided in Fig. 5.7. As can be seen, the surfaces are eroded with evidence of corrosion products and plastic deformation. The surface damage is consistent with the 3D surface topographies presented in Figs. 5.2a-c. However, the extent of erosion-corrosion damage on the exposed elbow surface in a slurry containing 10 wt.% particles (Fig. 5.7a) is more than those of the elbows tested using 20 and 30 wt.% particles (Figs. 5.7b and 5.7c). The increased severity on the exposed surface shown in Fig. 5.7a could be due to the enhancement of erosion by corrosion at low particle concentration. This can occur when the impact of particles on the surface results in the removal of corrosion products and enhances the dissolution of the strain-hardened surface layer [184]. This, in turn, will also increase the surface roughness of the material, as discussed above.



Figure 5.7. SEM micrographs obtained for the elbow entries after exposure to erosion-corrosion in saturated potash slurry flowing at a velocity of 4 m/s: (a) 10 wt.%, (b) 20 wt.% and (c) 30 wt.% particles.

Figure 5.8 shows the SEM surface morphologies for the mid-section of the elbows exposed to erosion-corrosion in a slurry containing 10, 20 and 30 wt.% particles. The inner wall of the exposed elbow subjected to erosion-corrosion in a slurry containing 10 wt.% particles is characterized by numerous micro-sized corrosion pits (metastable and stable) and few larger erosion or corrosion pits (Fig. 5.8a). Corrosion attack during erosion-corrosion resulted in the removal of plastically deformed flakes on the exposed surface and increased the quantity of stress concentration defects such as micro-pits. A gelatinous film covering, which acts as a diffusion barrier between the pitted area and the electrolyte is seen above the metastable pits in Figs. 5.8a and 5.9a. The nucleation and propagation of metastable pits can stop abruptly by re-passivation at low potentials or when the film covering above the pit is washed away. However, a stable pit (Fig. 5.9b) is formed at higher pitting potentials or when the pit is wide enough to grow continuously without the film covering [165,182].

As the concentration of solid particles in the slurry increased to 20 and 30 wt.%, severe mechanical erosion led to the formation of ridges and valleys on the exposed elbow surfaces (see Figs. 5.8b and 5.8c). Increased number of particles impacting the exposed surface of metallic materials could result in strain hardening of the surface and subsequent removal of the strain-hardened surface layer by sliding or abrasion. An increase in particle concentration in a multiphase flow system enhances surface damage through cutting, scratching and plastic deformation [185–187].



Figure 5.8. SEM micrographs obtained for the middle section of the elbows after exposure to erosion-corrosion in saturated potash slurry flowing at a velocity of 4 m/s: (a) 10 wt.%, (b) 20 wt.% and (c) 30 wt.% particles.



Figure 5.9. Schematic diagrams of pit nucleation and growth (a) nucleation of a metastable and (b) stable corrosion pits.

Figure 5.10 shows the SEM surface morphologies for the internal wall surfaces of the elbow exit after exposure to erosion-corrosion in a slurry containing 10, 20 and 30 wt.% particles. The inner walls of the exit region of the exposed elbow after erosion-corrosion in a slurry containing 10 wt.% particle loading is characterized by plastic deformation caused due to the impact of particles on

the elbow surface (Fig. 5.10a). In contrast, ridges and valleys are visible on the surfaces of the exposed elbows tested using a slurry containing 20 and 30 wt.% particle loadings (see Figs. 5.10b and 5.10c). It is proposed that sand particles could accumulate above the elbow surface at higher particle concentrations, thereby preventing other particles from impacting directly on the elbow surface. Thus, the accumulated particles could scour away materials from the surface as the slurry flows over it.



Figure 5.10. SEM micrographs obtained for the elbow exits after exposure to erosion-corrosion in saturated potash slurry flowing at a velocity of 4 m/s: (a) 10 wt.%, (b) 20 wt.% and (c) 30 wt.% particles.

The SEM morphology for the mid-section of the exposed elbow subjected to erosion-corrosion in saturated potash brine-sand slurry containing 10 wt.% particles and the corresponding EDS analysis is shown in Fig. 5.11. The EDS elemental mappings (Figs. 5.11b, 5.11c and 5.11d) of the eroded surface (Fig. 5.11a) suggests that Fe K α 1 (yellow colour) is distributed over the scanned

area. Although traces of O K α 1 (green colour) can be seen on the phase map, its intensity is high in the pits because of the presence of oxygen at high concentrations. The observed silicon (brown colour) on the phase map could be associated with the silica sand particles used as erodent in this study.

Furthermore, point scans inside and outside the pit found on the inner wall of the exposed surface (black and white circles, respectively) revealed the presence of iron, oxygen and other elements. The EDS spectrum acquired outside the pit showed that iron is the dominant element found on the surface (Fig. 5.11e). However, the EDS spectrum of the pit showed a substantial increase in the peak of oxygen while that of iron reduced (Fig. 5.11f). Thus, it can be deduced that the increase in oxygen peak is an indication that the corrosion products found inside the pit probably contain oxides of iron, such as FeO and Fe₂O₃. This increase could be attributed either to the fact that the stagnant electrolyte in the pits has a low pH, which is often more acidic than the flowing external electrolyte or the aggressive pit electrolyte has a high chloride concentration than the overall outer environment, thus resulting in an increased localized corrosion attack in the pit. The occurrence of iron and oxygen at high concentrations is consistent with the results of the elemental mappings acquired from the exposed elbow surface.



Figure 5.11. EDS maps and spectra showing the elemental composition inside and outside the corrosion pits.

5.3.2 Mass loss

Figure 5.12 shows the variations in the mass loss for AISI 1018 steel elbows tested in a slurry containing 10, 20 and 30 wt.% particles flowing at a velocity of 4 m/s. The mass loss increased with increasing particle concentration, which is attributed to an increase in the number of particles impacting the surface of the inner walls of the elbows at higher particle loading. This consequently lead to an increase in the rate of materials removal from the elbow surface. The rate at which

individual particles impinge on the surface decreased at higher particle concentrations, thus increasing material erosion. Upkai *et al.* [188] used a method based on acoustic emission to study the erosive wear of API X-65 steel in a submerged jet impingement rig. They reported that increasing particle concentration led to an increase in acoustic emission (AE) energy. This was attributed to the fact that slurry erosion with more solid particles generated higher AE energy due to frequent particle impacts and subsequently enhanced the erosive wear on the material surface.

Furthermore, a substantial increase in mass loss was observed for the exposed elbows when particle concentration increased from 10 wt.% to 20 wt.% (i.e., an average of 22.3% increase). However, on increasing the concentration of particles in the slurry to 30 wt.%, a further increase in mass loss is seen to be minimal for all the exposed elbows, except at location 3. The slow increment in mass loss with increasing particle concentration at elbow locations 1, 2 and 4 is attributed partly to the reduction in particle size due to increased probability of particle-to-particle interaction, as discussed later in Section 5.3.4. This consequently results in a decrease in the impingement energy of the particles impacting the elbow surface. Another plausible explanation could be because increasing the concentration of particles in the slurry may not necessarily increase the number of particles directly impacting on the elbow surface. It was reported in some previous works that mass loss for a metallic material decreased with increasing particle concentration above a certain threshold of particle loading due to the effect of particle-to-particle interaction [189–192]. In contrast, the observed substantial increase in mass-loss at elbow location 3 is caused by the contribution of gravitational force on the free-flowing slurry.



Figure 5.12. Mass loss per unit area obtained for exposed AISI 1018 steel elbows installed at different locations in the flow loop. Erosion-corrosion tests were performed in saturated potash brine-sand slurry containing 10, 20 and 30 wt.% particles for a slurry flow velocity of 4 m/s.

5.3.3 Hardness

Figure 5.13 shows the surface hardness values obtained for the exposed inner wall surface of the elbow exit. The hardness of the as-received (A-R) elbow increased from 158 ± 10 HV1 to 212 ± 4 HV1 after sandblasting because the impinging action of abrasive particles strain-hardened the surface. The hardness of the sandblasted elbow initially reduced to 196 ± 9 HV1 and 199 ± 3 HV1 after erosion-corrosion tests in a slurry containing 10 and 20 wt.% particles, respectively. The observed reduction in hardness could be because the strain-hardened surface layer was removed by the impacting particles or by metal dissolution. This consequently exposed an underlying softer layer with a reduced hardness to further corrosion or erosion damage. Nevertheless, the measured hardness remained higher than that of the as-received (before sandblasting) elbow. The surface hardness of the exposed elbow further increased to 220 ± 3 HV1 after erosion-corrosion in a slurry containing 30 wt.% particle loading.

Generally, the increase in surface hardness could be due to the induced compressive stresses or strain hardening, which subsequently strengthened the elbow surface at higher particle loadings. However, the presence of a strain-hardened layer on the surface could result in the formation of surface discontinuities, which act as preferential corrosion sites. This could lead to grain loosening at grain boundaries and subsequent removal of the grains by the impacting particles.



Figure 5.13. Surface hardness for elbows before (as-received and sandblasted) and after erosioncorrosion tests in a slurry containing 10, 20 and 30 wt.% particles. The standard deviations are represented by the error bars.

The high surface hardness could be responsible for the observed low mass loss after erosioncorrosion in a slurry containing 30 wt.% particles. Thus, increased hardness due to strain hardening on the surface most likely increased the elbow resistance to erosion-corrosion. The continuous impact of particles may lead to an increase in dislocation density on the surface, which impede dislocation motion and subsequently increases the surface hardness of the material.

5.3.4 Particle degradation

The interactions between solid particles and the inner walls of a target material during slurry erosion-corrosion could lead to a change in particle size and shape. Figure 5.14 shows the particle size distribution for the silica sand used in this study, before and after slurry erosion-corrosion. It can be observed that the size of the particles reduced with increasing particle concentrations (from 10 to 30 wt.%). An increase in particle concentration is bound to enhance particle-to-particle interaction and subsequently lead to particle fragmentation. Goodwin *et al.* [193] reported that the degradation of particles is dependent on factors such as hardness of the target materials, impact velocity and particle size.



Figure 5.14. Particle size distribution plots obtained for the silica sand particles before and after erosion-corrosion tests in a slurry flowing at a velocity of 4 m/s.

Figure 5.15 shows the typical SEM morphologies for the sand particles before and after erosioncorrosion tests. The as-received sand particles have an irregular shape with sharp edges (Fig. 5.15a). However, the sharp edges of the particles became blunt after erosion-corrosion tests, leaving sand particles with roundish/nodular edges (Figs. 5.15b - 5.15d). The increase in particleto-particle interaction at high particle concentration could lead to the breakdown of individual particle or dulling of sharp and irregular edges. Thereby reducing the abrasiveness of the particles on the elbow surfaces. This suggests that change in particle size and shape due to particle fragmentation at high particle concentration could also be responsible for the minimal increase in mass loss that was observed in Fig. 5.12 when particle concentration increased from 20 wt.% to 30 wt.%.



Figure 5.15. SEM micrographs obtained for the sand particles before and after erosion-corrosion tests: (a) as-received, (b) 10 wt.%, (c) 20 wt.% and (d) 30 wt.% particles.

Table 5.2 shows the circularity of the erodent particles before and after erosion-corrosion tests. The circularity of a particle ranges from 0 to 1, where a value of 0 indicates that particles are of an irregular shape with sharp angles, and 1 indicates they are rounded or fully circular. As expected, circularity increased with increasing particle concentration, with the as-received silica particles having the lowest circularity value. The increment in circularity suggests that dulling of particle

edges with increasing particle concentration is due to particle-to-particle or particle-to-wall interactions.

Silica sand	Circularity (-)		
As-received	0.64		
10 wt.% particle concentration	0.76		
20 wt.% particle concentration	0.79		
30 wt.% particle concentration	0.82		

Table 5.2. The circularity of the silica sand particles used in the study.

5.4 Conclusions

Changes in surface roughness and other properties due to erosion-corrosion of AISI 1018 steel elbows after exposure to saturated potash brine-sand slurry were investigated using an optical profilometer, a scanning electron microscope, a mass balance, and a Vickers hardness tester. The following conclusions are drawn from the test results:

- The surface roughness on the internal walls of the exposed elbows after erosion-corrosion tests is location dependent. The average surface roughness at the elbow entry is greater than those of the middle and exit sections.
- 2. Ridges and valleys formed on the internal walls (middle and exit sections) of the elbows at higher particle concentrations (20 and 30 wt.%). Sand particles accumulated on the surface of the elbow at 20 and 30 wt.% particle loadings, resulting in the removal of material from the surface by abrasion/sliding wear as slurry flow over the surface.
- 3. Hardness at the elbow exit increased with increasing particle concentration. The impact of abrasive particles on the internal walls of the elbows strain hardened the surface and led to an increase in surface hardness. Therefore, strain hardening of the elbow surface is one of the factors responsible for the increased resistance of the material to erosion-corrosion at high particle concentration.
- 4. Particle degradation increased with an increasing number of particles in the slurry due to particle-to-particle or particle-to-wall interactions.

Chapter Six

Full factorial, microscopic and spectroscopic study of erosion-corrosion of carbon steel elbows in saturated potash brine-sand slurry

In this chapter, the erosion-corrosion behaviour of a 90°, schedule 40 AISI 1018 steel elbows was studied using full factorial analysis. Examination of the exposed elbow surface was carried out using SEM, XPS and Raman spectroscopy. The experimental results for these investigations are discussed in this chapter, which has been published in "*Tribology International*" as follows:

Elemuren R, Evitts R, Oguocha INA, Kennell G, Gerspacher R and Odeshi A, "Full factorial, microscopic and spectroscopic study of erosion-corrosion of AISI 1018 steel elbows in potash brine-sand slurry". *Tribology International* 142, (2020) 105989.

My contributions to this research paper include a review of relevant literature, design of experiment, conducting all research investigations, analysis of experimental results and preparation of the manuscript. My supervisor and the other contributors reviewed the draft manuscript before it was submitted for publication. The final manuscript was submitted for publication in *Wear* after implementing their suggestions. The manuscript presented in this chapter is a modified version of the published paper. The experimental procedure of this paper has been removed to avoid repetition, and detailed information about materials and method are provided in Chapter three. The copyright permission for manuscript reuse is provided in Appendix F.

Abstract

Carbon steels are commonly used in mineral processing facilities. However, their susceptibility to erosion-corrosion attacks can lead to pipe leakage. Hence, the development of a predictive model that estimates equipment lifespan has become essential. In this study, a developed linear regression model was used to predict the erosion-corrosion rates of carbon steel elbows in potash brine-sand slurry using full factorial analysis. Results showed that velocity, particle concentration and the interaction between them are found to be significant, with velocity having the most statistically significant influence on erosion-corrosion rate. Surface evaluation of the elbows that suffered erosion-corrosion damage indicated pitting corrosion, cutting and horseshoe-shaped depressions

as the primary forms of material damage. Analysis of the corrosion products using Raman spectroscopy and XPS revealed the presence of γ -FeOOH, α -FeOOH, α -Fe₂O₃ and Fe₃O₄.

Keywords: Erosion-corrosion; Full factorial analysis; XPS; SEM; Raman spectroscopy; Wear; Residual plots

6.1 Introduction

Accelerated wear of metallic materials due to the simultaneous actions of mechanical erosion and electrochemical corrosion is a major concern in the minerals processing industries. Erosion-corrosion processes are most common in regions of turbulent flow, where there is an abrupt change in the flow direction, as in the case of elbows. The extent of corrosion damage during erosion-corrosion is influenced by: (i) the temperature of the environment, (ii) the rate of mass transfer of oxygen from the bulk solution to the metal surface, (iii) the rate at which electrochemical reactions occur, and (iv) the concentration of reacting species such as oxygen and chloride ions. Likewise, the severity of erosion damage during erosion-corrosion is a function of (i) the turbulence intensity, (ii) the concentration of entrained particles in the liquid, (iii) the angle of particles impingement on the target material, and (iv) the surface roughness of the exposed material. Lotz and Postlethwaite [50,194] reported that an increase in the surface roughness of a mild steel pipe enhanced the mass transfer of oxygen due to increased turbulence in the system.

The problem of erosion-corrosion has received a lot of attention over the years because it causes leakage of pipelines transporting slurries [23,40,41,118,156,195–201]. The ability to predict the erosion-corrosion rate in minerals processing facilities is essential to ensure the reliability of the equipment and safety of employees. However, developing a model that can predict the erosion-corrosion rates of materials is complicated because of the complexity of the interactions between several independent flow properties (i.e., velocity, particle concentration, equipment type, particle and target material properties), which must be considered. These interactions can also have a substantial effect on erosion-corrosion rates. A good predictive model should be system-oriented, and the extent of its applicability must be specified. The robustness of the model regarding its precise prediction when parameters are altered must also be taken into consideration [202]. Rajahram *et al.* [180] used a semi-empirical model to predict the erosion-corrosion rate of UNS S31603 in a slurry pot erosion tester in which temperature, flow velocity and sand concentration

were varied. They reported that velocity had the most significant effect on erosion-corrosion rate, followed by sand concentration and then temperature.

Several researchers have employed mathematical methods to predict the erosion or erosioncorrosion rates of different engineering materials [42,61,115,118,120,203–205]. Finnie [206] used mathematical analysis to explore the relationship between impact angle and dry erosion of ductile materials. The single-particle model he [206] developed predicted material erosion at low impact angles (i.e., below 20°) under the impingement of multiple particles. However, it underestimated the amount of erosion at high impact angles and predicted no erosion at 90°. The disparities between the predicted and experimental data were ascribed to the fact that the predictive model assumed that particles impinged on a smooth, flat surface. On the contrary, the particles impacted on a rough surface as a result of previous particle impingements.

Furthermore, Keating and Nešic [111] used a combination of Eulerian computational flow dynamics and Lagrangian particle tracking methods to predict erosion-corrosion in a 3D, square-sectioned U-bend. They obtained good consistency between predicted and experimental data, but there were a few discrepancies because the model couldn't predict the underlying flow field. Wang *et al.* [207] used a similar approach to predict the erosion rate in 90° bends, but the Lagrangian particle tracking methods in their model did not incorporate the effect of turbulence on particle motion.

The lack of a thorough understanding of the parameters affecting materials behaviour has made it difficult to estimate erosion-corrosion rate. This has resulted in the development of several models. Unfortunately, the parameters used during laboratory tests from which these models were derived are often different from what is obtainable in the industries where erosion-corrosion is a primary concern. The objective of the study is to use a 5×4 full factorial design to determine the statistical significance of the flow parameters (velocity and particle concentration) and their interaction on the erosion-corrosion rate of AISI 1018 steel elbow in saturated potash brine slurry containing sand particles. The information obtained from the experiment was used to develop a predictive erosion-corrosion model for the 1018 steel elbow. The null hypothesis in the current study is that velocity, particle concentration, and their interaction will have no significant influence on the erosion-corrosion rate of AISI 1018 steel elbow. The developed predictive model was validated by conducting separate erosion-corrosion tests using two concentrations of particles that differ

from the parameters used to develop the model. It is worth mentioning that the limitation associated with the developed regression model is that it cannot be used to accurately predict erosioncorrosion rate using test conditions that fall outside the range of parameters used to develop the model.

6.2 Material and methods

6.2.1 Test material

The material used in this study is a schedule 40, 90° long radius AISI 1018 carbon steel elbows described in Section 3.1. The test elbow was installed at the bottom right corner of the loop since previous studies (Chapters four and five) have shown that materials in this location experienced the highest material loss when compared to the other three locations.

6.2.2 Full factorial analysis

A 5x4 full factorial design matrix was used in this study. A full description of the design of experiment is presented in Section 3.6. The influence of flow velocity, particle concentration, and their interaction on erosion-corrosion rates of the steel elbows was determined using a 95% confidence interval. As mentioned in the introduction, the null hypothesis is that velocity, particle concentration, and their interaction will not significantly influence the erosion-corrosion rate of the investigated AISI 1018 steel elbow. If the *p*-value in this study is less than 0.05, then the null hypothesis (H₀) would be rejected. Rejecting H₀ implies that the flow parameters and the interaction between them will have a statistically significant effect on erosion-corrosion rate. On the contrary, null hypothesis would be accepted if the *p*-value is greater than 0.05. Thus, indicating that the parameters will have no significant influence on the erosion-corrosion rates of the elbows.

6.2.3 Erosion-corrosion

Erosion-corrosion tests were conducted using saturated potash brine-sand slurry flowing at velocities of 2.5, 3, 3.5 and 4 m/s. The concentrations of particles used in this study are 10, 20, 30, 40 and 50 wt.%. Tests were carried out in the flow loop for 48 hours. The erosion-corrosion rates reported in this study are calculated based on the mass loss per unit area per unit time after a test period of 48 hours.

6.2.4 Material characterization

The material characterization techniques used in this study are described in Sections 3.8.2 (XPS and Raman spectroscopy) and 3.8.3 (SEM). All microstructural and macrostructural analyses in this study were conducted on the internal walls of the exposed elbows. The specimens used for microscopic evaluation of the damaged surface were sectioned from the exit region of the elbow using a Buehler IsoMet[®] precision cutting machine. The outer curved surfaces of the exposed inner walls of the elbows were investigated.

6.3 Results and discussion

6.3.1 Regression analysis

The correlation of erosion-corrosion with velocity and particle concentration was analyzed using the least square multiple regression method and the following predictive equation was obtained:

$$E - C = 28.82 + 13.16V + 10.28P + 3.60VP + e$$
 6.1

where *E*-*C* is erosion-corrosion rate while velocity, particle concentration and the interaction between them are denoted as *V*, *P* and *VP*, respectively. Equation 6.1 showed that erosioncorrosion rate had a positive correlation with velocity, particle concentration and the interaction between velocity and particle concentration. An adjusted coefficient of determination (adj R^2) value of 0.9871 and a residual error (*e*) of 1.37 was obtained for the predictive erosion-corrosion model. The adj R^2 values range from 0 to 1 (i.e., 0 to 100%), where 0 implies that the predictive model does not explain any variability in the response data, whereas a total variation in the response variable is represented by a value of 1. The adj R^2 value in this study suggests that the prediction model explains about 99% of the variability in the response data (i.e., erosion-corrosion rate) around its mean. Ideally, a high adj R^2 value is preferred, but it does not imply that the regression model is acceptable [208]. This is the reason why the residuals, which is the difference between the experimental and the predicted data obtained from the model, must be examined. The parameter estimates presented in Table 6.1 test the significance of each variable in the regression model. All the parameters considered in this study had a significant effect on the erosion-corrosion rates of the exposed elbows.

	Estimate	Std Error	t-Ratio	p-value
Intercept	28.82	0.32	89.09	< 0.0001
V	13.16	0.43	30.33	< 0.0001
Р	10.28	0.46	22.46	< 0.0001
VP	3.60	0.61	5.87	< 0.0001

Table 6.1. Parameter estimates for the generated erosion-corrosion model.

It is important to ascertain the adequacy of the model because there is no assurance that the fitted regression is a line of best fit for the erosion-corrosion data. The acceptability of the model (Eqn. 6.1) can be ascertained by analyzing: (i) the plot of residuals versus normal quantile (ii) the plot of residuals versus predicted data and (iii) the histogram of the residuals. Figure 6.1 shows the plot of residual and histogram obtained for the generated erosion-corrosion model in Eqn. 6.1. The normal quantile plot (Fig. 6.1a) is a plot of the actual residuals versus the quantiles of a standard normal distribution. The E-C residuals are scattered around the diagonal line and are within the 95% confidence interval, which indicates the linearity of the erosion-corrosion regression model and that the residuals are normally distributed. A deviation from the diagonal line or if most of the residual falls outside the 95% confidence interval would suggest that the data are not normally distributed [209,210]. The frequency distribution plot of the residuals is shown in Fig. 6.1b. The histogram showed that the data have a normal distribution (bell-shaped) and most of them fall within the -2 and +2 regions, ensuring that no response transformation is needed for the model. Although the distribution curve is slightly skewed to the left, however, no outliers were observed in the data according to the outlier labelling rule suggested by Tukey [211] and Hoaglin et al. [212,213].


Figure 6.1. Erosion-corrosion residuals plots: (a) normal quantile showing that the data are randomly distributed around the diagonal line and are within the 95% confidence interval and (b) histogram.

The plots of erosion-corrosion residuals versus the predicted erosion-corrosion rate derived from the fitted regression model (standard residuals and the studentized residuals) are shown in Fig. 6.2. The data points are scattered around a mean value of zero with no regular pattern (Fig. 6.2a), suggesting that residuals are contained within the 95% confidence horizontal band (as indicated by the red horizontal line (Fig. 6.2b). These patterns indicate that the variance of the experimental errors is constant.



Figure 6.2. Plots of erosion-corrosion residuals (a) standard and (b) studentized.

6.3.2 Erosion-corrosion

The contour plot showing how the erosion-corrosion rate varies with flow velocity and particle concentration is provided in Fig. 6.3. It is observed that erosion-corrosion rates are greater at high velocity and particle concentration. At a flow velocity of 2.5 m/s, the predicted erosion-corrosion rates of the elbows tested in a slurry containing 10 and 20 wt.% particles lie within the same colour band (i.e., less than 15 g/m²h). The erosion-corrosion rates for the elbows tested in a slurry containing 30 and 40 wt.% particles follow a similar trend. The observed similarities in the predicted erosion-corrosion rate could be because the kinetic energy of the impacting particles at 2.5 m/s is not high enough to cause severe material damage when particle concentration was increased from 10 to 20 wt.% and from 30 to 40 wt.%. Another plausible explanation could be because the applied shear stress on the surface of the target material is low due to the slow-moving particles impacting the surface at 2.5 m/s. On the contrary, the predicted erosion-corrosion rates for the elbows are proportional to velocity at all particle concentrations, suggesting that the contribution of velocity to the total materials removal rate is more severe than that of particle concentration. Higher flow velocity is expected to increase surface damage as a result of the greater impact energy of the particles and correspondingly higher shear stress.



Figure 6.3. Contour plot showing the effects of velocity and particle concentration on the erosioncorrosion rate of AISI 1018 steel elbow.

6.3.3 Model sample statistics and validation

The significance of the test parameters on the erosion-corrosion rates of the elbows in saturated potash brine-sand slurry was studied using multiple linear regression analysis. The Pareto plot in Fig. 6.4 shows the influence of velocity, particle concentration and the interaction between velocity and particle concentration. According to the Pareto principle, approximately 80% of the output in any given system is produced by 20% of the input [214–218]. Flow velocity had the highest frequency, followed by particle concentration and the interaction between velocity and particle concentration. This indicates that the cumulative contribution of velocity to erosion-corrosion damage of the elbows is more than that of particle concentration and the interaction between them. Velocity accounted for about 49% of the total erosion-corrosion damage, while the summation of velocity and particle concentration resulted in approximately 87% of the overall damage. The interaction between velocity and particle concentration accounted for only 13% of the material's damage.



Figure 6.4. A ranked order histogram showing the cumulative effect of velocity, particle concentration and the interaction between them on erosion-corrosion damage of the AISI 1018 steel elbows.

Particle-to-particle interactions at high particle concentration could reduce the speed of the impacting particles, thus reducing damage to the material surface at low velocity. However, erosion damage becomes the controlling material removal process at high velocity and particle

concentration as a result of the increased kinetic energy of the particles impinging on the elbow surfaces. The dominance of velocity could also be associated with the increased frequency of particles impinging on the material surface at high velocity, which may cause a change in the flow condition and the depassivation/repassivation rate. The empirical power law equation showing the relationship between velocity and slurry erosion is given by the following equation:

$$E = kV^n \tag{6.2}$$

where *E* is the erosion rate, *k* is a constant, *V* is the velocity and *n* is the velocity exponent. Figure 6.5 shows the influence of velocity on erosion-corrosion rate at various particle concentrations with their corresponding velocity exponents. The estimated velocity exponents at different particle concentrations range from 1.80 to 2.29. Factors such as corrosion and erosion rates, temperature, experimental method, particle and material properties could substantially change the velocity exponent; and is usually reported to vary from 1 to 5 for erosion-corrosion [71,219] and from 2 to 3 for pure erosion of metallic materials [38,200].



Figure 6.5. The plot of erosion-corrosion rate versus velocity, showing the variation of velocity exponents at different particle concentrations.

Validation of an empirical model is important to determine its range of applicability. In the present study, model validation was done by conducting erosion-corrosion tests using particle concentrations that were different to the range of test parameters, but still within the limits of the correlation used in developing the model. Figure 6.6 shows the generated normal quantile plot of the parameters (15 and 25 wt.% particles) used to verify the model. It is seen that erosion-corrosion rates follow a normal distribution since both experimental and validation data lie along the diagonal line. Also, the validation data fall within the 95% confidence interval. Thus, the empirical model (Eqn. 6.1) predicted erosion-corrosion rate of the elbow with good accuracy (Adj $R^2 = 0.9875$ and RMSE = 1.3701).



Figure 6.6. Residual versus normal quantile plot showing the data points used to verify the accuracy of the regression model.

6.3.4 Surface evaluation

Microstructural examinations of the worn elbow surfaces were performed to determine the severity of damage for different test parameters. Figure 6.7 shows the SEM morphologies for the exposed surfaces of the elbow exit after erosion-corrosion in saturated potash brine-sand slurry containing 10 wt.% particles at different flow velocities. The exposed surfaces show the presence of corrosion pits and signs of plastic deformation. Pitting of the elbows occurred because the slurry environment contains chloride ions, which is an aggressive chemical specie that promotes pitting corrosion in steel (i.e., AISI 1018 carbon steel) with low alloying elements. Hence, the alloy composition and slurry chemistry are the main factors affecting the formation of corrosion pits on the exposed elbow surfaces. The elbow subjected to the highest flow velocity of 4 m/s experienced severe surface damage when compared to elbows tested at 2.5, 3.0 and 3.5 m/s, which could be due to the increased plastic deformation on the surface at high velocity.



Figure 6.7. High magnification SEM images obtained for the elbow exit showing the effect of 10 wt.% particle loading on erosion-corrosion at different flow velocities: (a) 2.5, (b) 3.0, (c) 3.5 and (d) 4.0 m/s.

The low magnification SEM micrographs of the surfaces subjected to erosion-corrosion in a slurry containing 50 wt.% particles at velocities ranging from 2.5 - 4 m/s are shown in Fig. 6.8. The formation of corrosion pits and horseshoe-shaped depressions on the exposed surfaces are a result of electrochemical corrosion and plastic deformation, respectively, but could lead to the occurrence of vortex around the depression. Vortices often occur as a result of disturbed/turbulent fluid flow in systems and would lead to an increase in swirling velocities. Increasing swirling velocities around the depressions could reduce localized erosion damage due to the dispersion of the impacting particles. It is expected that increasing slurry flow velocity and particle concentration would significantly reduce the overall contribution of electrochemical corrosion to the damage as suggested in a previous study after a five-day erosion-corrosion test [52]. On the contrary, evidence of electrochemical corrosion attack is visible with the presence of corrosion pits on the surfaces of the elbows tested at 50 wt.% particles. The explanation of the discrepancy between these two studies can be attributed to the variation in the test period. It is possible that electrochemical corrosion attack on the elbow surface within 48 h of testing is severe, whereas the formation of adhering corrosion products or protective film (from degraded particles or slime) on the surface over an extended period (i.e., above 48 h) could decrease the influence of further corrosion attack on the surface. Several authors have reported that the corrosion rate of metallic materials is not constant; it could either increase or decrease rapidly till a steady-state corrosion rate is achieved over time [220–223]. It should also be mentioned that the contribution of localized corrosion attack such as pitting corrosion to the total erosion-corrosion rate could be significantly greater than that of uniform corrosion, which is due to the increased influence of turbulence around the pits.



Figure 6.8. Low magnification SEM images obtained for the elbow exit showing the effect of 50 wt.% particle loading on erosion-corrosion at different flow velocities: (a) 2.5, (b) 3.0, (c) 3.5 and (d) 4.0 m/s.

Figure 6.9 shows the exposed inner walls for elbows subjected to erosion-corrosion tests using 50 wt.% particles. High magnification images show that in addition to the observed pits, worn surfaces are characterized by impact craters and extruded lip, which are signs of plastic deformation due to the impinging effect of the abrasive particles. There was a progressive increase in the severity of surface damage due to increasing flow velocity. The extent to which abrasive particles degrade the surface of a material is determined by factors such as velocity, particle shape and size, hardness and impingement angle of the particle. Some authors [38,224] have hypothesized that a buildup of particle layers at high concentrations could prevent direct particle impingement on material surfaces, and result in materials removal primarily by abrasion/sliding wear as the particles flow parallel to the surface.

Furthermore, the exposed elbow surface in Fig. 6.9c suggests the contribution of cutting wear to the total material removal at a velocity of 3.5 m/s. The occurrence of low angle cutting could be a result of the increased turbulence at high particle concentrations and velocities. This could also occur if the speed of an incoming particle is high enough to displace the particle immediately adjacent to the elbow surface, as illustrated in Fig. 6.10. In contrast, particle-to-particle impingement at angles that are perpendicular to the target surface may lead to the formation of impact craters observed in Fig. 6.9d. In general, cutting removes more material from a ductile surface when compared to wear mechanisms such as sliding. The presence of corrosion pits at higher velocities and particle concentrations could be due to: (i) increased damage on the material surface exposed a wider area to the corrosive potash environment, (ii) higher materials surface roughness and (iii) increasing mass-transfer of oxygen in the system at higher velocities.



Figure 6.9. High magnification SEM images obtained for the elbow exit showing the effect of 50 wt.% particle loading on erosion-corrosion at different flow velocities: (a) 2.5, (b) 3.0, (c) 3.5 and (d) 4.0 m/s.



Figure 6.10. Representation of cutting mechanism due to low angle impingement of particle at high velocity.

6.3.5 XPS analysis of corrosion products

The results of the XPS analysis of the corrosion products found in one of the pits of the elbow tested in a slurry containing 10 wt.% particle at a flow velocity of 2.5 m/s is provided in Fig. 6.11. After erosion-corrosion test, the XPS spectrum of the corrosion product showed C, O and Fe to be the main elements found inside the pit (Fig. 6.11a). The atomic quantitation of the peaks was done using CasaXPS software, and the values obtained are approximately 59.4 at.% C, 31.6 at.% O and 9.0 at.% Fe.



Figure 6.11. (a) XPS survey-scan spectrum obtained for the corrosion product, high-resolution scans of (b) C 1s, (c) O 1s and (d) Fe 2p after a 48-hour erosion-corrosion test in a saturated potash brine-sand slurry containing 10 wt.% particles flowing at a velocity of 2.5 m/s.

The deconvoluted high-resolution XPS spectra of C, O and Fe are shown in Fig. 6.11b-d. The C 1s spectrum shows four photoelectron peaks (Fig. 6.11b), indicating four different carbon environments. The first peak at 284.5 eV can be ascribed to contaminants of hydrocarbons (C-H) and C-C bond; the peaks at 286.4 eV and 288.0 eV is attributed to the carbon atoms bonded to oxygen (C-O bond) [225]. The last peak at 290.0 eV is mainly attributed to shake-up satellite due to the presence of C-C bond [226]. Figure 6.11c shows that O 1s spectrum decomposed into two photoelectron peaks. The low binding energy peak at about 530 eV corresponds to oxides of Fe, such as Fe₂O₃ or Fe₃O₄, while that at the high binding energy peak (532.1 eV) is attributed to hydroxides such as FeOOH and Fe(OH)₃ [227–229].

Fitting of spectral lines for Fe is complicated because it contains several magnetic regions with different orientations and intensities, which leads to peak broadening [229]. It has also been suggested that the broadening of Fe peaks is caused by the interactions of electrostatic inclusion [230]. The high-resolution XPS spectrum for Fe 2p has been deconvoluted into five peaks as shown in Fig. 6.11d. The two distinct peaks (doublet structures) are due to multiplet splitting of Fe 2p spectrum into Fe $2p_{3/2}$ and Fe $2p_{1/2}$. The two photoelectron peaks at approximately 710 eV and 725 eV correspond to Fe³⁺ in Fe₂O₃. The peak at the lowest binding energy (706 eV) is attributed to Fe⁰ [231], while the two satellite peaks at 720 eV and 733 eV are ascribed to Fe³⁺ [232]. The presence of satellite peak structures is due to charge transfer [233] or shake-up processes, which have been attributed to the migration of an electron from a 3d orbital to an empty 4s orbital during ejection of the core 2p photoelectron [234]. Generally, the presence of peaks with binding energies corresponding to compounds such as FeO, Fe₂O₃ or Fe₃O₄ suggests that oxidation of the elbows resulted in the formation of corrosion products inside the pits. It can be inferred from the XPS spectrum of O 1s that most of the oxygen found in the pits are present as oxides with only a small quantity accounting for hydroxides.

Figure 6.12 shows the Raman spectra obtained for an elbow after erosion-corrosion in a slurry containing 10 wt.% particles. Two spots on the exposed elbow surface were selected for Raman analysis: outside the pit (black spectrum) and corrosion product found inside the pit (red spectrum). No peak was detected in the spectrum of the exposed elbow surface outside the pit, but the spectrum of the corrosion product found inside the pit showed peaks at 250, 298, 411, 666 and 1320 cm⁻¹. The peaks at 250 cm⁻¹ and 298 cm⁻¹ are attributed to Lepidocrocite (γ -FeOOH)

[235,236] and Geothite (α -FeOOH) [237], respectively, while the peak at 411 cm⁻¹ is attributed to Hematite (α -Fe₂O₃) [238]. The Raman peaks observed at 666 cm⁻¹ and 1320 cm⁻¹ are attributed to Magnetite (Fe₃O₄) [239,240]. These phases can co-exist as crystalline and amorphous structures, and the fraction of each is dependent on the test conditions [241].



Figure 6.12. Raman spectra obtained with a laser power of 0.35 mW for an AISI 1018 steel elbow subjected to erosion-corrosion in a saturated potash brine-sand slurry containing 10 wt.% particle flowing at a velocity of 2.5 m/s: (a) outside the pit and (b) inside the corrosion pit.

The electrochemical process that resulted in the dissolution of the carbon steel elbow during erosion-corrosion and formation of the corrosion products can be expressed by the following reactions:

Anodic reaction:	$2Fe_{(s)} \rightarrow 2Fe^{2+}_{(aq)} + 4e^{-}$	6.3
Cathodic reaction:	$O_{2(g)} + 2H_2O_{(1)} + 4e^- \rightarrow 4OH^{(aq)}$	6.4
Net reaction:	$2Fe_{(s)} + O_{2(g)} + 2H_2O_{(l)} \rightarrow 2Fe^{2+}_{(aq)} + 4OH^{(aq)}$	6.5

The iron (II) ions will react with hydroxyl ions to produce $Fe(OH)_2$ as indicated in Eqn. 6.6 while the $Fe(OH)_2$ will be further oxidized in the presence of oxygen to $Fe(OH)_3$ (Eqn. 6.7). The overall reaction for the corrosion process is written in Eqn. 6.8.

$$2Fe^{2+}_{(aq)} + 4OH^{-}_{(aq)} \rightarrow 2Fe(OH)_{2(s)}$$

$$6.6$$

$$2Fe(OH)_{2(s)} + \frac{1}{2}O_{2(g)} + H_2O_{(l)} \rightarrow 2Fe(OH)_{3(s)}$$
 6.7

$$2Fe_{(s)} + \frac{3}{2}O_{2(g)} + 2H_2O_{(l)} \rightarrow 2Fe(OH)_{3(s)}$$
 6.8

The Raman peaks in Fig. 6.12 shows that FeOOH, Fe_2O_3 and Fe_3O_4 formed as the stable corrosion products in this study. It has been reported that $Fe(OH)_3$ decomposes to FeOOH and Fe_2O_3 in the presence of oxygen as shown in Eqns. (6.9 and 6.10) [242,243]; but the formation of magnetite occurs when there is a limited supply of oxygen (Eqn. 6.11) [237].

$$Fe(OH)_{3(s)} \rightarrow FeOOH_{(s)} + H_2O_{(l)}$$
 6.9

$$2\text{FeOOH}_{(s)} \rightarrow \text{Fe}_2\text{O}_{3(s)} + \text{H}_2\text{O}_{(l)} \tag{6.10}$$

$$8\text{FeOOH}_{(s)} + \text{Fe}_{(s)} \rightarrow 3\text{Fe}_3\text{O}_{4(s)} + 4\text{H}_2\text{O}_{(l)}$$

$$6.11$$

6.4 Conclusions

In this study, a full factorial statistical investigation was used to analyze experimental results and determine the most crucial operating variable affecting the erosion-corrosion rate of AISI 1018 steel elbows in saturated potash brine-sand slurry. An empirical regression model was developed to determine the contribution of flow velocity and particle concentration to the erosion-corrosion rates of the carbon steel elbows. Validation of the predictive model with an independent data set was done to determine its level of accuracy, and the measured erosion-corrosion rates were within 95% confidence interval of the correlation. The conclusions drawn from this study are:

- 1. The developed empirical model can predict the erosion-corrosion rates of the carbon steel elbows under the specified flow conditions.
- Flow velocity had the most significant influence on erosion-corrosion rate, followed by particle concentration and the interaction between them. The cumulative effect of velocity and particle concentration accounted for about 87% of the total erosion-corrosion rates of

the elbows. The increase in velocity and particle concentration enhanced the severity of surface damage, which is attributed to the rise in the impact energy of the particles and the higher number of particles impinging on the elbow surface.

- 3. Erosion-corrosion damage of the exposed elbows occurred as a result of both corrosion and severe plastic deformation. The permanent surface damage by the formation of crater, cutting and plastic deformation due to the impingement of particles and the dissolution of the carbon steel elbows are the main material removal processes.
- 4. XPS and Raman results showed that corrosion products formed on the tested steel elbows comprise of both iron oxyhydroxides (γ -FeOOH and α -FeOOH) and iron oxides (α -Fe₂O₃ and Fe₃O₄).

Chapter Seven

Synergistic erosion-corrosion behaviour of AISI 2205 duplex stainless steel elbows in potash brine-sand slurry and the associated microstructural changes

In this chapter, the combined actions of corrosion and erosion on the performance of a 90° , schedule 40 AISI 2205 duplex stainless steel elbows are discussed. The microstructural changes that occurred after erosion-corrosion tests were analyzed using XRD and EBSD techniques and the results of the investigations are also discussed in this chapter. The manuscript discussed in this chapter is under review in the "*Journal of Materials Engineering and Performance*" as follows:

Elemuren R, Evitts R, Oguocha INA, Szpunar J, Kennell G, Gerspacher R, Tiamiyu A and Odeshi A, "Synergistic erosion-corrosion behaviour of AISI 2205 duplex stainless steel elbows in potash brine-sand slurry and the associated microstructural changes". *Journal of Materials Engineering and Performance* (Under Review)

My contributions to this paper include a review of relevant literature, design of experiment, conducting all investigations, analysis of experimental results and preparation of the manuscript. My supervisor and the other contributors reviewed the draft manuscript before it was submitted for publication in the *Journal of Materials Engineering and Performance*. The manuscript presented in this chapter is a modified version of the submitted paper. The experimental procedure for this paper has been removed to avoid repetition, and detailed information about materials and method are provided in Chapter three.

Abstract

Erosion-corrosion is a common material degradation problem in mineral processing industries. It occurs as a result of the combined effects of electrochemical and mechanical processes. In this study, AISI 2205 duplex stainless steel elbows were subjected to slurry erosion-corrosion environments in a flow loop apparatus. The slurry consisted of a mixture of sand particles and saturated potash brine using 10, 20, 30, 40 and 50 wt.% particles at flow velocities of 2.5 and 4.0 m/s. The results of the gravimetric analysis revealed that erosion-corrosion rates of the elbows increased as particle concentration was increased from 10 wt.% to 50 wt.% at both velocities. The elbows exhibited negative synergy for all operating conditions, which is attributed to the presence of a passive oxide film on the surface during erosion-corrosion. Microstructural examination of

tested elbow surfaces showed that materials removal occurred by cutting and plastic deformation. Electron backscattered diffraction analysis suggests that plastic deformation of the elbow surface resulting from particle impingement led to the transformation of the metastable austenite phase to martensite. It is estimated that plastic deformation and the associated microstructural changes taking place on the elbow surface occurred approximately 4 μ m below the surface.

Keywords: Duplex stainless steel; phase transformation; EBSD; XRD; erosion-corrosion; synergy.

7.1 Introduction

Duplex stainless steels are used in applications where high resistance to corrosion and good mechanical strength are required. They have a dual-phase microstructure consisting of austenite (γ) and ferrite (α). As the austenite phase in duplex steels are metastable, their exposure to particle impacts at high velocities during erosion-corrosion may lead to deformation-induced martensitic transformation, thereby altering their mechanical properties and corrosion resistance. Giourntas *et al.* [244] investigated the erosion-corrosion performance of austenitic stainless steel, super duplex stainless steel and medium carbon steel in slurries consisting of 3.5% NaCl solution and sand particles. They reported that the super duplex stainless steels showed superior resistance to erosion-corrosion damage when compared to the other tested alloys.

The resistance of duplex stainless steels to corrosion in a wide variety of environments is attributed to the presence of stable chromium oxide (Cr_2O_3) layer on their surface. However, this Cr_2O_3 layer can break down when impacted upon by solid particles during erosion-corrosion, thereby leaving a fresh metal surface to be attacked [11,245]. From several studies on the synergistic effects of erosion and corrosion, it is widely accepted that the combined influence of erosion and corrosion on metallic alloys can either be synergistic (positive synergy) or antagonistic (negative synergy) [125,246–251]. A positive synergy occurs when the overall erosion-corrosion rate is higher than the summation of erosion and corrosion rates, whereas negative synergy occurs when the summation of erosion and corrosion rates is greater than the total erosion-corrosion rate. Wood [250] studied the synergistic erosion-corrosion behaviour of a super duplex stainless steel (Zeron 100) in a multiphase flow of 3 wt.% NaCl solution containing 2 wt.% sand particles using a slurry jet, with particles impinging at an angle of 90°. A negative synergy between erosion and corrosion

was reported for the alloy under this test condition. In another study, Neville and Hu [251] reported positive synergies for two super austenitic stainless steels (UNS S31254 and UNS S32654) and a super duplex stainless steel (UNS S32750) in a 3.5% NaCl solution containing varying concentrations of silica sand particle. The findings from these studies indicate that the synergistic effect of erosion and corrosion for different materials is dependent on flow parameters such as velocity [139], temperature [252], applied potential [253] and particle concentration [198]. These operating parameters can influence the response of passive alloys through the breakdown/repassivation of a stable oxide layer [254], oxygen mass transfer [165,255] and the base material removal rate [256].

Although numerous works have been conducted on the erosion-corrosion behaviour of carbon steels in various media, there is limited information on the resistance of duplex stainless steels to erosion-corrosion attacks in aggressive environments such as potash brines. Therefore, the erosion-corrosion behaviour of AISI 2205 duplex stainless steel elbows and the associated microstructural changes on their inner surfaces are investigated in this study. Erosion-corrosion tests were conducted in saturated potash brine-sand slurry using a laboratory-scale flow loop. Furthermore, a 5×2 full factorial design of experiment was used to determine the statistical significance of velocity, particle concentration and the interaction between these flow parameters on the erosion-corrosion rates of the AISI 2205 steel elbows. The null hypothesis is that velocity, particle concentration will not significantly influence the erosion-corrosion rates of the AISI 2205 steel elbows. A model was done by conducting separate experiments using two flow velocities and concentrations of particles that differ from the parameters used to develop the model.

7.2 Material and methods

7.2.1 Test material

The material used in this study is a schedule 40, 90° long radius AISI 2205 duplex stainless steel elbows described in Section 3.1. The schematic illustration showing the location of microstructural and macrostructural evaluations in this study is shown in Fig. 7.1.



Figure 7.1. Schematic diagrams for the: (a) AISI 2205 duplex stainless steel elbow and (b) elbow exit under study.

7.2.2 Mass loss

Corrosion, erosion and erosion-corrosion tests were performed at flow velocities of 2.5 and 4.0 m/s. Erosion and erosion-corrosion tests were conducted in saturated potash brine-sand slurry containing 10, 20, 30, 40 and 50 wt.% particles. All experiments were carried out in the flow loop for 48 hours. The material removal rate reported in this study is calculated based on the mass loss per unit area per unit time.

7.2.3 Synergistic effect of erosion and corrosion

Corrosion, erosion and erosion-corrosion tests were conducted under controlled test conditions to determine the synergistic effect of erosion and corrosion on the elbows. The contribution of synergy to the total material loss for the AISI 2205 duplex stainless steel elbows is estimated using the equations described in Section 2.6. The percentage contribution of the synergistic component (*PS*) during erosion-corrosion can be calculated using Eqn. 3.4.

7.2.4 Material characterization

Microstructural and macrostructural characterizations of the inner wall surfaces of the elbow exit, along the elbow rolling direction (RD), the transverse direction (TD), and the normal direction (ND) were observed using EBSD (Section 3.8.1), XRD (Section 3.8.2) and SEM (Section 3.8.3)

techniques. The elbow exit was selected because preliminary experimental studies showed it to be the location of maximum erosion-corrosion damage when compared to the elbow entry.

The XRD measurement was conducted on the TD-RD plane (region 1 of Fig. 7.1b), while EBSD scans were performed on both TD-RD and ND-RD planes (regions 1 and 2 of Fig. 7.1b, respectively). The specimen for metallographic examination was prepared for EBSD analysis by the electrolytic polishing process described in Section 3.8.1. All macrostructural examinations in this study were conducted on the internal walls of the exposed elbows. The outer curved surfaces of the exposed inner walls of the elbows were investigated.

7.2.5 Full factorial analysis

The two test variables investigated using the full factorial analysis are particle concentration and velocity. Particle concentration and velocity were investigated at five and two different levels, respectively. Thus, a 5x2 full factorial design matrix was used in this study. A full description of the full factorial experimental design is presented in Section 3.6.

7.3 **Results and discussion**

7.3.1 EBSD analysis of the as-received elbow

The results of EBSD analyses on the ND-RD plane for the as-received elbows are presented in Figs. 7.2 and 7.3. The EBSD phase map in Fig. 7.2a shows the presence of austenite (γ) and ferrite (α) phases with average grain sizes of 9.7 µm and 16.7 µm, respectively (see Fig. 7.3a). An evenly distributed FCC-austenitic phase in a BCC-ferritic matrix is observed, and both phases are elongated along the rolling direction. Phase analysis showed that the microstructure is composed of approximately 50.5% austenite and 49.5% ferrite. The inverse pole figure (IPF) maps depicting the γ (Fig. 7.2b), and the α (Fig. 7.2c) grain orientations show that the crystallographic textures of these phases are substantially different. Most of the α grains are textured towards [111]||TD orientation, whereas γ grains are almost randomly oriented, although most of the γ -grains are oriented near [110]||TD as indicated in Fig. 7.3b. The evolution of favoured misorientation in BCC structures during recrystallization has been attributed to the preferred nucleation and growth of grains with specific orientations such as the [111]||TD in the α phase [257,258]. The difference in the area fractions of texture components in γ and α grains suggests the inhomogeneity in the grains structure, which is due to the inherent properties of the material. The alloy under investigation also

shows the presence of annealing twins (area fraction of 27%) in the γ phase (Fig. 7.2d), which is attributed to the low stacking fault energy of the γ phase [259,260].

The kernel average misorientation (KAM) maps (Figs. 7.2e and 7.2f) indicate the local misorientation or distribution of strain energy present in both phases. It is estimated based on the misorientation ($<5^{\circ}$) that exists between each measured point and its neighbours in the EBSD scan. The local misorientations in both phases are unevenly distributed; they are observed to be higher around the boundaries than in the grains. The KAM values for austenite and ferrite are quantified to be 0.78° and 0.73°, respectively (Fig. 7.3c).



Figure 7.2. EBSD maps obtained for the as-received AISI 2205 duplex stainless steel elbow: (a) phase map, (b) IPF map of austenite, (c) IPF map of ferrite (d) map of twin boundaries, (e) KAM map of ferrite and (f) KAM map of austenite.



Figure 7.3. (a) Grain size distribution, (b) area fraction of selected fiber texture components, and (c) local misorientation distribution of austenite and ferrite phases in the as-received AISI 2205 duplex stainless steel elbow.

7.3.2 Erosion-corrosion rate and synergistic behaviour

There can be inconsistency in erosion-corrosion test data. In this study, adequate measures were taken to limit the error associated with all the tests conducted. The error associated with the repeatability test was calculated to be within \pm 7%, which is reasonable. The effects of particle concentration and flow velocity on erosion and erosion-corrosion rates of the duplex stainless steel elbows are shown in Fig. 7.4. The material removal rates for both erosion-corrosion (Fig. 7.4a) and pure erosion (Fig. 7.4b) increased with increasing particle concentration and velocity. The relationship between erosion or erosion-corrosion rate and particle concentration at each flow velocity can be determined using the following power law equation:

$$W = Ax^b \tag{7.1}$$

where W is the wear rate, x represents particle concentration, A and b are constants which depend on the test material and test condition. The values of A and b for different test conditions are provided in Fig. 7.4.



Figure 7.4. (a) Erosion-corrosion and (b) erosion rates for AISI 2205 duplex stainless steel elbows as a function of particle concentration and velocity after 48-hours in saturated potash brine-sand slurry.

The values for the constants are seen to vary. Large value for *A* indicates an increased mass loss at high velocity, especially for the erosion test. On the contrary, a small value for *b* indicates increased fragmentation of particles due to increased particle-to-particle interaction at higher particle concentration and velocity. It is seen that erosion and erosion-corrosion rates increased practically rapidly with particle concentration at 2.5 m/s. However, they initially increased rapidly with increasing particle concentration but became gradual as particle concentration in the slurry increased to 40 and 50 wt.% at 4.0 m/s. The gradual increase in erosion and erosion-corrosion rates at higher particle concentrations is due to increased impacting particle-to-rebounding particle interactions as well as degradation of the silica sand particles. Particle fragmentation reduces the ability of the particles to remove materials from the surface of the target.

Figure 7.5 shows the material loss rates and the contribution of synergy for the exposed AISI 2205 duplex stainless steel elbows at different particle concentrations and flow velocities. The mass-loss rate for the elbows under corrosion, erosion and erosion-corrosion conditions using different

particle concentrations at velocities of 2.5 and 4 m/s is presented in Figs. 7.5a and 7.5b. There was no material loss when the elbows were exposed to pure corrosion at both flow velocities. The resistance of the elbows to corrosion at 2.5 and 4 m/s is due to the presence of a passive Cr_2O_3 layer on the surface. The erosion rate is consistently greater than that of erosion-corrosion for all flow conditions. The low erosion-corrosion rate can be attributed to continual depassivation and repassivation of the protective oxide layer, which hindered the material removal rate during particle impacts when dissolved oxygen is present in the slurry. In contrast, damage to the inner wall surface of the elbows after exposure to erosion attack in deaerated slurry was due to mechanical erosion. The absence of dissolved oxygen in the slurry could prevent the steel surface from re-passivating once the impacting particles removed the protective oxide film. This resulted in direct impingement of particles on the substrate material.

Figures 7.5c and 7.5d show the variation of synergy with particle concentrations for the two flow velocities. Negative synergy is observed for all particle concentrations. The magnitudes of the negative synergy at different particle concentrations are greater at 4 m/s than at 2.5 m/s, except at 40 wt.% particle loading. The plausible explanation for the negative synergy is that the passive layer of film on the elbow reduced mechanical erosion during erosion-corrosion. Neville *et al.* [34] reported that SAF 2205 duplex stainless steel showed better resistance to erosion-corrosion when compared to UNS S31603 austenitic stainless steel. Wood [250] observed negative synergy for Zeron 100 in a slurry consisting of sand particles and 3.5% NaCl solution, while Rajahram *et al.* [53] reported negative synergy for AISI 316L in different slurries (3.5% NaCl + sand particles, 0.1 M NaOH + sand particles and 0.1 M HCl + sand particles). The occurrence of negative synergy is, therefore, an indication that the AISI 2205 duplex stainless steel elbows have good resistance to erosion-corrosion.

Moreover, the synergistic effect of erosion and corrosion is not always negative. Positive synergy can also occur in materials due to one or more of the following: (i) exposure of a large surface area to corrosion after permanent breakdown of passive oxide film, (ii) surge in corrosion activity on the material due to increased surface roughness after particle impact, (iii) increased mass transfer of corrosive species as a result of changing hydrodynamic intensities and (iv) the removal of strain hardened metal flakes by electrochemical corrosion process which could expose a fresh metal surface to further erosion attack [261]. Rajahram *et al.* [53] reported a positive synergy for erosion-

corrosion of AISI 316L in a slurry containing 0.3M HCl solution and silica sand particles. Other researchers [251,262,263] have also reported positive synergies on several stainless steels under different operating conditions. These research findings suggest that the synergy between erosion and corrosion can be complex and is dependent on target materials and environmental conditions.



Figure 7.5. Corrosion, erosion and erosion-corrosion rates and synergy percentage obtained for AISI 2205 duplex stainless steel elbows at different particle concentrations: (a & c) 2.5 m/s and (b & d) 4.0 m/s.

7.3.3 Microstructural analysis of eroded surfaces

A detailed microstructural investigation of the inner walls of the eroded elbows (TD-RD plane) after erosion-corrosion was carried out using XRD and EBSD techniques to ascertain the effect of particle impacts on the microstructure of the AISI 2205 duplex stainless steel elbows. Typical XRD diffractogram obtained for the elbows before and after erosion-corrosion test in a saturated potash brine-sand slurry containing 50 wt.% particles flowing at a velocity of 4.0 m/s are presented

in Fig. 7.6. As expected, the presence of α and γ phases is confirmed. A closer look at the diffractogram shows a reduction in the intensity of some austenite peaks, while that of some ferrite peaks increased for the elbow subjected to the erosion-corrosion test. This could be because the intensity of particle impingements under the test condition was high enough to cause the inner wall surface of the elbows to deform plastically, thereby leading to austenite-to-martensite transformation (i.e., deformation-induced martensitic transformation). Austenite phase is metastable at room temperature and could transform into martensite when plastically deformed [264,265]. It is difficult to differentiate between ferrite and deformation-induced martensite phases using XRD analysis since both have a BCC structure, and their d-spacing is close to each other [266]. As such, it is proposed that the observed increase in the intensity of ferrite peaks was a result of deformation-induced martensite transformation (DIMT); hence, the martensite formed was recorded as ferrite.



Figure 7.6. Typical XRD diffractogram obtained for the AISI 2205 duplex stainless steel elbow before and after exposure to erosion-corrosion in a slurry containing 50 wt.% sand particles at 4 m/s.

The EBSD phase map and the corresponding pole figures for austenite and deformation-induced martensite are provided in Fig. 7.7. Figure 7.7a shows the EBSD phase map acquired from the eroded surface (TD-RD plane) after erosion-corrosion in a saturated potash brine-sand slurry containing 50 wt.% particles. Traces of the transformed martensite (α '-martensite) can be seen

inside the austenite grain marked with a white broken line boundary. The low volume fraction of α' -martensite in the phase map is attributed to the fact that most of the transformed α' -martensite phase could have been removed during electrolytic polishing. This is because DIMT occurred only in the subsurface region of the plastically deformed elbow after erosion-corrosion. The marked austenite grain in Fig. 7.7a, where traces of α' -martensite is observed, was selected and further analyzed to ascertain any existing orientation relationship between the austenite and α' -martensite phases and the results are shown in Figs. 7.7b and 7.7c.



Figure 7.7. (a) EBSD surface map obtained for the AISI 2205 duplex stainless steel elbow after exposure to erosion-corrosion in a slurry containing 50 wt.% sand particles at 4 m/s. (b and c) $\{111\}\gamma$ and $\{110\}\alpha'$ pole figures for austenite and deformation-induced martensite, respectively.

The coincidence of a pole in the $\{111\}_{\gamma}$ pole figure with one in the $\{110\}_{\alpha'}$ pole figure is an evidence of the existence of Kurdjumov-Sachs [267] orientation relationship ($\{111\}_{\gamma} || \{110\}_{\alpha'}$) between the deformation-induced α' -martensite and the austenite phase (see Figs. 7.7b and 7.7c). This corroborates the XRD results in Fig. 7.6 as it pertains to the occurrence of deformation-induced martensitic transformation. Martensitic transformation in austenitic stainless steels is influenced by stacking fault energy and austenite stability [265,268,269], where the latter is dependent on temperature, strain rate and chemical composition. Further EBSD analysis was carried out on the transverse cross-section (ND-RD plane) of AISI 2205 elbows subjected to erosion-corrosion in a slurry containing 50 wt.% particles to evaluate the microstructural changes which took place on the exposed surfaces. The results are presented in Fig. 7.8. The grains labelled 1 and 2 in Fig. 7.8a are γ and α phases found close to the eroded surface.

The KAM map of the cross-section presented in Fig. 7.8b shows that plastic deformation occurred within a thin layer (~4 μ m) beneath the surface, indicating that the depth of the deformed zone after erosion-corrosion test is low. This explain why some of the transformed martensite was removed after surface preparation for EBSD analysis. The impingement substantially increased the distribution of strain or local misorientation at the subsurface layer after plastic deformation. The yellow and red colours indicate regions with high KAM values, which are found close to the eroded surface and at grain boundaries. The kernel average misorientation increases with an increase in strain energy, localized deformation or misorientation in both phases. The ferrite phase is harder and can resist more deformation when compared to the austenite phase. As such, it is expected that plastic deformation in the austenite grains should be greater than that of the ferrite grains. Thus, an increase in dislocation density or local misorientation often results in higher KAM values of the γ phase than the α phase [270,271].

The IPF map of the cross-section showing the orientation of the grains within the region of interest is presented in Fig. 7.8c. The corresponding IPF triangles of the labelled grains in Fig. 7.8c are shown in Figs. 7.8d and 7.8e. The deformed grains labelled 3 and 4 are depicted by the spread of orientations in the IPF triangle, whereas clustering of orientations such as those of grains 1 and 2 in the IPF triangles indicates undeformed grains. The spread of orientation in the deformed grains suggests that the dominant mechanism of plastic deformation occurred by slip in both austenite

and ferrite phases. The behaviour of the grains is dependent on the applied stress and is a function of the magnitude and orientation of the slip plane and direction (i.e., slip system) after deformation.



Figure 7.8. EBSD cross-sectional maps obtained for the eroded AISI 2205 duplex stainless steel elbow after erosion-corrosion in a slurry containing 50 wt.% particles flowing at a velocity of 4 m/s: (a) phase map, (b) KAM map, (c) IPF map, and (d & e) IPF triangles for austenite grains 1 & 3, and ferrite grains 2 & 4.

Plastic deformation of the austenite phase in stainless steels can result in the formation of two martensitic structures: ε -martensite (hexagonal close-packed) and α' -martensite (body-centred cubic). The order of martensitic transformation in stainless steels could follow $\gamma \rightarrow \varepsilon$ -martensite $\rightarrow \alpha'$ -martensite, $\gamma \rightarrow \alpha'$ -martensite or $\gamma \rightarrow \varepsilon$ -martensite [268,272]. ε -martensite is formed as an intermediate phase during the initial transformation stage. Deformation-induced martensite resulting from plastic deformation has been shown by some authors to exhibit better wear properties than γ and α phases [199,273,274]. However, the formation of a hard-martensitic phase could be detrimental to the wear resistance of stainless steels because its inherent brittleness can enhance material removal by microcracking during continual particle impingement. Brittle chipping of the material surface into smaller pieces and their removal by subsequent particle impingement can occur [275].

7.3.4 Microscopy and profilometry of the deformed surfaces

Figure 7.9 shows the SEM images obtained for the internal wall surface of the elbows after exposure to erosion-corrosion in a slurry containing 10 wt.% particles flowing at velocities of 2.5 and 4.0 m/s. The white arrows show the direction of slurry flow over the exposed elbow surfaces. The eroded surfaces revealed plastic deformation as the sole material removal process, which often occurs during solid particle erosion as a result of sliding or low-angle impact of the abrasive particles. The observed surface cracks in Fig. 7.9a are believed to be carried over from the asreceived elbow (see Fig. 3.9c) and indicates that surface damage at the elbow entry is minimal at low velocity and particle concentration. Closer examination of the micrographs shows an increase in the severity of surface damage due to particle impact, with elbows tested at 4 m/s experiencing the highest concentration of plastic deformation. This is attributed to the increased impact momentum of the particles at 4 m/s as compared with 2.5 m/s, which increased the level of plastic deformation/surface damage and rate of material removal. It is proposed that the repeated impacts of particles high enough to cause the elastic limit of the target material to be exceeded.



Figure 7.9. SEM micrographs showing the worn surfaces of AISI 2205 duplex stainless steel elbows after erosion-corrosion tests in saturated potash brine-sand slurry containing 10 wt.% particles: (a) elbow entry at 2.5 m/s, (b) elbow entry at 4.0 m/s, (c) elbow exit at 2.5 m/s and (d) elbow exit at 4.0 m/s.

Figure 7.10 shows SEM images obtained for the eroded surfaces of AISI 2205 steel elbows tested in a slurry containing 30 wt.% sand particles. It is apparent that increasing particle concentration from 10 to 30 wt.% resulted in more severe deformation and cutting of the surfaces of the exposed elbows at both 2.5 and 4 m/s. The observed cutting on the elbow surfaces occurred due to particles impinging on the surface at a low angle. The combined cutting and abrasive actions of the impacting particles can enhance surface deformation and subsequent detachment of the extruded lips by further particle impacts. Solid particles move freely at low concentrations because the tendency for them to interact with each other is minimal [12]. This could cause direct impingement of particles on the surface and increasing the propensity for cutting to occur, especially at lowangle impingement.



Figure 7.10. SEM micrographs showing the worn surfaces of AISI 2205 duplex stainless steel elbows after erosion-corrosion tests in saturated potash brine-sand slurry containing 30 wt.% particles: (a) elbow entry at 2.5 m/s, (b) elbow entry at 4.0 m/s, (c) elbow exit at 2.5 m/s and (d) elbow exit at 4.0 m/s.

Figure 7.11 shows the micrographs for the eroded surfaces of AISI 2205 elbows after erosioncorrosion in slurries a slurry containing 50 wt.% particles. The entry regions (Figs. 7.11a and 7.11b) of the eroded surfaces are characterized by plastic deformation. On the other hand, the exit sections (Figs. 7.11c and 7.11d) are characterized by plastic deformation and impact craters. Craters usually occur when particles impinge on the target material surface at 90°. Thus, displacing the surrounding material outward in the form of an extruded lip; material loss at normal impact occurs by removal of the extruded lip after subsequent particle impacts.



Figure 7.11. SEM micrographs showing the worn surfaces of AISI 2205 duplex stainless steel elbows after erosion-corrosion tests in saturated potash brine-sand slurry containing 50 wt.% particles: (a) elbow entry at 2.5 m/s, (b) elbow entry at 4.0 m/s, (c) elbow exit at 2.5 m/s and (d) elbow exit at 4.0 m/s.

The SEM images in Figs. 7.10 and 7.11 show that surface damage features of the exposed elbows at different test conditions are different for the elbow entry and exit sections. The elbows tested at high velocities and high particle concentrations experienced severe material loss by a combination of plastic deformation, crater, and cutting erosion. These material removal modes are common damage mechanisms for ductile materials. It is believed that the turbulence resulting from a change in flow conditions within the elbows led to the variations in wear modes. 3D surface topographies

obtained for the exit region of the elbows before and after exposure to a potash slurry containing 50 wt.% sand particles are presented in Fig. 7.12. Figure 7.12a shows the internal wall surface (outer curve) of the elbow after erosion-corrosion at a flow velocity of 4.0 m/s, whereas the typical surface topography of the as-received elbow is shown in Fig. 7.12b. The black arrows indicate the direction of slurry flow over the surface of the elbows. The manifestation of ridges and valleys on the surface of the eroded elbows after erosion-corrosion at 2.5 and 4 m/s (Figs. 7.12c and 7.12d) shows that plastic flow in the direction of slurg caused the removal of material from the surface.



Figure 7.12. Internal wall surface for the eroded AISI 2205 duplex stainless steel elbows after erosion-corrosion tests: (a), 3D surface topography for the as-received elbow (b), the eroded elbows after erosion-corrosion tests in a slurry containing 50 wt.% particles at 2.5 m/s (c) and 4.0 m/s (d).

As expected, the eroded surfaces exhibited greater surface roughness (i.e., $10.1 \pm 1.9 \mu m$ at 2.5 m/s and $20.2 \pm 2.4 \mu m$ at 4.0 m/s) than the as-received surface (i.e., $7.1 \pm 0.5 \mu m$). The increase is attributed to the enhanced influence of erosive wear at higher flow velocity and to the fact that the maximum peak-to-valley height and width between the peaks/ridges at 4.0 m/s is greater than at 2.5 m/s. The absence of corrosion products on the elbow surface after erosion-corrosion tests suggest that the observed surface damage is due solely to mechanical erosion.

7.3.5 Statistical analysis

This analysis aimed to develop an empirical model to predict the erosion-corrosion rates of the exposed steel elbows as a function of velocity and particle concentration. The following predictive equation was obtained using the least square multiple regression method:

$$E - C = 39.86 + 27.12V + 21.74P + 12.61VP + e$$
 7.2

The erosion-corrosion rates of the exposed elbows had a positive correlation with velocity (V), particle concentration (P) and the interaction between velocity and particle concentration (VP). A residual error (e) of 3.38 was obtained for the model. Figure 7.13 shows a plot of the actual erosion-corrosion rate versus the predicted erosion-corrosion rate. It is evident that the data lies along a diagonal line, and they are within the 95% confidence limit. This is an indication that the erosion-corrosion rates for all test conditions are normally distributed. A value of 0.9902 was obtained for the adj R^2 , which suggests that the empirical model explains about 99% of the variability in erosion-corrosion rate. The parameter estimates for the model are presented in Table 7.1. It tests the significance of the variables on erosion-corrosion rate in the regression model. All the variables investigated in this study were found to have a significant effect on the erosion-corrosion rates of the exposed steel elbows.



Figure 7.13. A plot of observed erosion-corrosion rate versus model's prediction.

Table 7.1	. Parameter	estimates	obtained	for the	generated	erosion-	-corrosion	model
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	Estimate	Std Error	t-Ratio	p-value
Intercept	39.86	1.07	37.31	< 0.0001
V	27.12	1.07	25.38	< 0.0001
Р	21.74	1.51	14.39	< 0.0001
VP	12.61	1.51	8.34	< 0.0002

The adequacy of the empirical model was confirmed by evaluating the plot of residuals. Figure 7.14 shows the residual plot obtained for the erosion-corrosion model (see Eqn. 7.2). The residuals are randomly scattered around a mean value of zero (blue line), suggesting that residuals are within the 95% confidence limit (red horizontal band). These patterns indicate that the variance in the residuals is constant.



Figure 7.14. A plot of the erosion-corrosion residuals obtained for the regression model.

7.3.6 Model sample statistics and validation

The significance of velocity, particle concentration and the interaction between these test variables is provided in the Pareto plot shown in Fig. 7.15. The velocity of the slurry was found to have the most significant influence on the erosion-corrosion rates of the exposed elbows, followed by particle concentration. The slurry flow velocity accounted for about 53% of the overall material removal rate, while the concentration of particles in the slurry accounted for approximately 30% of the erosion-corrosion rate. The interaction between particle concentration and velocity explained only 17% of the total material loss.



Figure 7.15. A Pareto chart showing the cumulative effect of the test parameters on the erosioncorrosion rates of the exposed AISI 2205 steel elbows.

Figure 7.16 shows the interaction effect between the minimum (red diagonal line) and maximum (blue diagonal line) erosion-corrosion rates for velocity and particle concentration. The significant of the interaction can be evaluated by the distance between the minimum and maximum erosion-corrosion rates. Ideally, the lines should not be parallel to each other if there is a significant interaction between the variables. The interaction plots show that the influence of particle concentration on the erosion-corrosion rate of the exposed elbow is more significant at high velocity, whereas that of velocity is greater at high particle concentration. Hence, it can be inferred that the exposed duplex stainless steel elbow is more susceptible to erosion-corrosion damage at higher velocities and particle concentrations. This is mainly attributed to the increased number of particles impacting the elbow surface at higher test conditions.


Figure 7.16. The interaction plot of velocity and particle concentration. It shows the influence of test variables on the overall erosion-corrosion rate for the exposed AISI 2205 steel elbows.

Validation of the obtained regression model in Eqn. 7.2 was conducted by exposing the elbows to erosion-corrosion conditions using saturated potash brine-sand slurry containing 15 and 45 wt.% particles. The flow velocities used for validating the model are 3.0 and 3.5 m/s. The plot of the actual versus the predicted erosion-corrosion rates is presented in Figure 7.17. It is evident that the erosion-corrosion data obtained for these test conditions lies along the red diagonal line. This is an indication that the regression model predicted the erosion-corrosion rate of the elbow with good accuracy.



Figure 7.17. A plot of actual versus predicted erosion-corrosion rate showing the test parameters used to validate the regression model.

7.4 Conclusions

The synergistic erosion-corrosion behaviour of AISI 2205 duplex stainless steel elbows in saturated potash brine-sand slurry and the associated microstructural changes were investigated in this study. The surface damage due to erosion-corrosion was analyzed using SEM, XRD, EBSD and OP techniques. The following conclusions are drawn from the test results:

- Deformation-induced martensitic transformation occurred during erosion-corrosion of AISI 2205 steel elbows as a result of plastic deformation caused by the repetitive impact of particles on the material surface. Martensite formed in a zone only a few micrometres below the eroded surface.
- 2. The AISI 2205 steel elbows exhibited negative synergy at all particle concentrations and flow velocities investigated in this study.
- Electrochemical corrosion of the elbows did not occur. Hence, the only material removal process during erosion and erosion-corrosion of the AISI 2205 elbows is mechanical erosion resulting from particle impingement.

Chapter Eight Summary, conclusions and recommendations for future work

8.1 Summary and conclusions

In this research, the erosion-corrosion behaviour of carbon steel and duplex stainless steel elbows in saturated potash brine-sand slurry was investigated using different test conditions. The effect of the synergy between corrosion and erosion on the overall materials removed from the surface of both alloys was also investigated. An empirical model was developed using full factorial analysis and the test parameter with the most significant influence on erosion-corrosion rate was determined. Finally, the microstructural changes associated with the duplex stainless steel elbows after erosion-corrosion damage was studied. Examinations of the exposed surfaces before and after erosion-corrosion tests were done using EBSD, EDS, OP, SEM, XRD, XPS and Raman spectroscopy. The main findings from this research are highlighted as follows:

- Erosion-corrosion rates for all the exposed AISI 1018 carbon steel and AISI 2205 duplex stainless steel elbows increased with an increasing concentration of particles and flow velocity. The amount of materials removed from an exposed surface were found to be dependent on the location which the elbows were installed, with elbow location 3 on the flow loop experiencing the highest material loss.
- The effect of the synergy between corrosion and erosion is greater at low velocity than at high velocity. The carbon steel elbows exhibited positive synergy for all the erosioncorrosion test conditions, whereas a negative synergy is determined for the duplex stainless steel elbows.
- 3. Damage to the surfaces of the carbon steel elbows occurred as a result of particles impacting the surface (mechanical erosion) and the dissolution of the carbon steel elbows (electrochemical corrosion). The mechanisms by which materials were removed from the exposed carbon steel surfaces include the formation of crater, pitting corrosion and plastic deformation, which produces easily removable lips. The corrosion products found inside the pits of the exposed carbon steel elbows are Lepidocrocite, Hematite and Magnetite. The materials removal from the duplex stainless steel elbows during erosion-corrosion occurred by mechanical erosion, resulting from particle impacts.

- 4. At higher particle concentrations, ridges and valleys formed on the internal walls of the elbow (middle and exit regions). The presence of sand particles at high concentrations caused materials to be removed from the surface by abrasion/sliding wear as slurries flow over the surface.
- 5. The surface roughness on the internal walls of the elbows after erosion-corrosion damage is location dependent. The presence of numerous islands and gullies on the elbow entry increased its average surface roughness when compared to the middle and exit regions of the elbows.
- 6. The empirical models developed in this research can be used to predict the erosioncorrosion rates of carbon steel and duplex stainless steel elbows under the specified flow conditions. Flow velocity was found to have the most statistically significant influence on erosion-corrosion rates of the exposed elbows, followed by particle concentration and then the interaction between the flow conditions.
- 7. For the carbon steel elbows, approximately 49% of the overall materials removed from the exposed surface was because of velocity, while the contribution of particle concentration was 38%. The overall contribution of velocity to the erosion-corrosion rate of the duplex stainless steel elbows is approximately 53%, and the concentration of particles in the slurry accounted for about 30% of the erosion-corrosion rate. The interaction between particle concentration and velocity accounts for only 13% and 17% of the total materials removed from the carbon steel and duplex stainless steel elbows, respectively.
- 8. After erosion-corrosion test, the hardness for the exposed internal walls of the carbon steel surface increased with an increasing concentration of particles in the slurry. The impact of particles on the internal walls of the carbon steel elbows strain-hardened the surface and caused a rise in surface hardness. Hence, strain hardening of the material surface is one of the factors responsible for the increased resistance of the material to wear at high particle concentrations.

9. When subjected to the repetitive impact of particles at high velocity, some austenite phase of the exposed duplex stainless steel elbows transformed into martensitic at high velocity as a result of plastic deformation. Although the duplex stainless steel elbows exhibited negative synergy for all the test conditions, they are susceptible to erosion damage at high velocity because martensite is a hard and brittle phase. Martensite transformation only occurred a few micrometres (~4 µm) below the exposed surface.

8.2 Recommendations for future work

- 1. There are several important parameters that influences the erosion-corrosion performance of engineering materials, but this research focused only on the effects of flow velocity and particle concentration. It is suggested that further investigation on the effects of dissolved oxygen content, temperature, particle size and shape on the performance of the selected alloys should be carried out.
- 2. The pressure difference along the pipe length caused by the flow resistance of the slurry moving through the pipes, and the frictional force between the exposed internal walls of the pipes and the slurry can affect the overall materials removed from the elbow surfaces. Therefore, investigation of the effect of pressure gain/drop along the vertical and horizontal pipes should be considered.
- The deposition of hard-coatings on a target surface could enhance the corrosion and wear resistance of engineering materials used in the mining and mineral industries. Hence, the erosion-corrosion behaviour of hard coatings in potash brine-sand slurry should be explored.
- 4. Although the current flow loop can be modified to allow for the impingement of particles at an angle, it is not possible to fully control the angle of impact without utilizing a fixed nozzle. Hence, for accelerated materials testing and ranking purpose only, the construction of a standardized slurry jet impingement apparatus is recommended.

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Appendix A

Packing slip and certified inspection certificate for the ordered AISI 1018 carbon steel elbows.

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v	400	06323	6860365	E 2-1/2 STD LR 90 WELD ELL WPB		0.200	0.400	0.015	0.007 0	0.240 0	0.010 0.	0 100.	010 0.0	020 0.	100	0.27	495	325	35	150	1
9	300	63187	6860381	E 3 STD LR 90 WELD ELL WPB		0.190	0.370	0.014	0.013 0	0.200 6	0:030 0	0 100	010 0.	010 0.	100	0.26	494	346	33.5	151	1
-	320	00609	6860420	E 4 STD LR 90 WELD ELL WPB		0.210	0.540	0.010	0.003 0	0.230 6	0.020 0	.001 0	006 0.	010 0.	100	0.31	502	317	36.5	140	1
00	50	60427	6860446	E S STD LR 90 WELD ELL WPB		0.210	0.550	0.013	0.004 0	1,210 6	0.010 0	001 0	010 0.	010 0.	100	0.31	480	303	31.5	140	-
6	350	63119	6860462	E 6 STD LR 90 WELD ELL WPB		0.210	0.550	0.023	0.013 0	0.230 6	0.030 0	010 0	010 0.	010 0.	100	0.31	492	306	34	150	-
10	200	1C920	6860489	E 8 STD LR 90 WELD ELL WPB		0.190	0.400	0.021	0.006 0	0.240 6	0.052 0	001 0	.036 0.	027 0.	100	0.27	485	305	33	141	-
11	50	65810	6860513	E 10 STD LR 90 WELD ELL WPB		0.210	0.550	0.016	0.006 0	0.240 6	0.070 0	0 100.	.040 0.	010 0	100	0.32	424	257	32.5	144	-
12	4	1C002	6860480	E 14 STD LR 90 WELD ELL WPB		0.202	0.410	0.014	0.008	0.200 0	0.131 0	0 100.	.041 0.	012 0	100	0.30	491	297	31.5	148	-
E	4	50658	6860657	E 16 STD LR 90 WELD ELL WPB		0.200	0.550	0.012	0.003	0.240 (0.050 0	0 010	.030 0.	060 0	100	0.31	471	281	31.5	155	1

Surface Appearance	Dimensional	RT N/A
H	Surface Appearance	Surface Appearance Dimensional

LEGEND:

TR: It is within the standard range <0.008% and include trace element. LA: Laddle Analysis WQ: Water Quenched PA: Product Analysis NDT: Non-destructive Test A: Annealing N: Normalized HB: Brinell Hardness HRC: Rockwell Hardness N/A: Not Applicable AC: Air Cooled WC: Water Cooled L: Longitudinal T: Transversal

OC: Oil Cooled G: Hot Dip Galvanized SR: Stress Relieving

	Heat Treatment: (HT)
1	Hot Forming Between 630 C and 980 C, AC
5)	Cold Forming: STRESS RELIEVED AT 600 °C X 120Minutes
3	Hot Forming Between 630 C and 980 C, AC: Normalized at 920 C X 60 Minutes
	· · · · · · · · · · · · · · · · · · ·

П

We hereby certify that the material described herein bas, been made in accordance with the requirements called for by the above order and is that which bash bigh tested to the satisfaction of the inspector.

Appendix B

Certified inspection certificate for the ordered AISI 2205 duplex stainless steel elbows.

V	INSP EN	ECTION 10204:	CERTIF	ICATE 3.1		Numbe Page: 1	r: 62452	3 Rev	:
S C H O E L L E R B L E C K M A N N EDELSTAHLROHR SEAMLESS-STAINLESS						Created Date: 1	d on: 12.03.201	5 Mod	lified on
NAHTLOS ZUM ERFOLG									
SCHOELLER-BLECKMANN Ed 2630 Ternitz, Rohrstrasse 1 Austria	leistahirohr GmbH			TL: 26303 FAX: 2630 E-MAIL: 1	16/601 0316/894 nicole.riegler(@sber.co.at			
CUSTOMER DESCRIPTION									
CLIENT SOLD TO		-		CLIENT	SHIP TO				
PINACLE STAINLESS STEEL ACIER INOXYDABLE 455 AMBASSADOR DRIVE MISSISSAUGA, ON LST 2J3 CANADA	INC.			PINACLE ACIER IN 455 AMB/ MISSISSA CANADA	STAINLESS OXYDABLE ASSADOR D AUGA, ON 1	STEEL ING B RIVE ST 2J3	2.	PINA Jan D	CLE
CLIENT ORDER: 79394									
SALING ORDER, 10420									
MATERIAL: SEAML. STAINL.	STEEL TUBES/P	IPES							
HEAT-TREATED, PICKLED,	PASSIVATED								
GRADE: \$31803,\$32205,									
STANDARD: ASME SECT.II	PART.A SA790/S	A790M-2013	ED.						
ASTM A790/A790M-14									
IMPACT TEST ISO-V AT -5	0 *C								
NACE MR0103-2012									
NACE MR0175/ISO 15156-3	:2009								
CORROSION TESTED ACC. T	O ASTM A262 PR	ACTICE E							
FERRITE CONTENT IN ACC.	WITH ASTM E56	2							
TOLERANCES: ASME SECT.I	I PART.A SA999	/SA999M-20	13 BD.						
ASTM 3999/8999M-14									
RANDOM LENGTHS 17/23 R	/L								
PLAIN ENDS,									
DIMENSIONS: 33,40 X 3,3	8 MM / 1 "NB X	SCH 40S							
COLD FINISHED									
Sales Item Client Item	Delivery No	Lot No.	Heat No	Pieces	Weight	Te	t Lgth	Un I	gth
30	8170051135	242634	4958	48	815 KG	1	324,93 M	5182	-7000 MN
RAW MATERIAL									
Heat Nra Supplier				Met	hod				
49581 ACERALAV	A (SPAIN)			Elect	tric furnace+/	AOD			
			Products			-	_		_
CHEMICAL COMPOSITION	%)	*L: Ladie C:	r rouucus						
CHEMICAL COMPOSITION (%) C Mn	*L: Ladie C: Si	P	8	NI	Cr	Me	N	Prez



SCHOELLER-BLECKMANN EDELSTAHLROHR GMBH

We hereby cartly that the making heard dearchad has been meanfactured, senged, sealed, and people in accordance with above entendence and specifications and satisfies conders measurements. This rithera is issued by a comparison particular platem and it is with utilized, original signature, it can be summing the contribute work measure as couply of 1, to must affect the control to make the maximum grant sponsibility for any universit or TUBACIEX, not allowed use. Any forance of the control to be co

S C H O E L L B L E C K M A I E D E L STAHLRO	E R N N OHR						Created on: Date: 12.03.2015	Modified or
NAHTLOS ZUM ER	FÓIĞ							
Let No	Sample	т	Rp0.2	Rm	A2"			
		°C	MPa	MPa	96			
Min		20	450	655	25			
Max		20						
242634	1	20	629	826	26			
HARDNESS TEST								
Let No	Sample	HRC						
Min								
Max		28						
242634	1	23						
	2	22						
IMPACT TEST	-		-	-	-			
Lot No	Sample	т	Wspec	Ecv	Туре	Le		
	1000	°C	101.005	J		205205		
Min		-50						
Max		-50						
242634	1	-50	2,5	39	L	0,90		
	2	-50	2,5	42	L	0,81		
			25	44	L	1.18		

HYDROSTATIC PRESSURE TESTED AT 2500 PSI DURING 5 SEC ON RACH TUBE/PIPE: SATISFACTORY

TECHNOLOGICAL TESTS FLATTENING TEST: SATISFACTORY

-

THE TUBES/PIPES CONFORM ALSO TO NACE STANDARD MR0175-2009, MR0103-2012

VISUAL INSPECTION AND CHECKING OF DIMENSIONS: SATISFACTORY

FOR MICROGRAPHIC EXAMINATION SEE ENCLOSED REPORT



Certified Management System acc. to ISO 9001, ISO 14001 and OHSAS 18001 by LRQA SCHOELLER-BLECKMANN EDELSTAHLROHR GMBH

We have young worthy that the marketer largers see schedule law have manufactured, samplink, toxing and investoria is assumed in a schedule market and approximations and an advances in the uniform is insulated by a computerisand system and it is water whole update and the schedule update of the certificate and character and system and it is water whole update and the schedule of the certificate and character and spaces that is advanted and. Any forgot of the land attaction of the certificate and langel procession.

Appendix C

Physical and chemical properties of the as-received silica sand particles

SILICA ABRASIVE

February, 2018 Page 1 of 3

PRODUCT

TARGET[®] Silica Abrasives are high quality, close graded silica sands which are produced at our Morinville, Alberta, plant in ten size grades. Typical gradations are presented in the following tables.

USES

TARGET Silica Abrasives are suitable for all applications requiring a high quality, clean, well graded sand. Typical uses include:

- Sandblasting of steel, concrete or wood in preparation for surface coating or repair
- Sandblasting of concrete or wood to obtain architectural effects
- Aggregate for cementitious or polymer mortars, grouts and concretes
- Foundry mold sand
- Traction sand

PHYSICAL PROPERTIES

Colour Grain Shape **Bulk Density** Hardness, Moh Specific Gravity Moisture Content Tan Sub-rounded 92 - 95 lb/ft3 (1474 - 1522 kg/m3) 6.5 - 7 2.65 <0.1 % weight

PACKAGING

TARGET Silica Abrasives are available in 22.7 kg (50 lb) multi-wall paper bags, 1680 kg (3700 lb) bulk bags, or as bulk deliveries in dump trucks or pneumatic tankers.



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SILICA ABRASIVE

TYPICAL CHEMICAL ANALYSIS

February, 2018 Page 3 of 3

Chemical Oxide	% weight	Typical Value
Silica	SiO ₂	93.2 - 93.6
Alumina	Al ₂ O ₃	3.6 - 4.6
Iron	Fe ₂ O ₃	0.30 - 0.35
Calcium	CaO	0.25 - 0.65
Magnesium	MgO	0.08 - 0.15
Sodium	Na ₂ O	0.75 - 0.85
Titanium	TiO ₂	0.1 maximum
Loss on Ignition		0.3 maximum

Т A R G E Т Ρ R 0 D U C T s L Т D

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Appendix D

Flow rate calibration.

Pump speed (Hz)	Time 1 (secs)	Time 2 (secs)	Time 3 (secs)	Average time (secs)	Flow rate (GPM)
10	46.56	46.58	46.59	46.58	5.15
20	23.82	23.54	23.96	23.77	10.10
30	15.62	15.42	15.87	15.64	15.35
40	11.9	12.05	12.06	12.00	19.99
50	9.38	9.47	9.5	9.45	25.40
60	7.43	7.69	7.71	7.61	31.54
70	6.42	6.5	6.62	6.51	36.85
80	5.45	5.32	5.6	5.46	43.98

Table D.1. Flow rate and the average time it takes a slurry to fill up the 4 gallons calibration tank at different pump speed.

The flow rate is then plotted against pump speed, and a linear line of best fit is used to determine the equation of a straight line (Eqn. C.1).



Figure D.1. A plot showing the variation of flow rate at different pump speed.
$$y = 0 \cdot 5471x - 1.0738$$

where x is the pump speed and y represent the volumetric flow rate at 2.5 m/s.

The volumetric flow rate of the slurry at 2.5 m/s is calculated using Eqn. 3.2.

$$F_r = 2.5 \times 3.1416 \times \left[\frac{0.0266446^2}{4}\right] = 1 \cdot 39 \times 10^{-3} \ m^3/s$$

Since 1 m^3s^{-1} equals 15,850.3 GPM, thus the volumetric flow rate of the slurry at 2.5 m/s is estimated to be 22.09 GPM. The corresponding pump speed at 2.5 m/s can then be calculated using the equation of a straight line (Eqn. C.1). Table C.2 shows the calculated pump speeds at different velocities used in this study.

$$x = \frac{1.0738 + 22.09}{0 \cdot 5471} = 42.4 \, Hz$$

Pump speed (Hz)	Velocity (m/s)	Volumetric flow rate (GPM)
10.1	0.5	4.43
18.1	1.0	8.85
26.2	1.5	13.28
34.3	2.0	17.70
42.4	2.5	22.13
50.5	3.0	26.55
58.6	3.5	30.98
66.7	4.0	35.40

Table D.2. The volumetric flow rates of the slurry through the 1-inch schedule 40 pipes used in this study.

Appendix E

Tests	Elbow 1	Elbow 2	Elbow 3	Elbow 4
1	13.72	10.89	16.91	15.05
2	14.82	12.26	19.41	14.63
3	13.42	11.78	19.45	14.47
Average	13.99	11.64	18.59	14.72
Std. dev.	0.74	0.70	1.45	0.30
Uncertainty	13.0%	14.8%	19.23%	5.0%

Repeatability test results.

Table E.1. 48-h erosion-corrosion reproducibility data for AISI 1018 steel elbows using 30 wt.% sand particles flowing at 2.5 m/s.

Table E.2. 120-h erosion-corrosion reproducibility data for AISI 1018 steel elbows using 30 wt.% sand particles flowing at 2.5 m/s.

Tests	Elbow 1	Elbow 2	Elbow 3	Elbow 4
1	16.66	11.00	17.93	12.54
2	16.40	11.52	18.73	11.95
3	17.01	10.97	17.51	12.47
Average	16.69	11.16	18.06	12.32
Std. dev.	0.31	0.31	0.62	0.32
Uncertainty	4.6%	6.9%	8.6%	6.6%

Table E.3. 48-h erosion-corrosion reproducibility data for AISI 2205 steel elbows using 30 wt.% sand particles flowing at 4 m/s.

Tests	Elbow 1	Elbow 3
1	50.16	67.76
2	49.95	64.14
3	51.72	65.94
Average	50.61	65.95
Std. dev.	0.97	1.81
Uncertainty	4.7%	6.8%

Appendix F

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Chapter 2, Figure 2.1



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Chapter 4



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Chapter 5

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Chapter 6

	Full factorial, microscopic and spectroscopic study of erosion-corrosion
2	Author:
	Raheem Elemuren, Richard Evitts, Ikechukwuka N.A. Oguocha, Glyn Kennell, Regan
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