Heat Transfer in Brine Solutions at Supercritical Pressure

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Master of Science

Thomas G. Johnson
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This thesis titled
Heat Transfer in Brine Solutions at Supercritical Pressure

by
THOMAS G. JOHNSON

has been approved for
the Department of Mechanical Engineering
and Russ College of Engineering and Technology by

Jason P. Trembly
Assistant Professor of Mechanical Engineering

Dennis Irwin
Dean, Russ College of Engineering and Technology
ABSTRACT

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Heat Transfer in Brine Solutions at Supercritical Pressure

Director of Thesis: Jason P. Trembly

Heat transfer and differential pressure in a heat exchanger system at supercritical pressure were investigated. A single dimensional heat transfer model was developed to predict heat transfer rates and temperature profiles in a heat exchanger flowing water near the critical point. A series to trials were performed using test fluids representing produced water over a pH range of 4.0 to 9.0. The temperature profile and differential pressure were measured as the test fluids were heated from 150°C to 350°C at 3450 psi (23.8 Mpa) while flowing through the tube side of an experimental concentric tube counterflow heat exchanger. The flow conditions were in the transition region with Reynolds numbers ranging from 4,000-6,000. The test fluid was heated by supercritical de-ionized water flowing through the shell section of the heat exchanger. Seven trials were performed with a duration of 7.5 hours each reporting an average heat flux of 34.22 ± .64 kW/m². Results showed little to no decrease in heat flux due to fouling. No measureable increase in differential pressure across the heat exchanger was observed during any of the trials.
ACKNOWLEDGEMENTS

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CHAPTER 1: INTRODUCTION

1.1 Introduction

The United States is in the midst of a boom in shale energy exploration. The amount of proved domestic reserves has increased dramatically in the last decade. Much of this increase in accessible supply is due to advancements in drilling and hydraulic fracturing technology. While hydraulic fracturing is a key component in extracting these energy reserves, there is widespread public concern about the heavy water usage and potential water pollution caused by the process. A single well may require up to 8.7 million gallons of fresh water [1]. Technology is being developed in order to address these water usage and quality issues. In particular, Ohio University is developing an innovative method for processing hydraulic fracturing wastewater using supercritical water technology. The purpose of this research is to evaluate the heat transfer and fouling properties of hydraulic fracturing wastewater at supercritical pressure.

1.2 Shale Energy Reserves

Much of the new accessible domestic energy resources are contained in the form of shale gas and oil. Shale is a formation of dense rock that appears in thin horizontal seams far below the ground. Large formations of resource-containing shale plays vary widely in their local geology as well as geographic location. Figure 1 is a recent map showing all of the major shale plays in the United States.
Even more significant than the widespread nature of these deposits is the quantity of available reserves that they contain. The massive influx of available resources that new shale exploration has brought is best described as shown in Figure 2. The figure shows that throughout the 1980’s and 90’s, the proven reserves of natural gas was fairly constant. In addition, there was a clear trend of increasing U.S. gas imports. Starting in the early 2000’s, there was a massive jump in proved natural gas reserves with the new shale exploration. It is also visible that after this jump, domestic production began to rise steadily, while imports began to decline. This illustrates the point that energy from shale will be an incredibly important resource for the U.S. and its domestic energy security.
Figure 2: U.S. Natural Gas Imports, Production and Proved Reserves [3]

1.3 Hydraulic Fracturing and Water Usage

The driving force behind the explosion in shale energy availability in the United States is the mass adoption of horizontal drilling and hydraulic fracturing technologies. Horizontal well drilling, a technology seeing widespread application, allows for greater access to the relatively thin resource-containing shale layers by creating a borehole which travels horizontally. The Marcellus shale play ranges in depth from 4,500-8,000 feet below the surface, but the average thickness is only 100 ft. The use of precise directional well drilling allows the access to more than 1 mile of horizontal distance through the
shale layer with a single hole [4]. Without this technology, extraction of resources from thin shale deposits would be far less economical.

The second key piece of technology driving the emergence of shale energy is hydraulic fracturing. This is the process of fracturing the shale surrounding the well bore in order to gain access to gas deposits. During the fracturing process, a mixture of water, sand and a blend of chemicals are injected at high pressure. After fracturing, natural gas along with fluid returns to the surface. The water which returns within days of completing the fracturing process is termed as flowback water, while water returned during the well production period is termed produced water. Produced water differs in that it often contains a much high concentration of contaminants and dissolved solids than flowback water. The flowback/produced (F/P) water returned to the surface must be managed so it does not impact the surrounding environment. Figure 3 illustrates a shale gas well incorporating directional drilling and hydraulic fracturing. In addition, the key components of the hydraulic fracturing water cycle are labeled.
The heavy use of water for shale gas operations is a technical issue that has become a cause for concern. A single well operating in the Marcellus shale region may require 1.8-8.7 million gallons of water throughout its lifespan [1]. This not only requires a significant amount of water from local supplies, it also produces wastewater in large volumes. Annual F/P water volume generation of 15-20 billion barrels is estimated for the U.S. alone [6]. To put some of these figures into context, it is estimated that the
consumption of freshwater in the United States for energy production will increase 70% from its 2005 figure by 2030 [7].

The F/P water produced as part of shale operations includes a number of contaminants which make it difficult to treat. The quality and constituents within F/P water makes cleaning and recycling a very cost prohibitive proposition. As mentioned in Section 1.1, F/P water contains a number of different constituents. These constituents include suspended solids, dissolved solids, organics, and naturally occurring radioactive material (NORM) [8]. Some dissolved solid constituents and their concentrations are provided in Table 1. This data focuses mainly on fluids sampled from Marcellus Shale operations in Pennsylvania.

Table 1: Composition of F/P Water [8]

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Concentration (mg/L)</th>
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<tr>
<td>TDS</td>
<td>680-345,000</td>
</tr>
<tr>
<td>Na⁺</td>
<td>70-117,000</td>
</tr>
<tr>
<td>Ca²⁺</td>
<td>40-41,000</td>
</tr>
<tr>
<td>Ba²⁺</td>
<td>0-13,800</td>
</tr>
<tr>
<td>Mg²⁺</td>
<td>17-2,550</td>
</tr>
<tr>
<td>Sr²⁺</td>
<td>0-8,460</td>
</tr>
<tr>
<td>Cl⁻</td>
<td>64-196,000</td>
</tr>
<tr>
<td>SO₄²⁻</td>
<td>0-763</td>
</tr>
</tbody>
</table>

1.5 The IPSC Process

Ohio University is currently developing a process for efficient, cost effective treatment of produced water from unconventional shale gas wells. The integrated precipitative supercritical (IPSC) process takes advantage of several existing technologies
for water processing, as well as a supercritical reactor system (SCR) to remove dissolved solids and residual hydrocarbons. An overview of the IPSC process is shown in Figure 4. The individual processes contained within the overall IPSC process include hydrocyclone separation, UV treatment, chemical precipitation, NORM sorption, and the supercritical reactor solid removal system.

Figure 4: IPSC Process Overview

The SCR is projected to provide more effective removal of constituents from the F/P water versus conventional distillation or membrane separation methods. However, due to the high temperatures involved in supercritical processing, extensive thermal inputs are required. An efficient and well-designed reheat and thermal management system is vital to cost effective operation of the IPSC process. Initial economic modeling shows that one of the most significant cost streams for the IPSC process is the energy input, most of which is used to heat the process fluids and the SCR itself. Reducing the
thermal input required by optimizing the design of thermal management systems is an important factor in the system design.

1.6 Heat Exchanger Performance and Operation

Nearly every industrial process requires thermal management systems. The key components in any thermal-fluid system are heat exchangers, as they allow for the efficient transfer of energy between two physically separated fluids. In most cases, this may involve removing heat from a fluid in order to properly regulate temperature within a system. It is important that heat exchanger systems are designed and sized correctly for each application.

There are two modes of heat transfer at play within a heat exchanger, conduction and convection. Since the two fluid streams are, by definition, physically separated, there will be conduction through the tube or other separation medium. Heat transfer by conduction in this type of situation is fairly straightforward and easily quantified. The other mode of heat transfer is convection within the fluid streams. Quantifying heat transfer by convection is much more complex. The heat transfer rates will depend on the flow arrangement, fluid properties, and the fluid mechanics at play [9].

There are a number of environmental and operational factors that will affect the heat flux of a heat exchanger. Foremost among them is fouling. Most fluid systems will not have perfectly clean fluids; deposition and other contamination from the fluids can affect the heat exchanger surfaces and cause reductions in performance. The main
mechanisms of heat exchanger fouling are driven by particulates, reactions, corrosion, precipitation, biological activity, and solidification [10].

Fouling in heat exchanger tubes causes a number of negative effects on performance. The buildup of material on the tube walls increases thermal resistance and lower the heat transfer rate within the heat exchanger. In addition, the reduction in cross sectional area impedes fluid flow, causing pressure buildup or reducing flow capacity. Figure 5 illustrates the physical effects of fouling on a tube wall.

![Clean Fouled](image)

**Figure 5: Fouling in Heat Exchanger Tubing**

There are significant problems and costs associated with fouling in heat exchangers. It is estimated that additional costs associated with heat exchanger fouling result in an annual losses of $4.2-10 billion in the U.S. alone. [11] There are a number of direct and indirect costs that may be seen as a product of fouling including:

- Increased capital expenditures due to oversizing
- Energy loses associated with poor performance
- Treatment cost to lessen corrosion and fouling
• Loss in production due to maintenance
• Reduction in service life [12]

Proper sizing and design for each application is a vital component in minimizing costs due to heat exchanger system fouling. However, designing heat exchangers to account for future fouling in most applications is not a precise process. There are generally too many variables or conditions to precisely calculate future fouling effects. In many cases, heat exchanger systems are simply overdesigned by a factor of 20-25% to account for future fouling and reduction in efficiency [12].

Although fouling design factors are difficult to calculate, lab tests under similar controlled conditions can improve predictions of operational conditions. The standard procedure for evaluating the performance of a heat exchanger under fouling is to run at steady state conditions and observe changes in outlet conditions. These tests involve measuring outlet temperatures as well as differential pressure across devices. [13] Data from such tests can be used to aid in the design of a larger scale system.

1.7 Research Objectives

The overall goal of this thesis is to investigate heat transfer and hydraulic resistance in a flowing heat exchanger system with a brine solution and clean water at supercritical pressure. To achieve this goal three research objectives are proposed and described in the following sections.

The first objective of this thesis is to develop a basic one-dimensional heat transfer model to simulate the near supercritical operating conditions of the heat recovery
heat exchanger. Deliverables for this objective include theoretical heat transfer rates and fluid temperature profiles for the baseline operating condition. In addition, the heat transfer model results will be compared to the experimental data.

The second objective of this thesis is to quantify the average heat flux of the experimental heat exchanger as a function of time. To meet this objective, a set of experimental trials will be completed which vary the composition and pH of the simulated F/P test fluid. During these trials the inlet and outlet temperatures of the fluid streams will be measured and used to calculate the average heat flux in the heat exchanger. This data will then be presented graphically, as a function of time.

The third and final objective of this thesis is to quantify the change in differential pressure across the heat exchanger as a function of time. To meet this objective, differential pressure will be measured during the same set of experimental used trials to fulfill Objective 2. This data will then be presented graphically as pressure over time.
CHAPTER 2: LITERATURE REVIEW

2.1 Introduction

This research leverages several subjects in the field of thermal science and engineering including supercritical fluid properties and heat transfer at supercritical pressure, as well as solvent properties and deposition in fluids at supercritical pressure. An understanding of current research in these fields, in addition to the fundamental concepts, is helpful in understanding the presented results. This chapter provides a brief overview of the most important concepts and related research to the subject at hand.

2.2 Supercritical Water Thermophysical Properties

Supercritical fluids, including water, are useful in industrial processes because of the predictable shift in their properties near their critical temperature and pressure. A number of mainstream commercial processes use supercritical fluids, including:

- Supercritical CO$_2$ as a refrigerant in vapor compression air conditioning and cooling systems [14]
- Supercritical water as an agent for Supercritical Water Oxidation (SCWO) reforming of military and industrial wastes [14]
- Supercritical steam driven turbines for electricity generation [15]

The usefulness of supercritical fluids is derived from the fact that their thermophysical properties change significantly and predictably with changes in temperature and pressure. Therefore, the fluids can be ‘engineered’ and adapted to various applications [16]. Figure
Illustrates the drastic changes in volume that occur in pure water as the temperature is increased at a constant supercritical pressure.

Figure 6: Volume Changes in Pure Water at 23.8 MPa

In addition to shifts in physical properties, like density, the thermal properties of water vary significantly at supercritical pressure. Figure 7 shows the dramatic change in specific heat of pure water as it nears the pseudo critical point. The application of supercritical steam in fossil-fuel fired power plants allows for a significant increase in overall thermal efficiency [15].
2.3 Heat Transfer at Supercritical Pressure

Nearly every heat transfer scenario involves a system bordered by a fluid. This is true of an object in still air, moving air, or a liquid medium. Conductive heat transfer within a solid object is well understood and can be accurately described and quantified. Conduction in cylindrical coordinates can be derived and generally described by

$$\rho c_p \frac{\partial T}{\partial r} = k \left( \frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} + \frac{1}{r^2} \frac{\partial^2 T}{\partial \theta^2} + \frac{\partial^2 T}{\partial z^2} \right) + q^*$$

Equation 1 [17]

where,
\( \rho \) = density, (m^{3}/kg)
\( c_p \) = specific heat, (kJ/kg*K)
\( \frac{\partial t}{\partial \tau} \) = time derivative of temperature, (K/S)
\( K \) = constant
\( r \) = radial distance, (m)
\( z \) = vertical longitudinal distance, (m)
\( \theta \) = angular distance, (rad)
\( q^* \) = internal heat generation, (kW)

All single cases of conductive heat transfer within a known system of cylindrical coordinates can be reduced to this equation.

Convective heat transfer describes heat transfer between a solid object and a bordering fluid. However, unlike conduction, convective heat transfer cannot be easily described and quantified with a general formula. The primary component within convective heat transfer is the fluid boundary layer condition determined by the fluid mechanics at each specific location. As a result, there are numerous variables which affect the flow behavior at each location. In order to estimate convective heat transfer rates, a convective heat transfer coefficient, \( h \left( \frac{W}{m^{2} \cdot k} \right) \) is used. This coefficient accounts for the fluid forces which affect heat transfer in the boundary region without the need for otherwise incredibly complex computations. The primary function of convective heat transfer calculations is to estimate the \( h \) value, with which heat transfer can be estimated using convectional equations.

The convective heat transfer coefficient, \( h \), is determined both from the fluid properties as well as the fluid flow scenario. In general, it is determined from the Nusselt number using Equation 2-2 [17].
\[ \text{Nu}_L = \frac{h \cdot L}{k} \]  

Equation 2

where,

\( \text{Nu} \) = Nusselt number
\( h \) = convective heat transfer coefficient, (W/m\(^2\)K)
\( L \) = characteristic length, (m)
\( k \) = thermal conductivity, (W/m·K)

The Nusselt number represents the dimensionless ratio between convective and conductive heat transfer. It is determined using accepted correlations based on the heat transfer scenario. For forced convection with interior flow and primarily turbulent flow, such as in a heat exchanger, one of the oldest and most general correlations is one developed by Dittus and Boelter (1930). It is shown in Equation 2-3 [18].

\[ \text{Nu}_b = 0.023 \cdot \text{Re}_b^8 \cdot \text{Pr}_b^{0.4} \]  

Equation 3

where,

\( \text{Re} \) = Reynolds number, (dimensionless)
\( \text{Pr} \) = Prandtl number (dimensionless)

Dittus and Boelter is widely accepted as the standard correction under single phase compressed liquid flow, however its effectiveness deteriorates rapidly as fluid properties shift under supercritical pressure conditions [19]. In the 1950s, the use of supercritical steam in fossil fuel-based power plants necessitated the need for more accurate analytical methods for quantifying heat transfer in this region. Bishop (1964) conducted experimental work to produce the correlation shown in Equation 2-4 [20].
While Bishop’s correlation was shown to be a vast improvement over that of Dittus and Boelter in the transition region, it was far from a complete representation of the phenomena. Numerous other studies were completed in the following decades, testing heat transfer rates at supercritical pressure with various heat and mass fluxes and different flow regimes. The application of supercritical water for cooling nuclear reactors spurred additional interest in improving the correlation and work has continued through the present day. One of the best modern results was published by Mokrey et. al. [21] [22]. Using modern equipment and experimental methods, they researched heat transfer correlations in near-supercritical water flowing in bare vertical tubes. The result is shown in Equation 5 [21].

\[
\text{Nu}_b = 0.0061 \times \text{Re}_b^{0.904} \times \text{Pr}_b^{-0.684} \times \left(\frac{\rho_w}{\rho_b}\right)^{564}
\]

Equation 5

Mokrey demonstrated that the correlation is not only a significant improvement from all earlier efforts, it has been shown to be applicable under a wide range of heat fluxes in the sub-critical, transition, and supercritical region.

2.4 Solvent Properties of Supercritical Water

Wider potential application of supercritical water systems in industrial applications, such as supercritical water oxidation reactor systems has brought about a
need for a better understanding of the behavior of salt solutions near and above the critical point. Armellini and Tester (1991) conducted a series of experiments describing the shift in phase transition temperature of solutions of sodium chloride. These experiments were conducted under isobaric conditions at 250 bar (3625 psi) and transition behavior was observed visually through a sapphire glass window. A distinct increase in nucleation temperature was observed from 1.0 wt% up to 20.0 wt%, raising to a maximum temperature of 397 °C [23]. The critical point of pure water, where phase transition occurs, is accepted to be 374°C at 22.06 MPa. Armellini and Tester demonstrated how this critical temperature shifts for a solution of dissolved sodium chloride. This research was later continued, investigating both sodium chloride further, as well as sodium sulfate [24].

Leusbrock, Metz, Rexwinkel, and Versteeg (2008) took a slightly different approach in developing a description of the solubility of inorganics at different temperatures and pressures around the critical point [25]. Their experiments measured the remaining concentration of sodium chloride in solution after being heated near the critical point. Their data at a number of temperatures and pressures allows for a comparison of previous empirical models. This publication was soon followed up by addition experimental work by Leusbrock, Metz, Rexwinkel, and Versteeg (2009), investigating the solubility of magnesium chloride and calcium chloride under a similar set of near-critical and supercritical conditions [26]. A similar experimental methodology and setup was used.
The research discussed so far in this section included general descriptions of the solubility of salts in supercritical water. There has been additional research focusing more on specific industrial applications involving supercritical fluids. One of these applications is supercritical water oxidation reactors. Dong et. al. (2010) investigated the negative effects of salt deposition in supercritical water oxidation reactor systems. The research developed a novel reactor design which allows solid salt nucleation while preventing deposition and corrosion to reactor components [27]. Additional research on supercritical water oxidation systems, focusing specifically on the deposition of Na$_2$CO$_3$ has also been conducted [28].
CHAPTER 3: PLAN OF WORK

3.1 Introduction

The research work was completed in three phases. Phase one involved the creation of a single dimensional heat transfer model to predict temperatures and heat transfer rates in the planned experimental system. The results of the heat transfer model were later compared against operational data from the physical experimental heat exchanger test system. In phase two, the results of the heat transfer model were used to finalize designs for the experimental system. The system was then constructed and tested with clean water to establish baseline operating characteristics. In phase three, a series of trials were performed with the heat exchanger test system using several compositions of simulated flowback and produced fluids.

3.2 Test Plan

A series of planned experiments were designed to test the effects of fluid composition and pH on heat transfer and hydraulic resistance at supercritical pressure. The tests were conducted using an experimental counterflow concentric tube heat exchanger test system. A simplified diagram of the experimental heat exchanger is shown in Figure 8.
The diagram illustrates the flow scheme of the two fluid streams as well as the location of the 9 integrated thermocouples. The brine test fluid was designed to flow in the inner (tube) side of the heat exchanger while the outer (shell) side of the heat exchanger flowed clean, de-ionized water. The inlet temperature and pressure of each fluid remained constant throughout all of the trials. Outlet temperatures and test fluid differential pressure were recorded during the trials. Process variables evaluated were the composition and pH of the test fluid. Table 2 shows the evaluated test fluid compositions.

<table>
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<th>Fluid</th>
<th>pH</th>
<th>Na(^+) (mg/L)</th>
<th>Ca(^{2+}) (mg/L)</th>
<th>Ba(^{2+}) (mg/L)</th>
<th>Mg(^{2+}) (mg/L)</th>
<th>Sr(^{2+}) (mg/L)</th>
<th>Cl(^-) (mg/L)</th>
<th>HCO(_3)(^-) (mg/L)</th>
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<td>7</td>
</tr>
</tbody>
</table>

Due to the difficulty of producing large volumes of test fluid of a precise composition, concentrations within 20% of those shown in Table 2 were chosen to be...
acceptable. A total of 9 trials were planned with 3 trials conducted for each fluid composition in order to demonstrate experimental repeatability. The initial test plan is shown in Table 3.

Table 3: Initial Test Plan

<table>
<thead>
<tr>
<th>Trial</th>
<th>Test Fluid Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>B</td>
</tr>
<tr>
<td>2</td>
<td>B</td>
</tr>
<tr>
<td>3</td>
<td>B</td>
</tr>
<tr>
<td>4</td>
<td>A</td>
</tr>
<tr>
<td>5</td>
<td>A</td>
</tr>
<tr>
<td>6</td>
<td>A</td>
</tr>
<tr>
<td>7</td>
<td>C</td>
</tr>
<tr>
<td>8</td>
<td>C</td>
</tr>
<tr>
<td>9</td>
<td>C</td>
</tr>
</tbody>
</table>

The test fluids used in the trials were designed to simulate the composition of dissolved solids expected to be present in produced water at the supercritical side of the heat exchanger. The key parameter that was varied between the trials was fluid pH. Initial modeling of the IPSC process showed that the pH and composition of the effluent to the supercritical portion of the process is highly dependent on the operating parameters of the upstream processes, such as softening and sulfation. The proposed set of trials test the potential effects that changes in fluid pH have on heat transfer and heat exchanger fouling in the immediate subcritical temperature range.

Test fluid was introduced to the system at approximately 200 °C and heated up to 350 °C; the critical point at test pressure is approximately 375 °C. Through this temperature range, the test fluids were expected to experience distinct shifts in their
thermophysical properties. These properties include density, specific heat, and dynamic viscosity. All trials will be conducted at 3,450 psi (23.8 MPa). Each trial was initially planned to be run for 96 hours continuously. Due to the potential for fouling the inner tube of the test heat exchanger was replaced between each trial.
CHAPTER 4: EXPERIMENTAL METHODS

4.1 Heat Transfer Model

To fulfill Objective 1, a simple single dimensional steady-state heat transfer model representing the experimental heat exchanger system was developed. Theoretical heat transfer calculations for a consistent and predictable geometry, such as a concentric tube heat exchanger, are well established. However, the near critical operating conditions of the fluids being tested complicated calculations. Since the fluids are operating near the critical point, several of their properties, including specific heat and specific volume, do not remain constant as is normally assumed. As part of the model, these values were approximated from tables of established values, based on the fluid temperature and pressure at each nodal point. This required the use of a numerical approximation of fluid properties, rather than the use of average values, to calculate the overall heat transfer values.

Figure 9 shows the cross section of the experimental concentric tube heat exchanger. Both fluid streams are shown; the cold, tube side test fluid is shown in the in the annulus between \( r_3 \) and \( r_2 \) and the hot shell side fluid is shown in the center. In addition, the radii of each tube section is labeled. Heat transfer to the fluid is denoted as \( Q \) and heat loss to the surroundings is denoted as \( Q_{\text{loss}} \), heat transfer to the test fluid as well as heat loss from the system.
Table 4 lists the specific dimensions of radii within the heat exchanger cross section. These specific dimensions were chosen based on the availability of tubing, fittings, and related components. The experimental heat exchanger was built using components of these dimensions, so these dimensions are constant throughout the heat transfer modeling and design.
Table 4: Test Heat Exchanger Cross Section Dimensions

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>r1</td>
<td>0.245 in (0.62 cm)</td>
</tr>
<tr>
<td>r2</td>
<td>0.375 in (0.95 cm)</td>
</tr>
<tr>
<td>r3</td>
<td>0.53 in (1.35 cm)</td>
</tr>
<tr>
<td>r4</td>
<td>0.75 in (1.91 cm)</td>
</tr>
</tbody>
</table>

Formulae describing heat transfer phenomena within the system were derived using the dimensions shown in Equation 6, as well as the fluid thermophysical properties. The specific heat transfer rate \( \left( \frac{\text{KW}}{\text{m}} \right) \), for the system is expressed by Equation 6.

\[
\frac{\dot{Q}}{L} = \frac{2\pi(T_2 - T_1)}{1 + \frac{R_{fi}}{r_1 h_a} + \frac{\ln\left(\frac{r_2}{r_1}\right)}{k_{12}} + \frac{1}{r_2 h_b}}
\]

Equation 6

where,

\( \dot{Q} \) = Heat transfer rate, (kW)
\( L \) = Linear dimension, (m)
\( r_x \) = Tube radius, (m)
\( T_x \) = Fluid temperature, (K)
\( R_{fi} \) = Surface fouling factor, \( \left( \frac{\text{mK}}{\text{W}} \right) \)
\( h_a \) = Convective heat transfer coefficient, inner stream, \( \left( \frac{\text{W}}{\text{m}^2\text{K}} \right) \)
\( h_b \) = Convective heat transfer coefficient, outer stream, \( \left( \frac{\text{W}}{\text{m}^2\text{K}} \right) \)
\( k_{12} \) = Thermal conductivity of dividing tube wall, \( \left( \frac{\text{W}}{\text{mK}} \right) \)
In addition, Equation 7 expresses the heat loss per unit length from the system \( \left( \frac{K_W}{m} \right) \).

These two formulas were used to calculate the temperature change of each of the fluid streams over a fixed length.

\[
\frac{Q_{\text{loss}}}{L} = \frac{2\pi(T_2 - T_3)}{\frac{1}{r_3 h_b} \ln \left( \frac{r_4}{r_3} \right) + \frac{1}{r_4 h_c}}
\]

Equation 7

Equations 6 and 7 are relatively simple and well-established equations governing heat transfer in concentric tubing system. The most significant term in these equations is the convective heat transfer coefficient; this is especially true for a system at supercritical pressure.

Since the thermophysical properties of the fluids vary significantly over the temperature range within the heat exchanger, Equation 6 and Equation 7 are not accurate for the entire length. However, they can be used to approximate heat transfer for a very small \( L \), in which the properties of the fluids can be assumed to be constant. Since the system pressure is constant, the fluid properties can be calculated at each nodal point as a function of temperature. By dividing the entire length of the heat transfer surface into \( n \) number of increments, the total temperature profile for the heat exchanger can be approximated by numerical methods. Each increment was calculated with length \( \frac{L}{n} \). The inlet temperatures were known from the experimental parameters and could be applied to Equations 6 and 7. From these results, the conditions at increment \( n+1 \) were calculated using Equations 8 and 9. This process was repeated for all \( n \) increments of the length \( L \), to calculate the complete temperature profile.
\[ T_{c,n+1} = T_{c,n} + \left( \frac{\dot{Q}L}{c_p \dot{m}} \right) \]  \hspace{1cm} \text{Equation 8}

where,

- \( C_p = \text{Specific Heat, } \left( \frac{KJ}{Kg \ K} \right) \)
- \( \dot{m} = \text{Mass flow rate, } \left( \frac{Kg}{S} \right) \)
- \( T_{c,n} = \text{Temperature of test fluid at point } n, \text{ (K)} \)
- \( T_{c,n+1} = \text{Temperature of test fluid at point } n+1, \text{ (K)} \)

\[ T_{h,n+1} = T_{h,n} - \left( \frac{(\dot{Q}_{\text{loss}}+\dot{Q})L}{c_p \dot{m}} \right) \]  \hspace{1cm} \text{Equation 9}

where,

- \( T_{h,n} = \text{Temperature of DI water at point } n, \text{ (K)} \)
- \( T_{h,n+1} = \text{Temperature of DI water at point } n+1, \text{ (K)} \)

The raw results of the heat transfer model were two coupled series of fluid temperatures and properties representing the profile of the experimental heat exchanger, calculated at a discrete amount of points along the length. Figure 10 shows a simplified version of the heat transfer model results profile. The diagram shows a ¼ cross section of the tube-in-tube heat exchanger. Each dot theoretically represents a point where the fluid properties and temperature were calculated. At a number of points along the length, the calculation was made for both the shell and tube fluid. When combined, this data set produced the complete temperature profile of the test heat exchanger.
4.2 Equipment and Methods

To complete the experimental component of the research, an experimental heat exchanger was designed, fabricated, and operated. The data collected from operating the experimental heat exchanger was compared to the results of the heat transfer model to fulfill the Objective 1.

The main component of the experimental setup is the concentric tube heat exchanger, as shown in Figure 11. The experimental apparatus was constructed from two concentric lengths of seamless 316 stainless steel tubing. The inner tube has an OD of 0.375-in (0.953 cm), while the outer tube has an OD of 0.75-in (1.9 cm). Two separate streams of heated fluid were counter currently pumped through the heat exchanger. The inner tube fluid was composed of the test fluid brine solution, while the annulus or shell section contained the heated DI water representing the water product from the supercritical reactor. Heat was recovered from this hot, shell side fluid and used to preheat the cold, tube side fluid. The flow rates of both fluids were held constant at 97.5 ml/min.
The experimental heat exchanger was designed to incorporate thermocouples to continuously measure and record the bulk fluid temperatures of each of the two fluid streams. The device includes seven independent temperature sensors in the shell-side fluid stream. These sensors allow for measurement of the bulk fluid temperature at fixed intervals along the heat transfer length. For each stream, temperature was measured at the inlet and outlet, with 5 evenly distributed points measured along the length of the shell-side. This setup allows for the real-time measurement of the fluid temperature throughout a trial and direct determination of heat flux. The tube-side temperatures are measured using two thermocouples located at the inlet and outlet and the shell side. Additionally, pressure drop across the tube side fluid is monitored using a precise differential pressure transducer. This allows for precise measurement of fluid pressure drop across the heat exchanger.

To precisely control the inlet conditions to the heat exchanger, as defined by the experimental parameters, auxiliary equipment was required. Figure 12 shows the complete experimental heat exchanger test system setup.
There are separate flow loops for both the shell and tube side streams of the heat exchanger. Each of these loops includes a pump, preheater, condenser, and back pressure regulator. This allows for the high temperature and pressure conditions defined by the test plan to be met. In addition, through the use of controllable preheaters, the inlet temperature of each fluid can be controlled to meet the requirements of the experimental
parameters. The system piping and instrumentation diagram (P&ID) is shown in Figure 13.

![Diagram](image)

**Figure 13: Experimental Setup P&ID**

Data acquisition from sensors and control of temperatures and pressures is accomplished using a central embedded controller, a National Instruments cRIO-9074. This controller uses closed-loop control logic to adjust preheaters in order to accurately maintain the determined experimental parameters. In addition, data from all sensors is
continuously acquired and logged. The system features built in thermocouple signal conditioning to ensure accurate readings. All control and functionality is automated by the embedded control system. A graphical user interface allows operating parameters to be set and current conditions to be viewed by an operator. However, once the operating parameters have been set, the system requires no direct interaction from an operator to maintain trial conditions. This setup allows for reliable data acquisition and control of the unattended experiment throughout the extended trial lengths.

The composition of all test fluids was verified using inductively coupled plasma atomic emission spectroscopy (ICP). Liquid samples were taken of the continuously mixed test fluids and diluted by a factor of 1000 for analysis. A Fischer icap 6000 ICP - AES was used for the analysis of test fluid ions. The ions of determination were Na\(^+\), Ca\(^{2+}\), Ba\(^{2+}\), Mg\(^{2+}\), and Sr\(^{2+}\). A measured test fluid composition within 20% of the target values was chosen to be acceptable for the trials.

4.3 Experimental Data Analysis

The data collected from each experimental trial is represented as a table of inlet and outlet temperature values from the heat exchanger, recorded at a number of time intervals. These values were used to calculate the net average heat flux of the heat exchanger. Figure 14 shows the labeling used for inlet and outlet temperature conditions.
In a counterflow heat exchanger, there is a theoretical maximum amount of energy transfer that can take place, based on the inlet temperatures. In the ideal scenario, with a heat exchanger of infinite length and no exterior heat loss, the outlet temperatures of each of the fluids would approach the inlet temperature of the other fluid. Since the system is at a constant pressure, the enthalpies of the fluids are based on their temperatures and can be calculated at any point, as shown in Equation 10. Enthalpies of the fluids at each point were calculated from tables of accepted values in EES.

\[ H_{x,x} = enthalpy(T_{x,x}) \]  

Equation 10

Enthalpy \( \left( \frac{kJ}{kg} \right) \) is used in the calculations as it represents the specific energy contained in the fluid stream at each condition. Temperature does not accurately reflect specific energy of the fluid in this system as the heat capacity of the fluid \( (c_p) \) isn’t constant near supercritical points.

The actual heat transfer within the heat exchanger can be calculated from the measured experimental values. \( T_{c,i}, T_{c,o}, T_{h,i}, \) and \( T_{h,o} \) were the raw values recorded.
throughout each trial. Equations 11 and 12 show determination of the total energy
transfer from the shell side fluid and net energy transfer to the tube side fluid,
respectively.

\[ \Delta H_{\text{total}} = H_{h,o} - H_{h,i} \]  \hspace{1cm} \text{Equation 11}

\[ \Delta H_{\text{net}} = H_{c,o} - H_{c,i} \]  \hspace{1cm} \text{Equation 12}

where,

\[ \Delta H_{\text{total}} = \text{Total system enthalpy change,} \left( \frac{\text{kJ}}{\text{kg}} \right) \]
\[ \Delta H_{\text{net}} = \text{enthalpy change of test fluid,} \left( \frac{\text{kJ}}{\text{kg}} \right) \]

The total change in enthalpy of the system, including heat loss is represented by \( \Delta H_{\text{total}} \).
The net enthalpy change, representing net heat transfer from the shell to tube side, is
\( \Delta H_{\text{net}} \).

The average heat flux in the heat exchanger is described as the energy transfer per
time, over the net heat transfer surface area \( \left( \frac{\text{Kw}}{\text{m}^2} \right) \). This can be calculated from the
calculated net enthalpy change and the known dimensions of the system. This is shown
in Equation 13.

\[ \dot{Q} = \frac{\Delta H_{\text{net}} \cdot \dot{m}}{L \cdot \pi \cdot 2 \cdot r_1} \]  \hspace{1cm} \text{Equation 13}

where,

\[ \dot{Q} = \text{Average heat flux,} \left( \frac{\text{Kw}}{\text{m}^2} \right) \]
\[ L = \text{Heat exchanger length,} \ (\text{m}) \]
\[ r_1 = \text{Tube interior radius} \]
In the case of the experiments conducted, $\Delta H_{\text{net}}$ could not be determined directly. While the inlet and outlet temperatures of the test fluid were measured, the enthalpies could be calculated using EES because they are not accurately known due to the high composition of dissolved solids. $\Delta H_{\text{net}}$ was instead calculated from $\Delta H_{\text{total}}$ by applying an efficiency correction factor to account for $Q_{\text{loss}}$, or heat lost from the system. The efficiency factor ($N$) for each set of trial data was calculated using the baseline operating temperatures determined for each trial. At the start of every trial, the system was heated while clean water is circulated in both the test fluid and DI water loops. The test fluid was not introduced until the operating temperatures were stable. At these stable operating temperatures, $\Delta H_{\text{net}}$ could be calculated directly because both of the fluids are clean water with known enthalpy properties. The system efficiency was then represented as the ratio of $\Delta H_{\text{net}}$ to $\Delta H_{\text{total}}$. This is shown in Equation 14.

$$N = \frac{\Delta H_{\text{net}}}{\Delta H_{\text{total}}}$$  \hspace{1cm} \text{Equation 14}

Once the system efficiency was determined for the trial, $\Delta H_{\text{net}}$ could be estimated for each temperature data point during the trial while the test fluid was circulating. The efficiency factor was applied to each calculated $\Delta H_{\text{total}}$ value, as in Equation 15.

$$\Delta H_{\text{net}} = N \ast \Delta H_{\text{total}}$$  \hspace{1cm} \text{Equation 15}
Once the entire set of $\Delta H_{net}$ values were determined, $\dot{Q}$ could be calculated at every point using Equation 13.

Due to fouling throughout an extended trial and an increase in the total thermal resistance, the heat flux of the heat exchanger could potentially reduce over time. The average heat flux was calculated at all collected data points in time throughout the trial, producing a set of new data, heat flux and elapsed time. The best way to demonstrate the reduction in heat transfer was by displaying it graphically. Graphing the experimental heat flux of the heat exchanger as a function of time illustrated both the total reduction in heat flux as well as the trend of this reduction.

No calculations are performed on the differential pressure data collected during the trials. The differential pressure data was displayed in a scatter plot as a function of trial time.
CHAPTER 5: RESULTS AND DISCUSSION

5.1 Introduction

The overall goal of this research was to experimentally investigate heat transfer and hydraulic resistance due to fouling in flowing brine solutions at supercritical pressure. To accomplish this, three objectives were proposed and completed.

- Objective 1: Create a one-dimensional supercritical water heat transfer model and compare its results to those obtained from the experimental results.
- Objective 2: Quantify and plot average heat flux as a function of time through a series of trials.
- Objective 3: Quantify and plot heat exchanger differential pressure as a function of time through a series of trials.

These objectives were completed in three phases. In Phase 1, the heat transfer model was created and the results were used to design the experimental heat exchanger test system. In Phase 2, the experimental system was constructed and tested with clean water to establish baseline heat transfer rates to compare with the heat transfer model results. In Phase 3, a series of trials were performed with three separate brine test fluids of varying composition and pH. Heat exchanger temperature profile and differential pressure were monitored and recorded throughout the trials.
5.2 Objective 1: Heat Transfer Model

5.2.1 Initial Results

The first set of results obtained were those from the heat transfer model. The heat transfer model calculations were completed in the initial stages of Phase 1 of the project. There were two main deliverables associated with the work towards Objective 1. The first deliverable was an estimate for the length of heat exchanger required for the design of the experimental heat exchanger test system. The second deliverable was the predicted heat flux for the experimental heat exchanger for the purpose of comparing the model results with the experimental results.

The heat transfer model result predicting the required dimensions of the experimental heat exchanger were vital to the completion of all research objectives. The experimental test plan specified that the experimental heat exchanger must be capable of heating the test fluid, flowing in the tube side of the heat exchanger, from 200°C to 350°C. Specific details concerning the methodology of the model are described in Section 4.1. The results are displayed as the calculated temperature profile of the experimental heat exchanger and are shown in Figure 15. The results indicate the length of heat transfer area required to meet the experimental temperature range is approximately 2.3 m. The relevant model inputs and results are displayed in Table 5.
Figure 15: Heat Transfer Model Initial Results

Table 5: Heat Transfer Model Initial Results

<table>
<thead>
<tr>
<th>Model Input</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shell Fluid Inlet Temperature</td>
<td>400 °C</td>
</tr>
<tr>
<td>Tube Fluid Outlet Temperature</td>
<td>350 °C</td>
</tr>
<tr>
<td>Heat Transfer Length</td>
<td>2.3 m</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Model Result</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shell Fluid Outlet Temperature</td>
<td>378 °C</td>
</tr>
<tr>
<td>Tube Fluid Inlet Temperature</td>
<td>214 °C</td>
</tr>
</tbody>
</table>

The results displayed in Table 5 show that with an inlet shell fluid temperature of 400 °C, and an effective heat transfer length of 2.3 m, the tube side fluid must enter at 214 °C to reach the target outlet temperature of 350 °C. This result differs slightly from the target temperature range of 200 °C to 350 °C. However, the heat transfer length of 2.3 m was chosen as the specific dimension used in the design of the experimental heat
exchanger. This decision was made for several reasons, the first being that 2.3 m was a conservative estimate of the length required. Going with a longer length of tube section would have significantly complicated operation and maintenance of the system. Specifically, with a longer length of tubing, only 1 test section would be available from a standard 20 ft. section of delivered tubing. In addition, committing to this dimension simplified the design and construction of the experimental heat exchanger.

5.2.2 Comparison of Model and Experimental Results

The second deliverable for Objective 1 was comparison of the heat transfer rates of the model with the actual results from the experimental heat exchanger. These results are presented as both a comparison of the predicted and actual heat exchanger temperature profiles, as well as the predicted and average heat flux of the model and experimental heat exchanger.

The initial shakedown trials of the experimental heat exchanger immediately showed there was a significant difference between the predicted and actual temperature profile. In order to meet the experimental design parameter of an outlet temperature of 350 °C, the inlet temperatures of both the DI water and the test fluid sides of the experimental system were reduced. For the purpose of direct comparison, the input parameters of the heat transfer model were changed to reflect the new experimental conditions and the model was re-run. Figure 16 shows a direct comparison of the actual heat exchanger temperature profile during a shakedown run vs. the temperature profile predicted by the heat transfer model.
Figure 16: Model and Experimental Results Comparison

The experimental data shown is direct temperature measurements made along the length of the experimental heat exchanger during steady state operation. This figure effectively highlights the discrepancy between the predicted temperature profile and the actual experimental temperature profile.

The second metric for comparing the heat transfer model with the experimental results was the average heat flux in the heat exchanger during steady state operation. The average heat flux predicted by the model is represented as a single value calculated using the inlet and outlet temperatures. Equations used to calculate heat flux are discussed in detail in Section 4.3. The value for the experimental heat flux is taken as the mean average of all 7 experimental trials. As part of each experimental trial, the system was preheated and allowed to come to steady state while flowing clean DI water through both
fluid loops. This occurred at the beginning of each trial before the brine test fluid was introduced. By using the average heat flux for all of the experimental trials, the results account for slight variations in the experimental setup between trials and more accurately reflect the potential range of heat fluxes that would be observed during operation.

The heat flux of the model and average of experimental trials, along with the percent difference, are shown in Table 6. With the model predicting a heat flux of 11.88 kW/m$^2$ and the mean average of the experimental trials being 35.68 kW/m$^2$, it is clear that there is a notable discrepancy between the predicted and experimental results. The percent difference between the two values is calculated using Equation 16

$$\% \text{ Difference} = \frac{\dot{Q}_{\text{Average Experimental}} - \dot{Q}_{\text{Predicted}}}{\dot{Q}_{\text{Average Experimental}}} \times 100$$  \hspace{1cm} \text{Equation 16}$$

where,

\begin{align*}
\dot{Q}_{\text{Average Experimental}} &= \text{Mean average experimental heat flux, } \left( \frac{kW}{m^2} \right) \\
\dot{Q}_{\text{Predicted}} &= \text{Heat flux predicted by model, } \left( \frac{kW}{m^2} \right)
\end{align*}

Comparison of the heat flux predicted by the model and the average experimental heat flux is an effective metric for directly quantifying the validity of the predicted results. Simply reporting the difference in the outlet temperatures of the model vs.
experimental results does not accurately reflect the total difference. Since the system fluids are at supercritical pressure, their specific heats are not constant and differences in temperature are not directly proportional to heat transfer. Reporting this comparison as heat flux instead of temperature accounts for this and gives a true comparison.

In order to further investigate the large error between the model and the experimental results, some verification calculations were performed. The heat flux in the heat exchanger was calculated by hand using the estimated overall heat transfer coefficient and the log mean temperature difference calculated from the experimental inlet and outlet temperatures. The result was an estimated heat flux of 39.4 kW/m². This much more closely matches the experimental result of 35.68 kW/m². It has a percent difference of 10.4% from the actual experimental results. The constant heat flux calculated was used to estimate a system temperature profile for comparison with the experimental results. This comparison is shown in Figure 17.
5.3 Experimental Testing Difficulties

A number of technical difficulties were encountered during the course of the experimental trials; mainly from debilitating corrosion to crucial system components. Approximately 14 hours into the 96 hour run of Trial 1, several fluids leaks were observed in the test fluid loop tubing upstream of the test heat exchanger. Review of the trial data suggested the leaks began around the 8 hour mark. Examination of the failed tubing lead to the obvious conclusion that corrosion had caused the tubing failure. Failure of vital components 8 hours into a planned 864 hours of tests made clear the test fluid loop’s material of construction, 316 stainless steel, was unsuitable for this application. Several modifications were made to the test system; all of the 316 SS tubing on the test fluid loop was replaced with tubing constructed from Hastelloy C-276 and the condenser was substituted for a replacement constructed from Inconel 625. In addition to
the system modifications, the test plan was modified to reduce the elapsed time of each trial from 96 hours to 12 hours. There was no evidence of fouling from either the heat flux or differential pressure results during the entire duration of Trial 1. Taking into account the significant damage to the system sustained in a short time during Trial 1, it was concluded that the extended trial length was both unnecessary and impractical.

During Trial 5, with the pH 4.0 test solution, additional technical difficulties arose. At the conclusion of the trial, after approximately 12 hours of operation, a steam leak was observed from the preheater on the DI water loop (HX-140). Analysis of the test tube section revealed significant corrosion and cracking. The exterior cracking is clearly visible in Figure 18 and the boroscope image shown in Figure 19 shows interior corrosion and cracking.
Figure 18: Test Tube Section Showing Corrosion Cracking
Extensive damage to the test section allowed test fluid to leak into the DI water loop. The introduction of the test fluid constituents into the supercritical-temperature preheater constructed from 316 SS caused the secondary tubing failure and subsequent steam leak. Following this incident, the damaged preheater (HX-140) was rebuilt. The decision was made to not conduct the planned Trial 6, the third trial with the pH 4.0 solution. It was determined that these operating conditions posed too high a risk of irreparable damage to the system since sufficient experimental data was obtained. In addition, the target length of the remaining trials was reduced to 7.5 hours. As a result of the extensive technical difficulties experienced during the course of the tests, planned Trials 6 and 9 were not conducted. The final modified test matrix is shown in Table 7.
Table 7: Modified Test Matrix

<table>
<thead>
<tr>
<th>Trial</th>
<th>Test Fluid Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>B</td>
</tr>
<tr>
<td>2</td>
<td>B</td>
</tr>
<tr>
<td>3</td>
<td>B</td>
</tr>
<tr>
<td>4</td>
<td>A</td>
</tr>
<tr>
<td>5</td>
<td>A</td>
</tr>
<tr>
<td>6</td>
<td>C</td>
</tr>
<tr>
<td>7</td>
<td>C</td>
</tr>
</tbody>
</table>

The removal of two trials from the test plan eliminated some of the ability to demonstrate repeatability of results. However, the trials that were conducted demonstrated a consistent result of no significant change in heat flux. These results proved sufficient enough to draw conclusions about the effects on the heat flux within the experimental system.

5.4 Objective 2: Heat Flux

The second objective of this thesis was to quantify and plot the average heat flux of the experimental heat exchanger as a function of time through a series of experimental trials. In addition, the average heat flux for each test fluid condition, as well as the overall average heat flux of the system was reported. During each of the trials, the system pressure and inlet temperature were held constant. Each trial was conducted under an identical set of conditions; the composition and pH of the test fluids were the only variables.

The original target inlet conditions for the system were 400 ℃ and 200 ℃ for the DI water and test fluid respectively. These parameters had been determined initially from
the results of the heat transfer model. The goal was to heat the test fluid to 350°C and these temperatures were predicted to accomplish this. Due to the inaccuracy of the heat transfer model predictions, the trial inlet conditions were modified. The most vital of the trial parameters was the outlet temperature of the test fluid of approximately 350°C. A set of clean-water shakedown trials were performed to determine the correct inlet conditions that would allow the system to meet this operational requirement. The modified set of operating parameters used in the experimental trials are listed in Table 8.

Table 8: Modified Experimental Operating Parameters

<table>
<thead>
<tr>
<th>Operating Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shell Fluid (DI Water) Inlet Temperature</td>
<td>375°C</td>
</tr>
<tr>
<td>Tube Fluid (Test Fluid) Inlet Temperature</td>
<td>150°C</td>
</tr>
<tr>
<td>Heat Transfer Length</td>
<td>2.3 m</td>
</tr>
</tbody>
</table>

In addition to the change in system inlet temperatures, the flow rates for both system loops were reduced slightly for the trials. The original test plan had called for a volumetric flow rate of 100 ml/min to be delivered to each loop using piston driven positive displacement pumps. The shakedown trials showed that the pumps used were not capable of reliably sustaining this max flow rate at pressure for the entire length of a trial. The flow rates were subsequently reduced to 97.5 ml/min for all trials in order to maintain reliable operation of the pumps.

The first set of three trials was conducted with a test fluid of pH 6.5. The test fluid was mixed in 50 gallon batches and dosed several times to achieve the correct composition. The actual constituent concentrations were verified with ICP analysis.
Table 9 shows the target composition and pH for the test fluids as well as the actual values measured before each trial. The test fluid was only fed through the system a single time to ensure uniform composition and pH throughout the trial. In the case of the trials performed at pH 6.5, no pH correction was required.

Table 9: Trial 1-3 Test Fluid Composition

<table>
<thead>
<tr>
<th></th>
<th>Na(^+) (mg/L)</th>
<th>Ca(^{2+}) (mg/L)</th>
<th>Ba(^{2+}) (mg/L)</th>
<th>Mg(^{2+}) (mg/L)</th>
<th>Sr(^{2+}) (mg/L)</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Target</td>
<td>68,805</td>
<td>26,543</td>
<td>1,517</td>
<td>1,864</td>
<td>1,437</td>
<td>6.50</td>
</tr>
<tr>
<td>Trial 1</td>
<td>65,490</td>
<td>26,060</td>
<td>1,743</td>
<td>1,837</td>
<td>1,652</td>
<td>6.60</td>
</tr>
<tr>
<td>Trial 2</td>
<td>65,490</td>
<td>26,060</td>
<td>1,743</td>
<td>1,837</td>
<td>1,652</td>
<td>6.50</td>
</tr>
<tr>
<td>Trial 3</td>
<td>65,490</td>
<td>26,060</td>
<td>1,743</td>
<td>1,837</td>
<td>1,652</td>
<td>6.50</td>
</tr>
</tbody>
</table>

The original test plan had called for 96 hour continuous trials, however extensive corrosive damage to the test system components showed this to be infeasible. In addition, the lack of evidence of fouling in the initial trials demonstrated that the extended trial length was unnecessary. The trial length was reduced to 12 hours after the results of Trial 1 and was later reduced further to 7.5 hours per trial. These changes are discussed in detail in Section 5.3.

At the beginning of each trial, the test system was pressurized, preheated, and allowed to stabilize while flowing with clean DI water. Once the system was stabilized at the correct operating temperatures, the fluid feed was switched to the test fluid and the trial was begun. As a result, the plots of system heat flux show an initial decrease but quickly stabilize. This initial decrease was due to the test system adapting to the shift from preheating DI water to the test fluid. The large difference in specific heat, density
viscosity, and other properties between the clean DI water and the brine test fluid caused a difference in heat transfer rates. The heat flux plots for the pH 6.5 tests, the initial three trials, are shown in Figure 20.

![Heat Flux Plots](image)

Figure 20: pH 6.5 Heat Transfer Results

The heat flux plots of the pH 6.5 trials show an initial decrease with the introduction of the test fluid but quickly stabilize by the 0.5 hour mark. It is immediately clear that once the heat flux stabilizes during each of the trials, there is no significant observable change in values for the duration of the trials. There is some obvious steady-state fluctuation in the values, especially during Trial 1, but this is merely a symptom of slight fluctuations in the inlet temperature and pressure values. For all trials, inlet temperatures fluctuate no more than ± 2% of their desired set point. It is also clearly observable that the constant heat flux values differ between trials run at identical
operating conditions. This variation is due to slight changes to the system properties between each trial. The test heat exchanger must be partially disassembled between trials and insulation and thermocouples must be removed and replaced. The required maintenance accounts for the lack of identical heat transfer results between trials.

The next set of trials was performed with a pH of 4.0. The fluids were mixed in 50 gallon batches and compositions were again verified with ICP. The test fluid compositions from ICP analysis are shown in Table 10.

Table 10: Trial 4-5 Test Fluid Composition

<table>
<thead>
<tr>
<th></th>
<th>Na⁺ (mg/L)</th>
<th>Ca²⁺ (mg/L)</th>
<th>Ba²⁺ (mg/L)</th>
<th>Mg²⁺ (mg/L)</th>
<th>Sr²⁺ (mg/L)</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Target</td>
<td>68,805</td>
<td>26,543</td>
<td>1,517</td>
<td>1,940</td>
<td>2,266</td>
<td>4.00</td>
</tr>
<tr>
<td>Trial 4</td>
<td>66,740</td>
<td>27,400</td>
<td>1,794</td>
<td>1,976</td>
<td>2,318</td>
<td>4.03</td>
</tr>
<tr>
<td>Trial 5</td>
<td>66,740</td>
<td>27,400</td>
<td>1,794</td>
<td>1,976</td>
<td>2,318</td>
<td>4.03</td>
</tr>
</tbody>
</table>

The pH of the test fluids for Trial 4 and Trial 5 required correcting to reach the target of 4.0. This was accomplished by dosing with HCl after the initial mixing. HCl was chosen for lowering the pH because of its availability and the fact that the addition of a small amount of addition chlorides to the test fluid would have minimal effect beyond the desired pH adjustment. The pH of the batch was monitored continuously during the process and allowed to stabilize before being recorded.

The procedure and experimental conditions of Trials 4 and 5 were identical to the previous trials, except for the composition and pH of the test fluid. Heat flux results for these trials are displayed in Figure 21.
Figure 21: pH 4.0 Heat Transfer Results

As with the first set of results, there is an observable drop in the heat flux initially before it quickly stabilizes. Additionally, there is again no observable change in the heat flux during either trial once the steady state condition is reached. Only two trials were performed using the pH 4.0 test fluid. A set of three trials had been planned, however significant damage to system components was sustained near the end of Trial 5. The results of this event showed that repeating the previous trial conditions was impractical. The system damage and test matrix modifications are discussed further in Section 5.3.

The final set of experimental trials was conducted with the basic solution pH of 9.0. The pH of each test batch was corrected immediately before each trial by dosing the test fluid with NH₄OH. Both NaOH and NH₄OH were investigated as potential bases, however lab tests showed that the addition of NaOH may cause unwanted deposition in
the mixed test fluids. The test fluid compositions and pHs for each trial are shown in Table 11.

<table>
<thead>
<tr>
<th></th>
<th>Na⁺ (mg/L)</th>
<th>Ca²⁺ (mg/L)</th>
<th>Ba²⁺ (mg/L)</th>
<th>Mg²⁺ (mg/L)</th>
<th>Sr²⁺ (mg/L)</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Target</td>
<td>68,805</td>
<td>26,543</td>
<td>1,517</td>
<td>1,812</td>
<td>1,271</td>
<td>9.00</td>
</tr>
<tr>
<td>Trial 7</td>
<td>67,039</td>
<td>26,024</td>
<td>1,727</td>
<td>1,867</td>
<td>1,504</td>
<td>8.89</td>
</tr>
<tr>
<td>Trial 8</td>
<td>64,290</td>
<td>26,650</td>
<td>1,368</td>
<td>1,694</td>
<td>1,293</td>
<td>9.04</td>
</tr>
</tbody>
</table>

Two trials were completed with the pH 9.0 test fluid. A repeat catastrophic system failure due to corrosion occurred early in Trial 8. The decision was made to cancel the last trial rather than risking further damage to the heat exchanger test system.

The heat flux plots for Trials 7 and 8 are shown in Figure 22.
Examination of the heat flux plot for Trial 8 reveals that it does not match the stable heat flux profile observed in the results of the other trials. At approximately the 4 hour mark of Trial 8 there is a distinct shift in the heat flux. This was caused by the beginning of a large leak in the heat exchanger tube section. This leak affected the flow rate of the test fluid through the test section and also allowed for contamination of the DI water loop with corrosive fluids. The increase and then sharp drop in heat flux at the 6 hour mark was caused by a sudden tubing failure and steam leak in 316 SS tubing of the DI water loop. This event is also described in Section 5.3.

During the course of all the experimental trials, approximately 200 gallons of test fluid brine was mixed and tested. The composition and pH of the fluids was the only variable between trials, making it vital the fluids be accurate and consistent. Figure 23 shows a comparison of the overall test fluid compositions. While it is clear that there was some small variation and error between batches of test fluids, they appear insignificant when taking into account the very high concentrations of dissolved solids. The test fluid compositions appear to be consistent overall and not a potential source of error in the results.
The individual heat flux plots in Figures 20, 21, and 22 show trends in heat flux but don’t clearly represent the overall average heat flux of the experimental heat exchanger under the test conditions. Table 12 shows the combined heat transfer results for each trial, and test fluid, as well as the overall average.

Table 12: Combined Heat Transfer Results

<table>
<thead>
<tr>
<th></th>
<th>pH 6.5</th>
<th>pH 4.0</th>
<th>pH 9.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trial Average</td>
<td>Trial 1</td>
<td>Trial 2</td>
<td>Trial 3</td>
</tr>
<tr>
<td>(kW/m²)</td>
<td>36.69 ± .16</td>
<td>34.06 ± .19</td>
<td>35.06 ± .22</td>
</tr>
<tr>
<td>Fluid Average</td>
<td>Trial 4</td>
<td>Trial 5</td>
<td>Trial 7</td>
</tr>
<tr>
<td>(kW/m²)</td>
<td>32.56 ± .25</td>
<td>34.99 ± .41</td>
<td>34.03 ± 1.45</td>
</tr>
<tr>
<td>Overall Average</td>
<td>Trial 1</td>
<td>Trial 2</td>
<td>Trial 3</td>
</tr>
<tr>
<td>(kW/m²)</td>
<td>35.27 ± .19</td>
<td>33.78 ± .34</td>
<td>33.61 ± 1.04</td>
</tr>
</tbody>
</table>

Figure 23: Test Fluid Overall Compositions
Objective 3: Differential Pressure

The third objective of this thesis was to quantify and plot the differential pressure of the experimental heat exchanger through a series of trials to assess fouling and impact on pressure loss and heat transfer. The differential pressure was measured through the same set of trials used to fulfill Objective 2.

The experimental data was collected using a precise differential pressure transducer acquiring pressure loss across the test heat exchanger. The results are presented as a scatter plot of the raw differential pressure data. The results of Trial 1 are shown in Figure 24. The plot shows pressure data points scattered around the average of 0 psi. In addition, the results show an even distribution around the average of zero. There is no significant measureable differential pressure trend over the length of the trials. The results of all seven trials closely match those of Trial 1. Some trials show slightly more pressure variation around the mean of 0 psi, however there is no measurable trend in the results of any of the 7 trials. The plots for Trials 2-8 are shown in Figure 25 through Figure 30.
Figure 24: Trial 1 Differential Pressure Results

Figure 25: Trial 2 Differential Pressure Results
Figure 26: Trial 3 Differential Pressure Results

Figure 27: Trial 4 Differential Pressure Results
Figure 28: Trial 5 Differential Pressure Results

Figure 29: Trial 7 Differential Pressure Results
Figure 30: Trial 8 Differential Pressure Results
CHAPTER 6: CONCLUSIONS

6.1 Introduction

The goal of this research was to investigate the heat transfer and differential pressure effects of brine solutions flowing at supercritical pressure. The first objective of the research was to create a single dimensional heat transfer model to predict temperature profiles and heat transfer rates for the planned experimental heat exchanger. These results were then analyzed and compared to the results of the experimental trials conducted. Objective 2 was to quantify and plot the heat flux of the experimental heat exchanger as a function of time for a series of trials. Seven trials were completed. Objective 3 was to quantify and plot the differential pressure across the experimental heat exchanger as a function of time for a series of trials. The data was recorded concurrently with heat flux measurements for the seven trials used to fulfill Objective 2.

6.2 Objective 1

The first deliverable for Objective 1 was a heat transfer model to predict the temperature profiles in the experimental heat exchanger. The heat transfer model was created in MATLAB and included thermophysical properties exported from EES. These properties included density, specific heat, and viscosity. The results from the heat transfer model were used to define the final dimensions of the experimental heat exchanger which was used to fulfill Objectives 2 and 3. Based on the results of the model, an effective heat transfer length of 2.3 m was chosen for the experimental heat exchanger.
The second deliverable for Objective 1 was to compare results from the heart transfer model to those obtained from the experimental heat exchanger system. The temperature profile and average heat flux of the model and experimental data were compared. Initial shakedown trials showed that the predicted temperature profile of the heat exchanger did not closely match the results, this is clearly shown in Figure 16. The heat transfer within the experimental system was much greater than that predicted by the model. In order to operate the system, the input temperature parameters were reduced to 150°C and 375°C for the test fluid loop and DI water loop, respectively. The model was adjusted with these new input parameters and re-run; this allowed for a direct comparison of the experimental and predicted temperature profiles. When plotted together, there is a clear discrepancy between the experimental and predicted temperature profiles. The heat transfer rates are under predicted by the model. Even with the difference in the predicted system temperatures, it was still able to be used for the planned series of trials by modifying the inlet temperatures. However, due to the significant deviation, the conclusion is that the initial heat transfer model is flawed and is not an effective method for predicting temperatures and heat transfer rates under the specific experimental conditions.

The second metric of comparison was the average heat flux in the heat exchanger. This was calculated using the inlet and outlet temperatures. The heat transfer model predicted a heat flux of 11.88 kW/m², while the average in the experimental system was 35.68 ± 1.74 kW/m². This represented a difference of 66.7 % between the predicted and actual experimental heat flux. Some amount of error is expected when predicting heat
transfer rates, however the difference of 66.7 % is unexpectedly high. This leads to the conclusion that the methodology of the heat transfer model used is not effective for predicting heat transfer rates under the specific test conditions.

An addition test of the model result was performed. Using the experimental inlet and outlet temperatures and the known dimensions of the heat exchanger, a set of hand calculations were performed to estimate the heat flux in the experimental heat exchanger. The result was an average heat flux of 39.4 kW/m², much closer to the experimental result than the model. In fact, this estimate calculation represents a difference of 10.42% vs. 66.7% for the heat transfer model. These calculations provide additional evidence that the heat transfer model methodology is flawed and is not applicable to the experimental conditions.

There is no single clear reason for the discrepancy in the model results, however, there are a number of factors that could contribute to the inaccuracy. First, the numerical methodology of the model may not be correctly accounting for the dramatic changes in the fluid properties near the critical point. These properties are characterized by nonlinear, stiff functions resulting in inadequate quantification of thermophysical properties near the critical point and could cause the model to underestimate the total heat transfer rates. In addition, the Nusselt correlation used (Mokrey et. al.) may not be ideal for the specific temperature and flow conditions of the trials. It had been demonstrated in the literature to be effective under a relatively wide set of conditions, however the heat flux and Reynolds number conditions in this study were below the applicable conditions in which Mokrey et. al.’s Nusselt correlations were drawn.
To fulfill Objective 1, heat transfer model results, heat exchanger design parameters and a comparison with experimental results was required. Figure 15, Figure 16, and Table 6 are provided to fulfill the requirements of this objective.

6.3 Objective 2

The deliverable for Objective 2 was plots showing the trend in the average heat flux of the experimental heat exchanger over time for the set of nine proposed trials. In total, only seven of the nine proposed trials were completed. This change was made to prevent damage to the experimental system and sufficient data could still be obtained to fulfill experimental objectives.

The first set of trials was conducted with the pH 6.5 test fluid. The test fluid was heated from 150°C to approximately 350°C at 3450 psi (23.8 Mpa). Heat flux results showed no significant measureable change in the average heat flux within the experimental heat exchanger throughout the full length of the trials. This was true for all three trials. The conclusion from this result is that at the experimental operating conditions with the pH 6.5 test fluid, no significant tube deposition occurs affecting the heat transfer rates within the system.

The second set of experimental trials used the acidic pH 4.0 test fluid at the same temperature and pressure conditions. Extensive corrosion and damage to the experimental system precluded the possibility of a third trial. The results of the pH 4.0 test solution trials closely matched those of the previous trials. Once steady state operating conditions were reached, there was no significant change in the heat transfer
The results showed that there was no measurable decrease in heat transfer due to deposition at the operating conditions with the pH 4.0 test fluid.

The third set of trials was conducted with the basic, pH 9.0 test fluid. The temperature and pressure conditions matched those of the previous trials. The data for the third set of trials is not as conclusive as the others. Trial 7 appears to show a slight decrease in heat flux throughout the length of the trial from approximately 34.5 kW/m$^2$ to 34.0 kW/m$^2$. However, there was a significant amount of pressure instability observed during this trial. The decrease in heat flux cannot be immediately attributed to deposition occurring on the tube. Trial 8 appears to show a stable heat flux once the system reaches steady state, however, a corrosion driven system failure prematurely ended the trial. The results of Trial 8 are not useful for confirming the trend observed in Trial 7. A third trial was planned, as system equipment sustained significant damage during Trial 8. Due to the potential damage and physical danger posed by another failure, Trial 9 was not attempted. The results for the pH 9.0 trials suggest that there may be a decrease in heat flux occurring due to tube wall deposition during the course of the trials, however there is not enough corroborating trial data for confirmation. In addition, the differential pressure data from the trial suggests that there is no measurable increase in differential pressure across the system due to potential fouling.

During the course of the experimental work, 7 of the 9 planned trials were conducted testing 3 different test fluid conditions, pH 6.5, 4.0 and 9.0. The data from the pH 6.5 trials conclusively demonstrates that there is no change in the average heat flux of the experimental heat exchanger over the length of the trials. The data for the pH 4.0
closely matches that of the first set of trials, there is no measurable change in average heat flux. The pH 9.0 trials data suggest that there may be a small decrease in heat flux due to fouling, however this is not supported by the differential pressure results and there is not enough data to confirm. The plots showing heat flux as a function of time, Figures 20-22 fulfill the requirements to meet Objective 2.

6.4 Objective 3

The deliverable for objective 3 was plots showing the differential pressure in the experimental heat exchanger as a function of time. This data was collected concurrently with the heat flux data for Objective 2 during the same set of seven trials.

The differential pressure data for Objective 3 is clear and conclusive. None of the differential pressure plots for any of the seven trials show a change in differential pressure throughout the trial. The average pressure for each data set is 0 psi with uniform variations positive and negative that would be expected from a differential pressure reading. Even the data for Trials 7 and 8, whose heat flux data suggested that tube deposition may be occurring showed no measurable deviation from 0 psi. It can be conclusively stated that there is no measurable increased differential across the experimental heat exchanger during any of the seven trials. Objective 3 required plots of differential pressure over time, Figures 24-30 are presented fulfill this objective.
CHAPTER 7: RECOMMENDATIONS

The heat transfer model developed to predict the heat exchanger temperature profiles, while it did allow for the design of a heat exchanger that could be used for the experimental trials, was overall ineffective. Future work should be done to identify and correct the flaws in the existing heat transfer model. The ability to accurately model and predict heat transfer and flow conditions in the reheat heat exchanger will be a vital tool in designing a full-scale IPSC system. There are several areas of the model which should be investigated further in order to improve its effectiveness. Foremost is the methodology used to numerically calculate the temperature profile. Hand verification calculations showed that heat flux and temperatures can be accurately predicted using the estimated combined heat transfer coefficient. Therefore, it is clear that a more accurate model can be developed. More trials should be conducted with the experimental heat exchanger at different temperatures and heat fluxes in order to tune and confirm the results of any modifications of the heat transfer model.

The experimental trials conducted were designed to simulate heat transfer to produced water in the immediate sub-critical temperature range of 200-350°C. Further trials should focus on the lower temperature range of 25-200°C. In addition, the composition of the test fluids used were designed with the consideration that almost all dissolved solids, except for chlorides, would have precipitated out before the inlet temperature of 200°C. The composition of test fluid for a set of lower temperature range trials should include additional constituents that would be observed in produced water, such as carbonates and sulfates. These alternative constituents may produce a more
significant deposition and fouling result than observed in the previous set of trials. In addition, trials conducted at a lower temperature could incorporate heat flux as a test variable by changing the inlet temperature of the DI water to the heat exchanger.

The differential pressure readings from the completed set of trials effectively showed that there was no measureable increase in the system differential pressure. However, there is a significant amount of noise included in the data recorded. This contributed to making the results slightly more difficult to interpret. Future trials on the test system should include signal filtering on the differential pressure readings recorded in order to produce a more clear result.
REFERENCES


