Experiments, Modelling and Validation of Crude Oil Fouling on Large Scale Rig

By

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DECLARATION OF ORIGINALITY

The work described in this dissertation was carried out in the Department of Chemical Engineering at Imperial College London between February 2011 and April 2015. Except where specially acknowledged, the material is the original work of the author and includes nothing which is the outcome of work in collaboration. No part of this material has been submitted for a degree in other university.

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Z.Tajudin
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ABSTRACT

Fouling is a complex phenomenon which commonly occurs in crude oil heat transfer equipment, reducing heat transfer, increasing pressure drops and in general resulting in reduced energy recovery efficiency and increased capital and operating costs of refineries. Measuring with accuracy the fouling behaviour of an oil at or close to industrial conditions is not easy. A High Pressure Oil Rig (HIPOR), designed to overcome current limitations and operate in a regime close to refinery heat and tube exchangers, has been developed at Imperial College under the direction of Prof. G.F. Hewitt and Prof. Macchietto.

In this thesis, an extensive and comprehensive commissioning procedure and automated system for HiPOR was presented. All instruments had been calibrated and tested in order to have an excellent precision and reliable measurements (sensors and controllers, as well as thermo-hydraulic behaviour) for baseline validation and crude oil fouling analysis. A predictive model of the HIPOR apparatus is briefly presented. The model is intended to assist in the analysis and interpretation of the experimental fouling data collected.

A dynamic, distributed thermo-hydraulic model of the tubular flow sections of HIPOR is presented. The model, an extension of Coletti and Macchietto (2011), considers the two dimensional (axial and radial) heat transfer in different domains with variable thermal conductivity (heat generation), boundary conditions and other aspects (for example, heat losses) which are carefully determined. The calibration and validation of various model components through a variety of tests is described. The model predicts temperature profile and pressure drop of fluid in the test section as well as local conditions of the physical plant and fluid properties.

Primary measurements were calibrated and showed excellent precision and reproducibility. The simulation results (temperature profile, wall temperature, differential pressure and temperature difference, outlet and inlet temperature) show a consistent agreement with primary measurements from the rig at different process conditions. A selection of friction factor correlations in the model depend on types of oil is used for the experiments.

It is concluded that the model of the HIPOR rig at ‘clean’ oil conditions and crude oil was successfully validated. Crude oil with no fouling model shows an excellent agreement against experimental data.
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# NOMENCLATURE

## LATIN

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<thead>
<tr>
<th>Symbol</th>
<th>Definition</th>
<th>Units</th>
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<tbody>
<tr>
<td>$A$</td>
<td>Surface area</td>
<td>$[\text{m}^2]$</td>
</tr>
<tr>
<td>$c_p$</td>
<td>Specific heat capacity</td>
<td>$[\text{J} \cdot \text{K}^{-1}]$</td>
</tr>
<tr>
<td>$d$</td>
<td>Diameter</td>
<td>$[\text{m}]$</td>
</tr>
<tr>
<td>$E_{\text{act}}$</td>
<td>Activation energy</td>
<td>$[\text{J}]$</td>
</tr>
<tr>
<td>$f$</td>
<td>Fanning friction factor</td>
<td>$[-]$</td>
</tr>
<tr>
<td>$h$</td>
<td>Heat transfer coefficient</td>
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</tr>
<tr>
<td>$H$</td>
<td>Height</td>
<td>$[\text{m}]$</td>
</tr>
<tr>
<td>$I$</td>
<td>Current</td>
<td>$[\text{A}]$</td>
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<tr>
<td>$k$</td>
<td>Thermal conductivity</td>
<td>$[\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}]$</td>
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<tr>
<td>$L$</td>
<td>Length</td>
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<tr>
<td>$m$</td>
<td>Mass flow-rate</td>
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</tr>
<tr>
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<td>Nusselt number</td>
<td>$[-]$</td>
</tr>
<tr>
<td>$Pr$</td>
<td>Prandtl number</td>
<td>$[-]$</td>
</tr>
<tr>
<td>$q$</td>
<td>Heat flux</td>
<td>$[\text{W} \cdot \text{m}^{-2}]$</td>
</tr>
<tr>
<td>$\dot{Q}$</td>
<td>Internal heat generation rate</td>
<td>$[\text{W} \cdot \text{m}^{-3}]$</td>
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<tr>
<td>$r$</td>
<td>Radius</td>
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<td>Fouling resistance</td>
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<tr>
<td>$T$</td>
<td>Temperature</td>
<td>$[\text{K}]$</td>
</tr>
<tr>
<td>$u$</td>
<td>velocity</td>
<td>$[\text{m} \cdot \text{s}^{-1}]$</td>
</tr>
<tr>
<td>$u^*$</td>
<td>Frictional velocity</td>
<td>$[\text{m} \cdot \text{s}^{-1}]$</td>
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## GREEK

<table>
<thead>
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<th>Symbol</th>
<th>Definition</th>
<th>Units</th>
</tr>
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<tbody>
<tr>
<td>$\delta$</td>
<td>Fouling layer thickness</td>
<td>$[\text{m}]$</td>
</tr>
<tr>
<td>$\delta$</td>
<td>Solubility parameter</td>
<td>$[\text{Pa}^{1/2}]$</td>
</tr>
<tr>
<td>$\Delta p$</td>
<td>Pressure loss</td>
<td>$[\text{Pa}]$</td>
</tr>
<tr>
<td>$\Delta T$</td>
<td>Differential temperature</td>
<td>$[\text{K}]$</td>
</tr>
<tr>
<td>$\lambda$</td>
<td>Thermal conductivity</td>
<td>$[\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}]$</td>
</tr>
<tr>
<td>$\mu$</td>
<td>Dynamic viscosity</td>
<td>$[\text{Pa} \cdot \text{s}]$</td>
</tr>
</tbody>
</table>
\[ \rho \quad \text{Density} \quad [\text{kg} \cdot \text{m}^{-3}] \]
\[ \beta(T) \quad \text{Electrical resistivity at a given temperature} \quad [\Omega \cdot \text{m}] \]
\[ \tau \quad \text{Shear stresses} \quad [\text{Pa}] \]

**SYMBOLS, SUPERSCRIPTS AND SUBSCRIPTS**

**ABBREVIATIONS**

API \quad \text{American petroleum institute}

CDU \quad \text{Crude oil distillation unit}

CFD \quad \text{Computational fluid dynamics}

DAQ \quad \text{Data Acquisition}

DOE \quad \text{Department of Environment}

ESDU \quad \text{Engineering Science Data Unit}

HAZOP \quad \text{Hazard and Operability Study}

HIPOR \quad \text{High pressure oil rig}

HTS \quad \text{Heat Transfer Society, UK}

HTRI \quad \text{Heat Transfer Research, Inc.}

SOP \quad \text{Standard Operating Procedure}

SARA \quad \text{Saturates, aromatics, resins and asphaltenes}

TEMA \quad \text{Tubular Exchanger Manufacturers Association, Inc.}

U.S.A \quad \text{United State of America}
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1 Introduction

Fouling in heat exchangers is still a major problem for the process industries, including oil refining. It is a complex phenomenon which commonly occurs in crude oil heat transfer equipment, reducing heat transfer, increasing pressure drops and in general resulting in energy recovery inefficiency and increased capital and operating costs of refineries, in addition to health and safety problems during maintenance and environmental impact of additional emissions of greenhouse gases (Coletti et al., 2014).

Establishing with some accuracy the fouling behaviour of a particular crude oil or oil blend is important to refining operations, as it would permit operators to make important decisions, such as on which refinery to process a crude, assessing the expected length of a run before a heat exchanger, a pre-heat train (PHT), or the whole refinery should be shut down for cleaning, and choice of control and mitigation strategies.

An extensive research on crude oil fouling at Imperial College has started back in 2006 where Imperial College lead a £2.1M Engineering and Physical Sciences Research Council (EPSRC) project funded in Crude Oil Fouling (CROF) project together with the University of Cambridge and University of Bath (Macchietto et al., 2011). The success and expertise on the first project has been retained and in part moved to a second phase within the UNIHEAT project (Centre of Applied Research on Energy efficient Heat Exchange and Catalysis), established on 15th February 2012 (Macchietto, 2015). This £9.3M project, funded by Skolkovo Foundation and BP Russia, involves leading researchers from Imperial College London, Boreskov Institute of Catalysis and British Petroleum. The main objective of this project is to increase energy efficiency and reduce heat losses in oil refining by up to 15% in Russia. The work presented in this thesis describes activities partially developed within Theme 2 (Oil fouling tests in HiPOR facility) in the UNIHEAT project.

1.1 Background

Fouling phenomena is not a new problem in heat transfer research area. For an oil refinery, it has been estimated 6% of energy being produced for production is used for their processing
units (ESDU, 2000). Furthermore, two third of total energy consumed in these processing units is used for heating up crude distillation unit (CDU). CDU is equipment where the separation process taking place. This process required significant energy to separate crude oil into their derivative for further purification process. The crude oil will be heated to their boiling points, and being streamed according to desired production. This production of each petroleum product depends on the demand of economic analysis.

The impacts with the crude oil distillation process operation which are ranging from economics, resources and environment aspects. In this process, the combustion process occurred where the energy being consumed and greenhouse gas (GHG) as carbon dioxide and carbon monoxide being released to atmosphere. In addition, the resources of crude oil have been depleted significantly for the past 20 years and the usage has been increased due to industrial development in developing and third world countries. It is relatively important to optimise the operation CDU to reduce the above mentioned issues.

In CDU process, one of methods to minimise combustion process, is by increasing the inlet temperature to CDU. Most refineries have series of heat exchanger before CDU unit. Multiple heat exchangers are also known as pre-heat exchanger trains (PHT). It is important to get the optimum heat exchange in this PHT. The output temperature of PHT will be the source for the CDU.

A heat exchanger is an equipment where a process of heating and cooling are taking place. The process of heat exchange involves fluid as heating or cooling medium. Normally, before any chemical plant being built, the study of heat integration in the whole plant shall be done, to identify the heat or cooling requirements. A heat transfer process involving heat exchanger has a long standing problem known as fouling. Fouling is a phenomenon where the efficiency of the heat exchanger is being reduced, which can contribute to thermal and hydraulic problem in heat transfer process. This fouling can be contributed by precipitation, particulate, chemical reaction, corrosion and bio-fouling.

As a result, continuous effort toward identifying the root causes of fouling phenomena has taken into place to fully understand the physical process of these phenomena, hence, increasing the efficiency of heat transfer process that could benefit the global industrial player in different process application. Refining crude oil could be considered as the initial stage of downstream processing. It is a process where the crude oil is transformed to intermediate and/or final product. Amongst the products from the refinery are kerosene, gasoline, liquefied petroleum gas, bitumen and etc. As mentioned earlier, normally 6% of product produced is used as energy for refinery processes and 4% mainly for heater before the crude distillation unit.
A refinery process technology commonly depends on the type of crude oil being used in the process. Crude oil can be divided into 3 categories; light crude oil, medium crude oil and heavy crude oil. These categories depend on the density of crude oil themselves and standard that commonly used is API number of relevant crude oil. There are a few standards being used, namely the American Petroleum Institute (API), New York Mercantile Exchange (NYMEX), and National Energy Exchange (NEC). Sometimes, the industrial players divided crude oil into 2 categories; sweet and sour oil. Most common factors of these crude oils are the existence of the sulphur content. The pre-treatment process or commonly known as the desulphurisation process normally is installed before the cracking process which occurs inside the reactor/reformer. This sulphur component actually can become poisonous to the catalysis in this process by reacting with an active side/pore of catalyst.

Figure 1.1 Schematic of Refining Process (Coletti, 2010)

Desalter is a process to remove all remaining precipitation/particle before it entered a series of heat exchangers. This process applied to minimise corrosion and/or aggregation inside the heat exchanger, especially in the tube side.
The main objective of heat exchanger being installed before heating process is entirely to take advantages of excess energy produced by product and by-product at the crude distillation column. The pre-heat train heat exchanger also can reduce energy consumption for heating processes before it enters crude oil distillation. The arrangement and design of this pre-heat shall be carefully identified to optimise heat transfer, hence increase their efficiency. There are few beneficial factors of this process; reduce energy consumption via fuel being used, decrease by-product of the combustion process, save GHG emitted to the atmosphere.

The transport phenomena or heat transfer process in particular required a thorough understanding of mass and energy transfer. This knowledge could lead towards better understanding of the science of thermal and hydraulic effect in heat exchangers networks.

The U.S. Department of Energy (2006) quantified the energy consumption in petrochemical application using three measurements:

- **Theoretical minimum energy (TME):** Measurement of the least amount of energy for particular process or in ‘ideal condition’. Data commonly obtained based on calculation with the first principle of process (thermodynamics/reaction rate and etc.) and/or industry publications.

- **Practical minimum energy (PME):** Measurement of practical amount of energy being used based on real industrial application by taking into consideration the operation/equipment's limitation. It is always referring to the best practice available and technically the most saved operating methods.

- **Current average energy (CAE):** Measurement of energy consumption under normal operation. This calculation includes the energy losses due to inefficient process/equipment.

These measurements basically identify the energy losses in petrochemical plant operation throughout the U.S.A and help developing methods to control this problem. Thus, these bandwidths differences can lead to exploring opportunities in improving current technologies, process design, best practice which can decrease capital and operation cost and GHG emission to atmosphere.

Figure 1.2 shows the current average energy being used for different chemical processes and potential energy saving based on (U.S. Department of Energy 2006). The graph has been simplified by taking the potential saving based on CAE deducted by PME. Even though TME value is relatively smaller compare to PME but it is impossible for a process to achieve ideal condition. Figure 1.2a shows the amount of current energy used in different processes, where the distillation processes (atmosphere and vacuum) representing nearly 43% of total energy consumption. Due to higher consumptions, figure 1.1b shows a potential energy saving can be
made by combining both distillation conditions are approximately 57% out of total potential saving.

![Figure 1.2 Percentage of current average energy (CAE) and potential energy (CAE - PME) saving based on (U.S. Department of Energy 2006).](a)

![Figure 1.2 Percentage of current average energy (CAE) and potential energy (CAE - PME) saving based on (U.S. Department of Energy 2006).](b)

As a result, it is important to explore on energy saving for the distillation process in the refinery. The most common problem for distillation unit is energy being used to heat up crude oil to the desired temperature before entering crude oil distillation. As mentioned previously, it consumed more than 60% of energy by total amount energy used in the refinery (ESDU, 2000). By increasing heat exchanger network efficiency, it probably could reduce energy used for the combustion process. The main issue with heat exchanger network particularly in a refinery is fouling deposition.

In this research, the contents will concentrate on chemical fouling. This fouling deposition could possibly contribute towards two types of effect vis. thermal and hydraulic effect. For thermal effect, it will decrease heat transfer coefficient, hence the outlet temperature for pre-heat train will slightly lower compare to 'clean condition. For hydraulic effect, commonly contributing towards 'off service' heat exchanger, it will decrease the output of processing crude via reduction of flow area inside the heat exchanger. The understanding of physical processes and the identification of fouling threshold could possibly minimise the potential shutdown due to thermal and hydraulic effect. Early detection via measurement of primary parameters and model prediction control would help to identify, hence mitigate these problems before it becomes severe. Furthermore, the idea of identifying uncertainty of primary measurement, calculate the error of propagation would bring the validation process to new benchmark in fouling phenomena. In addition, the development of control strategy that would be introduced for experimental rig could possibly help in term of imitation to industrial’s actual plant setup.
1.2 Motivation

Despite study of fouling being physically illustrated in Kern and Seaton method for more than 50 years ago, extensive studies have been made, fouling is far from been understood in its whole complexity (Müller-Steinhagen 2011). (Coletti et al. 2014) mentioned that:

"Although good progress has been made in experimental studies of crude oil fouling, it appears that an asymptotic state of knowledge has been reached. Data-led mitigation is an active area, but this provides a response rather than a cure. Whilst the results are useful, the mechanism by which the fouling proceeds are still not fully understood"

The impact of crude oil fouling of the heat exchange network, which includes operation, economics, environments and safety and related cost associated with it is shown in figure 1.3.

Figure 1.3 Study on impact of fouling for heat exchanger network and related cost associate with it (Coletti & Hewitt 2014)

Numerous reviews (ESDU 2000; Macchietto et al. 2011; Müller-Steinhagen 2011) have highlighted the complexity of crude oil fouling area and experiment data that have been collected still cannot fully explain this phenomenon.
Figure 1.4 Crude oil fouling challenges and current experimental rig availability [nano- micro scale, batch]

Figure 1.4 shows the challenges and issues need that to be addressed and the scales of experimental work that may be done. Under the direction of Prof G.F. Hewitt, Imperial College has developed a large experimental rig to overcome the limitations of primary measurements.

1.2.1 History of crude oil fouling research

The main contributors toward fouling phenomena are precipitation, particulate, chemical reaction, corrosion and bio-fouling. It also agreed amongst the researchers, the process of fouling undergone 5 processes namely; Initialisation, Transport, Attachment, Removal, Aging. (Epstein 1983) developed a 5 x 5 matrix (Figure 1.5(a)) to identify the research progress in fouling of heat exchanger to date. An updated version of the matrix was presented and discussed at recent conference EUROTERM in Schladming (Müller-Steinhagen 2009), which is illustrated in Figure 1.5b.

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Epstein (1983)

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Muller-Steinhagen (2009) (b)

Figure 1.5 The comparison progress of research in the fouling heat exchanger where the darker shade indicates the increment of research activities.

It is important to highlight that the chemical fouling is the type with more areas requiring further research. Referring to Figure 1.5a, it is assumed the fouling formation of heat exchanger
network occur mainly contributed by chemical fouling. Fouling like precipitation, particulate, corrosion and bio-fouling are minimum as deployment of desalter minimises or eliminates such associated fouling.

A compilation of study started as early as 1950s, when the Tubular Exchanger Manufacturer Association (TEMA) presented papers on how important fouling factor to heat exchanger duty. ESDU International produced a study that covers a different angle of heat exchanger fouling in refinery process (ESDU 2000). Energy bandwidth study (U.S. Department of Energy 2006) and annual saving using process simulation (U.S. Department of Energy 2004). The European Community has taken initiative back in 1992 as JOULE Non-Nuclear Energy Program (Pilavachi & Isdale 1993) and recently (Coletti and Hewitt 2014).

1.2.2 Chemical fouling

Chemical fouling can be divided into three categories (Figure 1.6) as described in (Panchal and Watkinson 1994). The differences between those are the occurrence of fouling deposition location. As illustrated in Figure 1.6, case 1 shows the fouling occur as early as it flows in bulk liquid and it will transfer its molecule (a) or particulate towards the heat transfer surface/wall for further reaction. The reaction becomes rapid as the temperature at the wall is higher compared to other places in the conduit or tube. Case 2 shows the reaction of fouling only occurs at thermal boundary layer where some of fluid flow into bulk of liquid and others will form deposits as the temperature is increasing due to flow near to the wall. If the bulk temperature is near to wall temperature, most probably case 3 will happen. The molecule directly 'attach' to hot heat transfer surface and all reaction of fouling formation to deposit layer would happen here.
The fouling deposition will apparently reduce the flow area of the fluid, hence increasing pressure drop in the tube and/or conduit. Most of the heat transfer network or heat exchanger is put out of service because higher pressure drop instead of large drop of temperature difference.

It is relatively important to understand further this fouling chemistry and types of fouling process occurs by getting primary measurements of the test section. In this case, Imperial College has developed high pressure oil rig (HiPOR) to cater this process. The rig will be operated as industrial operating condition with the option to validate with a distributed and dynamic model for different test sections. This exploration could possibly explain scientifically chemical fouling phenomena.

In any of these cases, it is expected that through the asphalthene precipitation via chemical reaction changes the crude oil characterisation, hence it could be compared with the oil before undergoing the heat transfer process.

A thorough survey on crude oil fouling science, models, and economic impact can be found in (Coletti et al. 2014), and the reader is referred to that reference for further details.
1.3 Aims and Objectives

This thesis aims to provide comprehensive testing of this large scale equipment including experiment design, commissioning, monitoring, control, modelling and validation in with safety and industrial standards. The specific objectives of this thesis are:

1. To develop a comprehensive method of commissioning and preparation for large scale crude oil fouling, design a new control system for important operating parameters, and installed and calibrated the primary measurements at strategic places with high accuracy and repeatability.
2. Develop and improve a mathematical model for the test section in order to evaluate thermal and hydraulic effects for crude oil fouling experiment.
4. Experiment runs with blended crude oil at different operating temperatures.

1.4 Thesis structure

In chapter 2, the experimental rig HiPOR will be discussed in detail. The chapter is divided into 3 important subchapters. It covers how the rig is commissioned, the principle and design of an automated control system in order to control the operating condition and lastly the data acquisition and reliability measurement. The HiPOR commissioning, for initial stage of HiPOR design done by Pental (2011), is explained in detail in this thesis as an extensive and new modifications had been made in compliance with industrial safety regulation and control framework. The automated control system was installed within LabView environment in order to monitor, control and logging all important parameters for the rig. The important parameters have been calibrated to the industrial standard and any disturbances have been taken into consideration.

In chapter 3, a dynamic and distributed mathematical modelling for a single tube model, which has been developed by Coletti (2010) and licenced to Hexxcel Ltd. (Hexxcell Ltd. 2015), has been adapted and used to describe the actual geometries test section of HiPOR. Some modifications to the original model have been made with inclusion of the joule heating and insulation model, and the dynamics of tube resistivity.

In chapter 4, a baseline validation is reported with non-fouling oil. This data is very important since it was used to characterise the thermal and hydraulic profile of the experimental rig without any occurrence of fouling. From the reliable temperature measurement of inlet and outlet of the test section, the heat losses have been quantified. This finding of the real characterisation of physical rig together with thermal and hydraulics profiles were useful for analysis and comparison with crude oil fouling experiment.
Chapter 5 discussed the experiments run with crude oil. Comparisons of thermal and hydraulic profiles were presented in this chapter between runs using non-fouling oil and crude oil. All experimental results were validated against HiPOR model.

Chapter 6 summarised all findings and contribution of this thesis for crude oil fouling research undergoing in the large experimental rig. Furthermore, future recommendations have been proposed for future exploration in crude oil fouling research.
2 HiPOR Commissioning and Automated System

2.1 Introduction
The preparation for the experiment of crude oil fouling is a challenging area for a large scale rig. There are various concerns which need to be taken into considerations involving equipment, safety, data qualities, measurement accuracy and reproducibility. HiPOR is not with the exception to a high standard practice which commonly implemented in the chemical industries environment. In this chapter, three main focus areas will be discussed thoroughly, namely commissioning procedures and experiment, commissioning and design of automated system and lastly data acquisition and its reliability. All discussed areas are essential elements that need to be carefully entertained before any attempt of running an experiment in order to obtain a good and quality data for further analysis.

2.2 HiPOR Apparatus
The High Pressure Oil Rig (HiPOR), has been developed at Imperial College London under the direction of Prof G.F. Hewitt and Prof Macchietto to overcome current experimental limitations and operate in temperature, pressure and flow regimes close to those of refinery heat and tube exchangers (Macchietto et al. 2011). The apparatus (shown in Figure 2.1) features a 1.8 m long tubular and annulus flow sections with fine control of heat input, supplied via Joule heating. The equipment is designed to operate at a maximum bulk temperature of 300°C and pressure up to 30 barg.

In the annular test section, a radiation equilibrium thermocouple is designed to measure the inner wall temperature of the heated tube. The thermocouple can be axially traversed into the annular gap to obtain temperatures at different locations along the test section. This is one of the key advantages of using an annulus section as it allows monitoring of the temperature profile along the axial directions providing insights on specially distributed phenomena involved with fouling. Moreover, the annulus test section allows an easy access to the deposits after the test is completed which can then be characterized by an array of chemical and physical analytical methodologies.
Figure 2.1 Simplified schematic of the HiPOR facility (Coletti & Hewitt, 2014)

Figure 2.2 Picture of the HIPOR test sections and the control panel.

Figure 2.2 shows a picture of the two HIPOR test sections and its control panel. The entire rig was allocated inside approximately 6.5 m$^3$ enclosure system. This enclosure system has been equipped with all necessary safety features which include rapid ventilation system with capacity 100 air exchange per hour, carbon dioxide (CO$_2$) suppression system and hydrocarbon sensors.

2.2.1 LabView Environment

It is essential for scale up process in the pilot scale experimental setup to have an excellent monitoring and control system. In HiPOR, LabView software that is developed by National Instrument is used for this purpose (National Instruments Corporation 2013). All schematic diagrams which include important equipment, valves, instrumentations, control loops was
designed in house and licensed to Hexxcell Ltd (2015). This custom made design is not only used for monitoring and controlling purposes, but it has extended to have safety features. It has the capabilities to protect the safety of processes and equipment if any occurrences of major hazardous items listed in standard operating procedures. As mentioned earlier, figure 2.3, 2.4, 2.5 illustrate some of the features which are included in LabView environment.
Figure 2.3 Overview schematic diagram of HiPOR in LabView
Figure 2.3 displays an overview schematic diagram of HiPOR in LabView environment. The important parameters and safety interlock have been displayed at this front view. It is noted, the selection of colour schemes also plays important roles for monitoring purposes as it can differentiate the current status of individual equipment. For example, V3 and V22 are isolation valves which isolated the main tank from the other sections of HiPOR. Red colour gives indication that both valves were closed and green colour means, they are opened. Another example is, the introduction of joule heating to preheater section and test sections. Initially, they appear in the blue colour pipe. Once electrical current is introduced to these sections, the colour is changing from blue to red. This shows how important to design overview schematic diagram for monitoring purposes that is compliance with industrial environment and gives an excellent indication for current status of the rig.

Another factor in LabView usage is controlling the important parameters as shown in Figure 2.4. The figure shows an example of the front view of control system that has been implemented for temperature control at preheater section. In this control display panel, all important features which include operator activities (changing mode and parameters), controller tuning, graphs (setpoint, process variables, controller output) have been displayed. Details on control system will be discussed later in this chapter.

Figure 2.4 Control system for temperature at preheater, TC01
As shown in figure 2.5, the parameters surrounded the preheater are monitored closely in real time to ensure the smooth in operation. In this case, power supply unit parameters, current, IR1, and voltage, ER1 were plotted and monitored closely. Other monitoring parameters are wall temperatures at outlet, T17 and middle, T18 of preheater and outlet bulk temperature, T16.

### 2.3 Commissioning Procedure and Experiments

Developing commissioning procedures is a vital aspect for every experimental rig especially when it involves the integration of various equipment and auxiliary systems. It is very important to ensure that all equipment meet the design specification. A systematic procedure needs to be developed for testing and verification of individual configurations. Baseline data need to be recorded to characterise its operation and for future reference, and to detect any abnormalities found during normal operation/experiment. This procedure can also be used to develop the competency of operation team in understanding the functionality and safety awareness of the equipment.

The commissioning procedure is not only on individual equipment but has to also be extended and integrated for the whole rig. It is a challenge to develop a standard operating procedure which covers the different phases of operation in order to ensure the rig is operated in a safe
and efficient manner. The most common works that need to be considered are the integration of operation process, preliminary checklists, start-up, experiment phase, shutdown, safety interlock and control system. This work required the ability to understand the experiment objectives, knowledge of equipment/instrumentations, control system and experience. Furthermore, the data that is obtained from commissioning process could be used as a benchmark for any future changes in operating conditions or upgrading the rig.

2.3.1 **Developing standard operating procedure**

It is essential to develop a standard operating procedure (SOP) in order to standardise every single step which is required to be followed and hence to uphold a high engineering practice in the experiment. These well-documented materials are required for safety, maintenance and smooth in operation.

In HiPOR standard, SOP was developed covering every major and auxiliary equipment in the rig, preliminary inspections prior to start-up, loading/unloading of raw materials, different phase of operation and shutdown. Shutdown process has been categorised into three categories namely normal shutdown, minor and major emergency shutdown. Emergency shutdown may involve the use of the CO₂ suppression system. These procedures detail systematic steps for the individual category, monitoring parameter(s) /variable(s), and safety checks before proceeding into different phase especially during operation. It also states critical parameters that need to be taken into account for safety purposes. Some of the items in SOP have to be read together with equipment technical data which contain very comprehensive information regarding that specific equipment.

SOP is generally reviewed quarterly or as soon as any modification has been made at any part of the rig. Normally, a team member who is in charge to make an amendment/update the SOP will circulate to team member for review purposes before it can be approved by Asset Owner/Principal Investigator. The updated version of SOP is shown in Appendix A.

2.3.1.1 **Objectives**

A major focus in developing SOP is to provide a convenient and conducive working environment not only to the operation team but also to the external personnel who are involved directly and/or indirectly with HiPOR. The external personnel could be the contractors that were involved with maintenance works, visitors or other researchers who shared the same laboratory with HiPOR. The main concern is to avoid unforeseen incidents which may cause fatality. Due to this reason, the personnel who involve directly with HiPOR operation has to undergo a comprehensive training to fulfil the competency level as an observer or an operator or senior operator.
In Appendix A, SOP has been divided into 7 sections and each section provides as much information as possible to guide the operator to run the rig in systematic and safe manner. It is very important to read and understand the first three parts of SOP which stated the list of equipment, main safety aspects and maintenance procedure/requirement. Although the information is not details as equipment technical specification, nonetheless they are sufficient to create awareness amongst operators as well as visitors.

2.3.1.2 Rig setting
The rig setting is one of the most crucial parts in preparing for any planned experiment. These procedures are detailed thoroughly in subsection 4.1 to 4.4 of Appendix A. They include the preliminary inspection, loading/unloading, and preliminary operations. The preliminary inspections emphasised on line setting (opening/closing of valves), visual inspection of auxiliaries, control and safety equipment and availability of process oil. It is an essential and mandatory procedure to ensure the readiness of rig for the start-up procedure. Figure (i), (ii) and (iii) in Appendix A show the line setting for preliminary inspections, loading procedure and unloading procedure respectively. The loading/unloading procedures only applies if the tank is empty or the planned experiment requires a change of oil. Lastly, the preliminary operations concentrate on starting up the auxiliary and safety systems (nitrogen, cooler, CO₂ suppression and rapid ventilation systems) and check its functionality with specific indicators which stated in items 4.4.2 in SOP.

2.3.1.3 Start up and shutdown
Start-up phase of experimental rig needs to be designed carefully in order to ensure a safe operation and to avoid sudden increase of adverse operating conditions which could damage the equipment. Primarily, the rig needs to be free from combustible gases that may induce the possibility of fire and/or explosion when the temperature starts to increase beyond permissible limits. This can be done by purging the system, mainly in the tank, several times with inert gas (pure nitrogen). Subsection 4.5.1 in Appendix A explained thoroughly the steps need to take to start-up of experiments until the operating conditions are achieved. The main concerns for start-up procedure are to ensure that the safety equipment such as rapid ventilation system, CO₂ suppression system, interlock system are in excellent condition. Moreover, the functionality of monitoring system (parameters and gas detector) via LabView is working properly. It has been highlighted in subsection 4.5.1.21, there should there be any deviation and/or abnormalities detected from the planned experiment, the experiment shall be ceased immediately by proceeding into the normal shut down procedure. It is also important for the operator to monitor the changes of operating parameters closely during the start-up phase.
Shut down phase is common for any experiment run. This phase is important as a start-up and normal experiment. This phase needs to be monitored closely so the sequence of events is followed accordingly. The consequences of a sudden drop of operating parameter may cause damage to equipment e.g. temperature, it can create thermal shocks to the properties of material. Hence, the lifespan of equipment may reduce.

2.3.1.4 Test, calibration and normal experiments
Design an experiment either it is a leak test or calibration of instrumentation or run a normal experiments, requires a proper planning and comprehensive understanding. In HiPOR case, all works involving the rig need to be planned and the permit to work needs to be obtained. Section 4.5.2 of Appendix A only provides the superficial information on how the experiment should be run and monitored. This procedure and workflow will be explained in section 2.2.2 in this chapter.

2.3.1.5 Emergency cases
Emergency cases have been identified through hazard and operability study (HAZOP) by HiPOR team. Based on this study, the emergency cases can be divided into two categories namely Scenario A – Major Hazard and Scenario B – Minor Hazard which has been identified and stated in item 4.5.4 and 4.5.5 respectively. The major concern is when scenario A happen. Even though the emergency shutdown procedure has been illustrated in details, most of the items have been included in the safety interlock system. For example, if no flow circulating through the main test sections, the power supplies to preheater and the main test section will be automatically CUT OFF. The emergency push button for heater is one of extra protection to ensure that the hardware connection has been disconnected. The other steps in emergency shutdown were made to minimise the impact of major hazard which may cause fatality and/or damage to equipment. Another example of major hazard is a severe leakage of oil either at the main tank or other section which releases substantial amounts of hydrocarbon and combustible gases. In order to prevent any occurrence of fire and/or explosion, a few hydrocarbon detectors had been installed at various points inside the enclosure. These detectors have an upper limit of 200 parts per million (ppm) which will be activated once these gases amount reaches and go beyond this value. Hence, the emergency procedure will automatically follows as scenario case A.

2.3.2 Component test and experiments
The experiment and designed test (leak test or calibration) required a proper planning before any attempts to execute them. Planning covers a wide range of itemised items which include the RUN number, objective (s), details of start-up and shutdown procedure, operating conditions, monitoring parameters and extra safety precautions (if any). The main reason is the experiment
can be run efficiently and could achieve the objectives. It is also useful to record any abnormalities occur during the experiment / test. A comprehensive document pertaining to design of experiment needs to be attached together with a permit to operate for approval from principal investigator/asset owner of the rig. This document normally will be used together with SOP in order to run the experiment/test. The workflow of run any experiment/test is illustrated in figure 2.6.

![Workflow for experiment process](image)

**Figure 2.6 Workflow for experiment process**

Figure 2-6 shows the process of workflow before and after the experiment. It demonstrates a systematic way of execution experiment in HiPOR rig. It is generally understood that crude oil fouling experiment is time consuming and could last up to a week. Designing an experiment is a crucial issue, thus all details need to be included in a document as shown in Figure 2.7. The figure displays the itemised information for the planned experiment which includes the experiment details, objectives, methods / procedures, expected data and any remarks (dates or extra information). This information is sufficient for anybody to have some rough ideas about the entire experiment. The expected data to be obtained should be in parallel with the initial objectives set before the experiment. In the 'methods' column, the necessary steps to be taken to achieve the operating conditions are explained. The steps should be read together with the SOP. In the 'remarks' section, any extra or important information, which is not covered by other section, will be highlighted e.g. planned date of the experiment. Based on this experiment planner, the permit to operate will be released/ approved.
Scheduling and planning

A systematic scheduling and planning for operation each run has been developed which include specific instructions with for start-up and shutdown operation. The schedule was also very useful as an input to simulation environment when validation took place. This will be discussed further in chapter 4 for baseline validation of HiPOR. An example is shown in figure 2.8 for experiment RUN 241.

The lists include the actual time of experiment start, experiment time, the desired valve openings, pump operations, input for power supply and pressure. Experiment run is designed to undergo each phase, which includes start-up, experiment and shutdown phase. Each phase is designed systematically based on the desired rate of temperature increment and limitation or constraints of equipment. For example, when heating process was taken place at preheater section, the wall temperature at the preheater, T17 and T18 and bulk temperature, T16 should be monitored closely. At any given time, the differences between wall temperatures and bulk...
temperature shall not be more than 50 °C. Amongst the reasons are to avoid the thermal stress for the pipe and fouling deposition in this area. Furthermore, the detail schedule also can be used to detect any deviation/abnormality between the actual measurements and projected measurement. All important parameters and action to be taken are being highlighted so that the steps can be easily followed and easy to trace by referring to actual and planned experiment time.

For example, the inlet temperature of the oil is controlled in automatic mode and the time is recorded when it has achieved a desired temperature setpoint. If the desired temperature has reached earlier / later than the schedule, this is recorded and the schedule sheet can be used in the analysis of the experimental data.

### 2.3.2.2 Data logging and acquisitions

It is a vital aspect for each experiment to log and acquire the process parameters and measurement. These data are essential in order to verify the planned experiment and subsequent analysis. In HiPOR, data logging and acquisition are done automatically in the LabView environment. Figure 2.9 displays the actual instrumentation setup in HiPOR for data acquisition and logging for pressure measurement. The block diagram in figure 2.10 shows an example of the actual implementation within the LabView environment in order to acquire and log the important parameters for the experiment.

![Figure 2.9 Actual instrumentation arrangement for pressure acquisition and logging in HiPOR](image)

![Figure 2.10 Block diagram in LabView for data acquisition and logging automatically](image)
2.3.2.3 Data processing and analysis

All logged data was recorded in a specific folder of HiPOR workstation with text based measurement file (LMV format). The main reason is this extension file offers a less amount of file size compared to other format like EXCEL. The data can be accessed in an extension of .txt file which is later transferred into Excel and/or MATLAB file for analysis. This process is shown in figure 2.11.

![Diagram of data processing and analysis workflow](image)

Figure 2.11 Data processing and analysis of crude oil fouling experiment workflow

Figure 2.11 shows each step of data processing and analysis for an experiment run for HiPOR. The data that have transferred in Excel or MATLAB can be plotted based on desired analysis on crude oil fouling studies. Some of the parameters need further analysis in order to obtain important calculated variables such as temperature difference between the inlet and outlet of the test section (delta T), heat transfer coefficient, and fouling resistance. As displayed in figure 2.11, the data also has been used as input to the gPROMS software (PSE 2015) for validation purposes. It is important for validation by inserting the actual conditions of an experiment such as inlet temperature, pressure, corrected flowrate and heat input in order to obtain the ‘real’ simulation results.

2.4 Data Acquisition and Measurement Quality

Crude oil fouling faces critical issues obtaining a reliable important measurement for data analysis. This measurement device not only need to be reliable, but it needs to be installed at strategic places for research purposes.
2.4.1 Primary measurement

The primary measurement in HiPOR covers temperature, pressure, differential pressure, flowrate, level, current and voltage. These measurements will generate secondary variables which widely used for data analysis. Secondary variables include area, volume, heat transfer coefficient, heat flux, etc. The accuracy and precision are essential. From these, fouling rate is calculated. Any deviation, error and uncertainty need to be quantified in order to avoid huge or large error of propagation of the main/final result.

Normally, control system has been setup according to international standard and best reference is The International Society of Automation (ISA). This reference would give the best practice which is commonly used by industrial players. They also recommend the suitable primary measurements that are capable to measure and capture process parameters for desired activities.

2.4.1.1 Signal Acquisition

A complete measurement consists three types of elements; sensor, transducer and transmitter. A sensor is a device that responds to a physical stimulus and converts the stimulus into a signal conveyed to another device. A signal could be received and transmitted in voltage or electrical mode. A transducer is a device which converts a physical quantity measurement into electrical signal. Sometimes, the sensor is embedded into the transducer. The output signal from the transducer is used for translating the quantity or range of the desired parameters. A transmitter is one of device that converts the measurement from sensor into signal form. This signal generally is sent to controller for control purposes. The types of signal are sent in voltage and current that normally set at 3-15 V or 4-20 mA respectively. In HiPOR, there are two types of instrumentation; analogue and digital. Analogue type of instrumentation such as thermocouple can be measured directly from the sensor itself to LabView via analogue input located at Data Acquisition (DAQ) board that shown previously in figure 2.9. For digital types instrumentation like differential pressure, which the data presents in terms of current form, needs to be converted into voltage and uses differential voltage input at DAQ board. The value read by LabView in the form of data value which based on differential voltage resolution and this value needs to be calibrated based on the span of instrumentation and offset value. Both examples will be shown in details in the later section of this chapter.

2.4.1.2 Instrumentations

The instruments used for setting up the experiment rig should be the most reliable with excellent precision and accuracy. Selections of instrumentations have been made towards its best capabilities and robustness to the process oil. Table 2.1 is a list of instrumentation with primary measurement data acquisition.
### Table 2.1 List of instruments for HiPOR

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Instrumentation</th>
<th>Manufacturer information</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>°C</td>
<td>Type K with tared pot &amp; tail -310 stainless steel</td>
<td>Range: -40 °C to 1100 °C Accuracy: ±1.1 °C or 1.5 %</td>
<td></td>
</tr>
<tr>
<td>Bulk</td>
<td>°C</td>
<td>Omega CO2-K style 2 &quot;Cement On&quot; Thermocouple</td>
<td>Range: -200 °C to 1250 °C Accuracy: ±1.1 °C or 1.5 %</td>
<td>Maximum continuous temperature - 540 °C</td>
</tr>
<tr>
<td>Wall</td>
<td>°C</td>
<td>DMK 331P - Pressure transmitter with flush stainless steel diaphragm</td>
<td>Range: 0 to 400 bar Accuracy: ±0.5 % FSO IEC 60770</td>
<td></td>
</tr>
<tr>
<td>Pressure</td>
<td>bar</td>
<td>Rosemount 3051 - CD2A22A1BS2B4</td>
<td>Range: 0 to 620 mbar Accuracy: ±0.04 % of span</td>
<td></td>
</tr>
<tr>
<td>Differential Pressure</td>
<td>mbar</td>
<td>YOKOGAWA RAMC - Metal stroke rotameter</td>
<td>Range: 0 to 7.5 l/d/h (custom calibration) Accuracy: class 1.6 for VDI/VDE 3513</td>
<td>Calibrated for crude oil at temp - 300 °C and 645 kg/m³</td>
</tr>
<tr>
<td>Current</td>
<td>Ampere</td>
<td>Power supply unit - Genesy 30-333 Genesy 20-500</td>
<td>Range: 0 to 666 A (Preheater) 0 to 1000 A (Main heater) Accuracy: Programming - ± 1 % Monitoring - ± 1.5 %</td>
<td>There are 2 similar units for each section which connected in parallel</td>
</tr>
<tr>
<td>Voltage</td>
<td>Volts</td>
<td>Power supply unit - Genesy 30-333 Genesy 20-500</td>
<td>Range: 0 to 30 V (Preheater) 0 to 20 V (Main heater) Accuracy: Programming - ± 1 % Monitoring - ± 1.5 %</td>
<td>There are 2 similar units for each section which connected in parallel</td>
</tr>
<tr>
<td>Level</td>
<td>%</td>
<td>Levelflex FMP45</td>
<td>Range: 0 to 100 % Accuracy: ±0.06 %</td>
<td></td>
</tr>
</tbody>
</table>

#### 2.4.2 Parameters accuracy and precision

The uncertainty of measurement can be divided into 2 categories; systematic errors and random error. ISO (2012) has elaborated further the definition of uncertainties and how it should be reported in experimental data. Nowadays, all instrument manufacturers will provide the value of uncertainties in their technical datasheet and all necessary tested has been done for verification purposes.

**2.4.2.1 Manufacturer data specifications**

Instruments used for temperature measurement in HiPOR are Type K thermocouple and variable area type flowmeter for flowrate with uncertainties of ±1.1 % of measured
temperature and ±1.6% of the measured flowrate for flowrate more than 50% of calibrated flowrate. Other instruments and their uncertainties have been listed in table 2.1.

Thermocouple has a unique principle of operation. It is acknowledged that the principle of operation is based on the finding by Thomas Johann Seebeck. The initial idea was when two wires with different electrical properties are joined together at one end, and other end uses for measuring between hot and cold (the difference in term of voltage), the value of voltage difference could be calibrated proportional with temperature increment as shown in figure 2.12.

![Figure 2.12 Working principle of thermocouple](image)

Figure 2.12 shows the working principle of thermocouple. In HiPOR, the tail end is connected directly to analogue input/output of DAQ board and the measurement of temperature can be read within LabView interface. For flowrate, type of flowmeter used is a variable area flowmeter. Flow measuring principle for this type shown in figure 2.13. The figure shows the influences that acted on the float for variable area flowmeter. The influences can be divided into 4 categories; buoyancy, flowrate, gravity and gas back pressure. In HiPOR environment, gas back pressure can be ignored since the process fluid is in liquid form. The buoyancy force is dependent on fluid density/viscosity and float density. The flowrate force is dependent on the changes or transitions of flow until it reaches a new equilibrium. The gravitational force is dependent on the mass of the float. This depends on the size and float material.

![Figure 2.13 Forces that acted on variable area type flowmeter](image)
Figure 2.14 Caliberation of variable flowmeter used VDI/VDE 3513 standard [ABB Instruments Manual]

Figure 2.14 shows the calibration of variable flowmeter used VDI/VDE 3513 standard. It is important to note that the measurements have different permissible errors and become constant at 50% or more when it measured in % of full scale. That means the selection of measurement range and span for respective instrument is important, especially the operating parameter during the experimental phase. In HiPOR, the flowmeter is calibrated at the lowest permissible error, 1.6% constant errors at ≥ 50% full scale measurement.

2.4.2.2 Calibration procedure

It is important to quantify the precision and accuracy of individual instrumentation for parameter measurement. In section 2.4.2.1, the details of instrumentation specification has been mentioned. The verification of this matter needs to be done in order to obtain reliable data for further analysis. Tatara and Lupia (2011) have developed an uncertainty checking procedure for temperature reading based on temporal and spatial effect and evaluate the confidence with the t - test distribution. The development of ISO standards (ISO 2012) to report on the uncertainties of measurement indicates the importance of this matter to any experiment/plant data acquisitions.

The calibration procedure commonly done in manufacturing environment and state precisely in the technical datasheet of a standard that is being used for this purpose. An experimental protocol for HiPOR primary measurement has been developed for validating important parameters namely thermocouple, flowmeter, pressure sensor, power supply.
Figure 2.15 Setup for calibration of thermocouple

Figure 2.15 shows the setup for calibration of thermocouple. The calibration was done to eliminate/minimise the systematic error generated by a signal which transmitted from thermocouple to LabView. Thus, the measurement will gives higher precision and reproducibility within a calibrated range.

Step determining thermocouple calibration formula
1. Thermocouple calibrator to simulate the desired temperature
2. Reading obtained from LabView where the initial expression should be y=x
3. Put all the reading inside the excel for data analysis

Analysis procedure
1. Develop a graph between the input signal and output signal; preferable as scatter data.
2. Add the trend line to see the correlation between the input signal and output signal. [Figure 2.16]
3. Try multiple correlations (exponential, linear, polynomial, power, etc.) and see which regression value near to a value equal to 1. It was tested the see whether it has linear or non-linear regression. [Figure 2.17]
4. The nearest correlation shall be put in the excel file.
5. Simulate the data with the original input and new correlation; obtain the value as the output
   a. This also called as a predicted value based on regression equation that initially developed by excel
6. Find the delta (difference) between input and output (original & simulate data)
7. Find median, variance, standard deviation, sum square for each and every delta value
8. Deduct the median for each individual delta value and calculate back the median, variance, standard deviation and sum square value.

9. Step no 2 is the expression for output signal [create deviation graph, input vs. delta] to show the deviation/dispersion with respect to input signal.

10. Median for step 7 is offset for respective thermocouple reading. It varies depending on the expression developed from the initial output signal.

**Equation 2.1**

\[ y = (\text{correlation expression}) + \text{median of total delta of measurement} \]

**Step identifying the correction of variable flowmeter**

It is very important to understand the manufacturer methods of calibrating the instrumentation and the used of recognised standard in order obtaining the mentioned value. This is useful information before any attempts of correction of flowrate can be made.

The calibration for different fluid can be made by inserting the variables into an equation.

**Equation 2.2**

\[ F_{\text{corrected}} = F_{\text{calibration}} \times \text{correction factor} \]

**Equation 2.3**

\[
\text{correction factor} = \sqrt{\frac{(SG_{\text{float}} - SG_{\text{new}}) \times SG_{\text{calibration}}}{(SG_{\text{float}} - SG_{\text{calibration}}) \times SG_{\text{new}}}}
\]

where \( SG_{\text{float}}, SG_{\text{new}}, SG_{\text{calibration}} \) are specific gravity of the float, crude oil and water as given in the technical equipment specification.

The correction factor shown in equation 2.3 needs to be calculated. Based on the value obtained for correction factor, it is inserted into equation 2.2 in order to obtain the corrected value for different types of fluid. The correction factor also can be extended into a different operating temperature of respective fluid. As stated earlier in table 2.2, this flowmeter was calibrated for crude oil at temperature 300 °C and density 645 kg/m³. Initially, HiPOR rig uses non-fouling oil for validation purposes, hence the correction factor for flowrate is very important. This factor is very useful to avoid false indication especially during start-up process when the temperature of the process fluid is still increasing before it reaches an equilibrium state or experiment phase.

**Equation 2.4**

\[
\text{correction factor} = \sqrt{\frac{T_{\text{actual}}}{T_{\text{calibration}}}}
\]
Equation 2-4 shows the correction factor for different temperature of the process oil. This correction factor only applies to crude oil which has the same properties as calibrated properties as mentioned earlier. If crude oil properties are different from the calibrated properties, the correction factor that uses specific gravity will be applied.

### 2.4.2.3 Results and discussion

#### 2.4.2.3.1 Thermocouple calibration

The tool has been used to simulate the desired temperature and reading is taken from LabView. Furthermore, the data should be analysis to find the correlation which commonly (reference to technical specification) linear. The average of the absolute error from this correlation will result the bias. Combine the correlation and bias into an equation for correction (calibration) of temperature input. It is also known as finding the corresponding curve-fit correlation.

![Figure 2.16](image.png)  
**Figure 2.16** Develop the correlation between input (simulator) and output (LabView reading) via Microsoft Excel

Figure 2-16 shows the example of development linear correlation between input and output signal. The development of correlation has been extended to power correlation as the value of regression for both correlations give more than 0.95. These trend correlations were done in Excel software as shown in table 2.2.

<table>
<thead>
<tr>
<th>Original DAQ T Correlation</th>
<th>New Linear Correlation</th>
<th>New Power Correlation</th>
</tr>
</thead>
<tbody>
<tr>
<td>$y=x$</td>
<td>$y=1.0003x-0.1578$</td>
<td>$0.9903x^{1.0018}$</td>
</tr>
</tbody>
</table>
Table 2.2 shows the correlations for linear and power expressions. These correlations, then being used to generate a new outlet data as shown in column 3 and 4 in table 2.3. The output data (calculated) were based on the input (from simulator) data which ranging from ambient temperature to 300 °C. The difference between the input and output data were calculated and shown in column 5,6 and 7 for all correlations displayed in table 2.2. All data in column 5,6,7 were plotted against input data and displayed in figure 2.17. These data will be used later to develop a coefficient for calibrated temperature measurement which increase its precision and reproducibility.

Figure 2-17 displays the dispersion of data of temperature different (delta T) between input and output signal for all correlations that listed in table 2.3. For linear correlation, the dispersion decreases as the temperature is increased. As power correlation is concerned, initially, it is increased as input temperature is increased, then it reaches a maximum value at input signal of 80 °C. Then, delta T starts to decrease until it reaches -0.15 °C. For y=x correlation, the dispersion is well distributed, unfortunately the standard deviation is relatively high compared to linear correlation. All statistical analysis for dispersion of data showed in figure 2.17 have been calculated and shown in table 2.4.

Table 2-5 shows statistical analysis for delta T value for DAQ, linear and power correlation. It shows that the sum of the square error for linear and power correlations gives smaller value compare to DAQ correlation. The median value is actually representing the offset value for calibration purposes. For variance and standard deviation, it shows that the linear correlation obtained the least value compare to DAQ and power correlation. Therefore, it could confirm that linear correlation is the best expression to be used for calibration of thermocouples. All of the correlations are corrected by adding median value (see equation 2.1), which represents the offset value, in order to finalise the calibration expression. The final expression for all thermocouple calibrations is shown in table 2.5. The graphs which previously plotted in figure 2.17, have been re-plotted with the new expressions and shown in figure 2.18.
Table 2.3 Data that acquired from input (simulated T) and output (DAQ T) and prediction value, delta T of predetermined correlation.

<table>
<thead>
<tr>
<th>Simulated T °C</th>
<th>DAQ T °C</th>
<th>New Linear Correlation °C</th>
<th>New Power Correlation °C</th>
<th>DAQ Delta T °C</th>
<th>Linear Delta T °C</th>
<th>Power Delta T °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>24.20</td>
<td>24.03</td>
<td>24.05</td>
<td>24.10</td>
<td>0.17</td>
<td>0.15</td>
<td>0.10</td>
</tr>
<tr>
<td>30.00</td>
<td>29.90</td>
<td>29.85</td>
<td>29.89</td>
<td>0.10</td>
<td>0.15</td>
<td>0.11</td>
</tr>
<tr>
<td>40.00</td>
<td>39.84</td>
<td>39.85</td>
<td>39.88</td>
<td>0.16</td>
<td>0.15</td>
<td>0.12</td>
</tr>
<tr>
<td>50.00</td>
<td>49.97</td>
<td>49.86</td>
<td>49.86</td>
<td>0.03</td>
<td>0.14</td>
<td>0.14</td>
</tr>
<tr>
<td>60.00</td>
<td>59.89</td>
<td>59.86</td>
<td>59.86</td>
<td>0.11</td>
<td>0.14</td>
<td>0.14</td>
</tr>
<tr>
<td>70.00</td>
<td>69.91</td>
<td>69.86</td>
<td>69.85</td>
<td>0.09</td>
<td>0.14</td>
<td>0.15</td>
</tr>
<tr>
<td>80.00</td>
<td>79.80</td>
<td>79.87</td>
<td>79.85</td>
<td>0.20</td>
<td>0.13</td>
<td>0.15</td>
</tr>
<tr>
<td>90.00</td>
<td>89.79</td>
<td>89.87</td>
<td>89.85</td>
<td>0.21</td>
<td>0.13</td>
<td>0.15</td>
</tr>
<tr>
<td>100.00</td>
<td>99.91</td>
<td>99.87</td>
<td>99.85</td>
<td>0.09</td>
<td>0.13</td>
<td>0.15</td>
</tr>
<tr>
<td>110.00</td>
<td>109.79</td>
<td>109.88</td>
<td>109.86</td>
<td>0.21</td>
<td>0.12</td>
<td>0.14</td>
</tr>
<tr>
<td>120.00</td>
<td>119.95</td>
<td>119.88</td>
<td>119.86</td>
<td>0.05</td>
<td>0.12</td>
<td>0.14</td>
</tr>
<tr>
<td>130.00</td>
<td>130.00</td>
<td>129.88</td>
<td>129.87</td>
<td>0.00</td>
<td>0.12</td>
<td>0.13</td>
</tr>
<tr>
<td>140.00</td>
<td>139.81</td>
<td>139.88</td>
<td>139.88</td>
<td>0.19</td>
<td>0.12</td>
<td>0.12</td>
</tr>
<tr>
<td>150.00</td>
<td>149.96</td>
<td>149.89</td>
<td>149.89</td>
<td>0.04</td>
<td>0.11</td>
<td>0.11</td>
</tr>
<tr>
<td>160.00</td>
<td>159.94</td>
<td>159.89</td>
<td>159.90</td>
<td>0.06</td>
<td>0.11</td>
<td>0.10</td>
</tr>
<tr>
<td>170.00</td>
<td>169.92</td>
<td>169.89</td>
<td>169.91</td>
<td>0.08</td>
<td>0.11</td>
<td>0.09</td>
</tr>
<tr>
<td>180.00</td>
<td>179.85</td>
<td>179.90</td>
<td>179.93</td>
<td>0.15</td>
<td>0.10</td>
<td>0.07</td>
</tr>
<tr>
<td>190.00</td>
<td>189.83</td>
<td>189.90</td>
<td>189.94</td>
<td>0.17</td>
<td>0.10</td>
<td>0.06</td>
</tr>
<tr>
<td>200.00</td>
<td>199.90</td>
<td>199.90</td>
<td>199.96</td>
<td>0.10</td>
<td>0.10</td>
<td>0.04</td>
</tr>
<tr>
<td>210.00</td>
<td>209.83</td>
<td>209.91</td>
<td>209.97</td>
<td>0.17</td>
<td>0.09</td>
<td>0.03</td>
</tr>
<tr>
<td>220.00</td>
<td>219.78</td>
<td>219.91</td>
<td>219.99</td>
<td>0.22</td>
<td>0.09</td>
<td>0.01</td>
</tr>
<tr>
<td>230.00</td>
<td>229.75</td>
<td>229.91</td>
<td>230.01</td>
<td>0.25</td>
<td>0.09</td>
<td>-0.01</td>
</tr>
<tr>
<td>240.00</td>
<td>239.86</td>
<td>239.91</td>
<td>240.03</td>
<td>0.14</td>
<td>0.09</td>
<td>-0.03</td>
</tr>
<tr>
<td>250.00</td>
<td>249.88</td>
<td>249.92</td>
<td>250.05</td>
<td>0.12</td>
<td>0.08</td>
<td>-0.05</td>
</tr>
<tr>
<td>260.00</td>
<td>260.05</td>
<td>259.92</td>
<td>260.07</td>
<td>-0.05</td>
<td>0.08</td>
<td>-0.07</td>
</tr>
<tr>
<td>270.00</td>
<td>269.91</td>
<td>269.92</td>
<td>270.09</td>
<td>0.09</td>
<td>0.08</td>
<td>-0.09</td>
</tr>
<tr>
<td>280.00</td>
<td>280.08</td>
<td>279.93</td>
<td>280.11</td>
<td>-0.08</td>
<td>0.07</td>
<td>-0.11</td>
</tr>
<tr>
<td>290.00</td>
<td>290.09</td>
<td>289.93</td>
<td>290.13</td>
<td>-0.09</td>
<td>0.07</td>
<td>-0.13</td>
</tr>
<tr>
<td>300.00</td>
<td>299.96</td>
<td>299.93</td>
<td>300.16</td>
<td>0.04</td>
<td>0.07</td>
<td>-0.16</td>
</tr>
</tbody>
</table>
Figure 2.17 Dispersion of data of temperature difference \(\delta T\) between input and output signal DAQ Delta T, Linear Delta T and Power Delta T

Table 2.4 Statistical analysis for delta T value for DAQ, Linear, Power correlation

<table>
<thead>
<tr>
<th></th>
<th>DAQ Delta T</th>
<th>Linear Delta T</th>
<th>Power Delta T</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sum square</td>
<td>0.534</td>
<td>0.3675142</td>
<td>0.34075</td>
</tr>
<tr>
<td>median</td>
<td>0.1041</td>
<td>0.1098</td>
<td>0.0544</td>
</tr>
<tr>
<td>variance</td>
<td>0.0078394</td>
<td>0.0006488</td>
<td>0.00911</td>
</tr>
<tr>
<td>Std. deviation</td>
<td>0.0885404</td>
<td>0.0254711</td>
<td>0.09543</td>
</tr>
</tbody>
</table>
Figure 2.18 Dispersion of data of temperature difference (\(\delta T\)) between input and output signal DAQ Delta T, Linear Delta T and Power Delta T with offset

Figure 2-18 illustrates the final dispersion of data of temperature different (\(\delta T\)) between input and output signal for all correlations with offset. It shows that the final expression of linear correlation give the least value of uncertainties, ± 0.05 °C. The other correlations have bigger uncertainties, power (-0.22/+0.10) °C and DAQ (-0.19/+0.15) °C. Based on this method of calibration together with statistical analysis data, it can confirm that the linear correlation is the best expression for calibration of thermocouples.

All thermocouples have been calibrated according to this method and the results is shown in table 2.5. Table 2.5 shows all thermocouples used for temperature measurements in HiPOR rig. The initial readings (used DAQ correlation) were represented the temperature different, delta T before any offset value being add/subtract from the initial value as shown in equation 2.1. As the results, the uncertainties still give a huge range of values. For linear correlation as described as a final expression in table 2.5, the uncertainties for all thermocouples give small value (up to ±0.08 °C). Therefore, this method provides reliable temperature measurement for crude oil fouling analysis.
### Table 2.5 Final expression that correlate between input and output for thermocouples in HiPOR.

<table>
<thead>
<tr>
<th>Thermocouple</th>
<th>Description</th>
<th>Initial Reading, ΔT</th>
<th>Expression (initial trial)</th>
<th>Precision °C</th>
<th>Expression (Final version)</th>
<th>Precision °C</th>
<th>Range °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1</td>
<td>Tank</td>
<td>-0.09/+0.25</td>
<td>( y = (x + 0.1) )</td>
<td>-0.19/+0.15</td>
<td>( y = ([1.0003x - 0.1578] + 0.1098) )</td>
<td>±0.04</td>
<td>0-300</td>
</tr>
<tr>
<td>T8</td>
<td>Test line return</td>
<td>+0.32 to +0.64</td>
<td>( y = (x + 0.45) )</td>
<td>-0.13/+0.19</td>
<td>( y = ([0.9996x - 0.3873] + 0.3934) )</td>
<td>±0.06</td>
<td>0-300</td>
</tr>
<tr>
<td>T9</td>
<td>Compabloc return</td>
<td>+0.24 to +0.55</td>
<td>( y = (x + 0.4) )</td>
<td>-0.15/+0.16</td>
<td>( y = ([0.9999x - 0.3774] + 0.4513) )</td>
<td>±0.02</td>
<td>0-300</td>
</tr>
<tr>
<td>T19</td>
<td>Cooling water return to the chiller</td>
<td>-0.17/+0.11</td>
<td>( y = (x) )</td>
<td>-0.17/+0.11</td>
<td>( y = ([1.0002x + 0.0341] - 0.0661) )</td>
<td>±0.03</td>
<td>0-300</td>
</tr>
<tr>
<td>T10</td>
<td>Paratherm Loop</td>
<td>±0.22</td>
<td>( y = (x) )</td>
<td>±0.22</td>
<td>( y = ([0.9996x + 0.0136] + 0.0504) )</td>
<td>±0.06</td>
<td>0-300</td>
</tr>
<tr>
<td>T18</td>
<td>Wall of preheater (middle)</td>
<td>-0.14/+0.16</td>
<td>( y = (x) )</td>
<td>-0.14/+0.16</td>
<td>( y = ([1.0003x - 0.0608] - 0.0022) )</td>
<td>±0.06</td>
<td>0-400</td>
</tr>
<tr>
<td>T17</td>
<td>Wall of preheater (downstream)</td>
<td>-0.12/+0.20</td>
<td>( y = (x) )</td>
<td>-0.12/+0.20</td>
<td>( y = ([1.0001x - 0.0206] - 0.0004) )</td>
<td>±0.02</td>
<td>0-400</td>
</tr>
<tr>
<td>T16</td>
<td>Pre-heater bulk</td>
<td>-0.08/+0.27</td>
<td>( y = (x + 0.1) )</td>
<td>-0.22/+0.13</td>
<td>( y = ([1.0001x - 0.1365] + 0.1205) )</td>
<td>±0.01</td>
<td>0-300</td>
</tr>
<tr>
<td>T5</td>
<td>Tube inlet</td>
<td>+0.20 to +0.54</td>
<td>( y = (x + 0.33) )</td>
<td>-0.11/+0.23</td>
<td>( y = ([1.0001x - 0.3355] + 0.3195) )</td>
<td>±0.01</td>
<td>0-300</td>
</tr>
<tr>
<td>T11</td>
<td>Wall Tube bottom (1/5)</td>
<td>+0.16 to +0.56</td>
<td>( y = (x + 0.33) )</td>
<td>-0.17/+0.23</td>
<td>( y = ([1.0004x - 0.4225] + 0.3385) )</td>
<td>±0.08</td>
<td>0-400</td>
</tr>
<tr>
<td>T21</td>
<td>Wall Tube bottom (2/5)</td>
<td>-0.33 to -0.03</td>
<td>( y = (x - 0.15) )</td>
<td>-0.19/+0.11</td>
<td>( y = ([1.0001x + 0.1534] - 0.1744) )</td>
<td>±0.02</td>
<td>0-400</td>
</tr>
<tr>
<td>T6</td>
<td>Wall Tube middle (3/5)</td>
<td>-0.42 to -0.04</td>
<td>( y = (x - 0.26) )</td>
<td>-0.15/+0.23</td>
<td>( y = ([1.0002x + 0.2072] - 0.2492) )</td>
<td>±0.04</td>
<td>0-400</td>
</tr>
<tr>
<td>T12</td>
<td>Wall Tube top (5/5)</td>
<td>-0.17/+0.13</td>
<td>( y = (x) )</td>
<td>-0.17/+0.13</td>
<td>( y = ([0.9998x + 0.0918] - 0.0498) )</td>
<td>±0.04</td>
<td>0-400</td>
</tr>
<tr>
<td>T20</td>
<td>Wall Tube top (5/5)</td>
<td>-0.07/+0.37</td>
<td>( y = (x + 0.11) )</td>
<td>-0.16/+0.28</td>
<td>( y = ([0.9999x - 0.0852] + 0.1062) )</td>
<td>±0.02</td>
<td>0-400</td>
</tr>
<tr>
<td>T7</td>
<td>Tube out</td>
<td>+0.06 to +0.36</td>
<td>( y = (x + 0.2) )</td>
<td>-0.14/+0.16</td>
<td>( y = ([1.0001x - 0.2052] + 0.1842) )</td>
<td>±0.02</td>
<td>0-300</td>
</tr>
</tbody>
</table>

# All thermocouples listed are installed in HiPOR (Refer to figure in Appendix A p.155)
2.4.2.3.2 Flowmeter calibration

In section 2.4.2.1 and 2.4.2.2, the explanation of principle in variable area flowmeter has been used for calibration purposes. Since the approved standard VDI/VDE 3513 has been used by the manufacturer and permissible errors have been supplied together with instrument technical specification, it would be much easier to calibrate the instrument. The calculation involves the technical specification which includes the permissible relative error and end value towards percent error shall be shown in details. Details steps of corrected flowrate as shown in figure 2.19.

Figure 2.19 Steps of determining the corrected flowrate for different types of oil used in HiPOR facilities

Figure 2.19 shows the steps in determining the corrected flowrate for different types of oil used for HiPOR rig. As previously discussed, the flowmeters in HiPOR rig were calibrated based on crude oil which properties shown in table 2.6. Equation 2.3 was used to calculate the correction factor for these purposes.

Table 2.6 Parameters used to calculate corrected flowrate

<table>
<thead>
<tr>
<th>medium</th>
<th>unit</th>
<th>float</th>
<th>water</th>
<th>crude oil</th>
<th>Paratherm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>oC</td>
<td></td>
<td></td>
<td></td>
<td>250</td>
</tr>
<tr>
<td>Pressure</td>
<td>bar</td>
<td></td>
<td></td>
<td></td>
<td>30</td>
</tr>
<tr>
<td>Density</td>
<td>kg/m³</td>
<td>7920</td>
<td>997.77</td>
<td>645</td>
<td>732</td>
</tr>
<tr>
<td>Viscosity</td>
<td>cP</td>
<td>0.9428</td>
<td>0.25</td>
<td></td>
<td>0.39</td>
</tr>
<tr>
<td>Range</td>
<td></td>
<td>570-5840</td>
<td>750-7500</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 2.6 displays the important parameters supplied by manufacturer that are used to calculate the correction factor, hence the corrected flowrate can be obtained by inserting the correction value from equation 2.3 into equation 2.2. Herewith, an example calculation for a measured flowrate, 1.6 m$^3$/h for Paratherm at temperature 250 °C.

\[
\text{correction factor} = \sqrt{\frac{(SG_{float} - SG_{new}) \cdot SG_{calibration}}{(SG_{float} - SG_{calibration}) \cdot SG_{new}}}
\]

\[
\text{correction factor} = \sqrt{\frac{(7920 - 732) \cdot 645}{(7920 - 645) \cdot 732}}
\]

\[
\text{correction factor} = 0.933065
\]

As the measured flowrate is 1.6 m$^3$/h, the corrected flowrate for Paratherm can be calculated by inserting calculated correction factor into equation 2.2.

\[
F_{\text{corrected}} = F_{\text{calibration}} \cdot \text{correction factor}
\]

\[
F_{\text{corrected}} = 1.6 \times 0.933065
\]

\[
F_{\text{corrected}} = 1.49 \text{ m}^3/\text{h}
\]

An experiment has been designed specifically to quantify the deviation of measured and corrected flowrate at a different inlet temperature of the test section, T5. This temperature, T5 is kept constant at the desired temperature and valve opening, V17 is varies from 10 % to 100 % opening at the step change of 10 %. The results of this experiment are shown in figure 2.20.

![Figure 2.20 Measured flowrate for different inlet temperature, T5](image-url)
Figure 2.20 shows the measured flowrate against the opening of the valve, V17 at an interval of 10%. The measured flowrate starts to deviate as valve opening increases. This should not be a case as the pump was used should provide a similar volumetric flowrate. As the flowrate was determined by a variable area flowmeter, the correction factor for measured flowrate shall be applied in order to obtain the correct flowrate for different types of oil and operating temperature as stated in the previous section. The corrected flowrates were calculated using equation 2.3 and 2.2 and the results is plotted in figure 2.21.

![Figure 2.21 The corrected flowrate after implementing the correction factor](image)

Figure 2.21 shows the corrected flowrate after implementing the correction factor [equation 2.3] in the previous section. It shows the corrected flowrate at different valve opening gives a close measurement with errors less than 0.05 m³/h. These results show the actual behaviour of pump capabilities as well as flowrate for respective flowrate.
Figure 2.22 The differences between measured flowrate and corrected flowrate at different inlet temperature

The dotted lines represent the corrected flowrate and the continuous points represent the measured flowrate as shown in Figure 2.22 shows the error between measured flowrate and corrected flowrate at different inlet temperature. The flowrate at 100 °C, 150 °C, 200 °C and 250 °C are 20 %, 15 %, 10 % and 5 % respectively. The errors were more significant when the oil temperature is low compared to high temperature. It happens as the flowmeter was calibrated for crude oil [0.25 cP and 300 °C]. This effect of non-corrected flowrate will be discussed further in chapter 4; baseline validation of non-fouling oil.

2.4.2.3.3 Differential Pressure

Differential pressure is one of the important parameters needs to be captured in this study. This variable would show the hydraulic effect of crude oil study. The reliability of this measurement is essential especially when validating with non-fouling oil and simulation results. Since the test section arrangement is in vertically installed, the hydrostatic pressure loss shall take into account apart from the friction losses. A type of differential pressure has been installed for HiPOR was Fisher Rosemount differential transmitter series CD2A which has a span between 0 to 620 mbar. The transmitter was calibrated with H₂O with a range of 0 to 250 inches at 4 °C.
2.5 Design of Control System, Commissioning and Testing

Designing a control system for a plant like HiPOR requires an extensive knowledge in a process and control engineering. It is very important to implement a control system in the HiPOR environment into selected process parameters in order to keep them constant from any external disturbances that might deviate them from the desired operating conditions. As mentioned earlier, HiPOR as pilot plant set-up, it is essential for the rig to incorporate a control system within their setup in order to replicate the actual conditions in industrial environment.

A control system requires several elements for acquisition, monitoring and controlling the parameters. These elements include instrumentations (thermocouple, flowmeters, etc.), software (LabView) and final elements (control valve, power supply unit). Each individual element will be connected in order to acquire, monitor and control the desired process. In HiPOR, the processes ranging from temperature, flowrate and heat input. A basic control system that commonly used in industry is illustrated as figure 2.23

![Figure 2.23 Common control system in chemical industry](image)

Figure 2.23 shows the general feedback control that has been widely used in industry. In HiPOR, the basis of the control system was based on a feedback control system. Each individual control system for HiPOR will be discussed further in the later section in this chapter. A basic control system will have 4 types of variables; setpoint, disturbance, manipulated and process variable. Setpoint is desired operating condition that needs to be controlled throughout the experiment. It is only useful if the controller is put in the automatic mode. The manipulated variable represents the value of controller output to the final elements. As a controller is trying to eliminate the error based on the differences between setpoint (desired) and process variable (measured), this manipulated variable is a given value that is obtained from a controller algorithm that need to transfer to final element. For example, the desired flowrate, setpoint is 1.2 m3/h and the measured flowrate is 1.15 m3/h. The controller will receive an error of -0.05 m3/h. It will send a signal (manipulated variable) to control valve (final element) to open the
valve further in order to eliminate the error. This is how a feedback control system works and
the response will be depending on the controller tuning of each and individual controller.

2.5.1 Objectives
In HiPOR setup, it has been identified that there are three important parameters need to be
controlled at any given time of experiment. They are inlet temperature, T5, flowrate to the test
section, F1 and F2 for annulus and tubular test section respectively, and heat input to the test
section. The control system is not only used for controlling the parameters, it can be extended
for safety purposes. This feature will be incorporated together in the HiPOR control system. It
created a safety interlock system to the process in order to avoid unforeseen circumstances or
incident if one of the elements that has been mentioned before is malfunction.

2.5.2 Control System for Inlet temperature
A control system for inlet temperature to test section, T5 has been implemented in HiPOR by
using LabView interface. A general feedback control system has been used as a basis for this
purpose. This control system is able to monitor and control the inlet temperature accurately at
deviation of ±0.5 °C.

2.5.2.1 Inlet temperature
The inlet temperature to the test section, T5 is one of the critical operating condition that needs
to be controlled precisely. The control of T5 was done by controlling the heat input via a
preheater section which installed after the pump. Preheater consists of two unit of power
supply, which connected in parallel, and capable to supply current up to 666 A and voltage up
to 30 V. The total power input can reaches up to 20 kW depending on resistance of preheater
tube.

2.5.2.1.1 Design philosophy
The design philosophy of temperature control for inlet temperature, TC01 based on a basic
feedback control as shown in figure 2.24. It has a feature to be controlled in manual and
automatic.
Figure 2.24 and 2.25 show a feedback control system and a block diagram which created within
LabView interface for inlet temperature, TC01 respectively. This control system can be
controlled in manual and automatic mode. In manual mode, current value (ranging 0 to 660 A)
will key-in manually by the operator via LabView. This signal is sent to power supply unit. The
increment of current input will result the increment of power input, therefore the wall
temperatures at preheater section, T17 and T18 increase. Both temperatures can be
monitored at the front panel of the monitoring system. As the wall temperature is increased, the
heat has been transferred from the wall to the oil inside the preheater tube. As a result, the
outlet bulk temperature of preheater, T16 increases as well. T5 is located downstream to the preheater section. The increment of T16 will instantaneously increase the T5.

Figure 2.24 Temperature control system, TC01 implements in HiPOR

![Temperature control system, TC01](image)

Figure 2.25 Block diagram of inlet temperature control system, TC01
As mentioned earlier, TC01 is designed to control T5 consistently. The concept of designing this system is to obtain a steady temperature by rejecting any disturbance during the experiment. In automatic mode, the desired value, setpoint of T5 is key into TC01. The value of setpoint is then compared to the actual value of T5, process variable inside the controller, TC01. Any discrepancies or errors which generated from this result, will be corrected using control algorithm that primarily set inside the controller itself. In HiPOR, the controller algorithm used is the convectional PID (Proportional, Integral, Derivative) control. This algorithm will generate the value of controller, manipulated variable and send it to final element. The final element, preheater (current) will be adjusted in order to achieve the desired setpoint, TC01. The loop of this process will be continued until the error between setpoint and process variable is eliminated. Therefore, it is important to understand the total process of the temperature behaviour before any attempts to design and tune the control algorithm. The safety features also have been included in order to avoid any incident happen. TC01 is tuned only to stabilise the temperature from any disturbances and it did not cover the ramp process for start-up or shutdown operation.

2.5.2.1.2 Experiment procedure

An experiment has been designed to commission, test and tune this TC01 control system. Amongst the objectives are;

- To test the functionality and reliability of safety features.
- To study the response when changing from a manual mode to automatic mode
- To study the response when TC01 is introduced to disturbance

There are three types of safety features/interlock system that have been installed to protect the preheater/ power supply unit from being operated. They are no flow signal, isolated valves, V3 and V22 are closed, and no RUN signal from main pump, M1. These signals are connected digitally in OR gate (see figure 2.25). If any of these signals is activated, the power supply unit will be automatically switch OFF. This test can be done in a dry experiment (computer based) and actual experiment (run at low temperature). The main idea of these interlock system is to prevent an enormous heat input to the static fluid. Since the volume inside the preheater section is relatively small, the increment of oil bulk temperature at this section will be increased exponentially. As the results, the thermal stress will happen to the pipe material and the gasket of the flanges, hence the unforeseen circumstance or disaster could happen in split seconds.

TC01 is only designed for rejecting the disturbances at the desired temperature. For future works, it may include the capabilities of ramping the temperature from a lower temperature to a higher temperature and vice versa. The main reason of second feature is not included, the disturbances for ramping the temperature will create conflict in controller algorithm. For
ramping the temperature, all algorithms (P, I, D) need to be used. At steady state, which will be discussed in this thesis, only P and I algorithm was used.

To control T5 at steady state, the tuning approaches is trial and error. Even though it seems to be easy, the guideline of tuning the controller is still followed. The overshoot value needs to keep within ± 1 % of desired setpoint, settling time shall be kept within 5 minutes and noise of process variable is within ±0.5 % of setpoint. The test had been made to test TC01 at setpoint 150 °C, 200 °C and 250 °C. The disturbance has been introduced by manipulating the flowrate (at an interval of 10 % valve opening) of the oil and the results will be analysed based on the set requirement.

2.5.2.1.3 Results and discussion

For safety features of TC01, the tests are run for all conditions in dry experiment and no flow condition in the actual experiment.

Figure 2.26 illustrates the response of TC01 when it changes from manual to automatic mode. . As a common practice, the temperature is changed from manual to automatic mode when the process variable has achieved the desired setpoint. From the time that the controller was put in auto mode, T5 keeps increasing until it reaches approximately 251.2 °C before the response is reversing. It is noted that the overshoot value is 1.2 °C from the setpoint value. This value represents approximately 0.5 % of the controlled value and it can be considered as excellent control for % of overshoot in temperature control system. The percentage of overshoot depending on the rate of oil temperature increment and the actual tank temperature, T1 at the time when TC01 is changed from manual to automatic mode

As mentioned earlier, the temperature control is designed to control inlet temperature, T5 at the desired operating temperature. Several tests of introducing disturbance by manipulating the flowrate, F2 via opening and closing valve, 17. The results were shown in Figure 2.27.
Figure 2.27 Response of TC01 for disturbance of flowrate

Figure 2.27 shows the response of TC01 towards the introduction of disturbance. The disturbance to this control system was flowrate. It shows the process variable, which is T5, starts to decrease when the disturbance is introduced. The main reason is the amount of heat input is supplied to control the temperature of the previous amount of flowrate. The decrement of flowrate will indicate that the heat input at preheater is overheat the oil as it can be shown that the temperature, T5 has slightly increased. When the control system, TC01 detected the error between the setpoint and process variable, the controller starts to react to eliminate the error. This can be seen that the final element starts to decrease accordingly until the error is eliminated. Hence, the temperature only fluctuates within ±0.1 °C from the desired temperature. In a crude oil fouling study, the main idea is to investigate the fouling when the joule heating is introduced at the test section.

2.5.3 Control System for Flowrate

A control system for flowrate to test section, F2 has been implemented in HiPOR by using LabView interface. A general feedback control system has been used as a basis for this purpose. Before any attempts of designing a flowrate control system, FC02, a control valve characteristic of this control system needs to be investigated. The main reasons are to understand the response of this final element and its limitation of control range, which will be explained later in this section. This control system is able to monitor and control the inlet temperature accurately at deviation of ±0.1 m³/h.

2.5.3.1 Control valve characteristic

Control valve characteristic is one of the tests of equipment for HiPOR. It verifies its functionality and technical specification provided by manufacturer.
Figure 2.28 Typical control valve characteristic is used in industries (Dunn, 2006)

Figure 2.28 shows typical control valve characteristic for a given valve opening and flowrate. The characteristic types can be divided into 4 categories, namely quick opening, linear, modified parabolic and equal percentage. It is important and very useful to verify the existing valves used in the laboratory especially for designing a control system. Testing has been done for all control valves in the HiPOR.

2.5.3.1.1 Experiment procedure
An experiment was designed carefully to study types of valve characteristic and its hysteresis for control valve, V17. Moreover, it also can be used to find a controllability range of control valve. This experiment was performed in the cold condition which no heating was supplied to the system during normal operation. Initially, the oil (Paratherm - non-fouling oil), was heated to 70 °C by the immersion heater, which located inside the main tank, as initial preparation for the start-up procedure. The main reason is to reduce the viscosity of the oil in order to overcome the limitation of motor current of the pump, M1. The start-up phase began as thoroughly explained in Appendix A from section 4.5.1.1 to 4.5.1.8. After the oil starts to circulate the rig via the tubular test section for 5 minutes, valve V17 was opened to 10 % opening. This valve opens with every 10 minutes interval and step change with a 10 % opening. The action is then reversed by closing the valve with the same interval time and step change process.

All results have been recorded and logged. The analysis of the data has been performed and analysed. Based on the results of the flowrate vs. valve opening, the valve characteristic can be determined and verified. It will be shown in section 2.5.3.3. On the other hand, another analysis could be made by using the same data is the control valve controllability range and hysteresis.
test. It is important to check the valve stem position error with the same opening but in the different valve's direction; opening and closing mode. The results will be shown in section 2.5.3.3. It will be determined whether the valve stem position errors are within an acceptable range or not.

2.5.3.1.2 Results and discussion

The experiment was done to verify the type of control valve which have been installed for HiPOR rig. As explained earlier in the experimental procedure, the results shown will be based on the response of the flowrate towards the increment and decrement of control valve opening in manual mode. The results then compare with the generic graph which was shown in figure 2.29.

Figure 2.29 shows control valve characteristic for V17. At 10 % of valve opening, the flowrate is representing approximately 40 % of the total flowrate. If compare to figure 2.29, control valve, V17 has a characteristic of quick opening. It also shows that at the valve opening between 80 - 100 %, the flowrate increment or decrement is less significant where the changes only between 0.05-0.1 m³/h or less than 5 % of the flowrate.
Figure 2.30 Control valve controllability range for V17 & RUN 217

Figure 2.30 displays control valve hysteresis for V17. It shows that the hysteresis is bigger at the valve opening less than 30% whilst at 80% valve opening, the flowrate has achieved the maximum. That means the control system for flowrate can be practically applied to the flowrate range at the valve opening between 30-70% in order to avoid large disturbance of noise during a smooth operation. This characteristic actually helps the control engineer to design and implement control strategy.

2.5.3.2 Flowrate

The flowrate to the test section, F2 is another critical operating condition that needs to be controlled precisely. The control of F2 was done by manipulating the control valve, V17 opening.

2.5.3.2.1 Design philosophy

The FC02 has manual and automatic mode. It can be used to monitor and control the flowrate into the tubular test section. It is a normal feedback control containing a sensor, controller and final element as shown in figure 2.32. PID control algorithm is used in the controller. It is important to control the flowrate of the oil automatically.
Figure 2.31 Flow control system, FC02 implemented in HiPOR

Figure 2.32 Block diagram for flowrate control at tubular test section, FC02

Figure 2.31 and Figure 2.32 show a feedback control system and a block diagram which created within LabView interface for flowrate to tubular test section, FC02 respectively. The similar control system has been designed and implemented for controlling the flowrate at annulus test section and cooling loop section. This control system can be controlled in manual mode and automatic mode. In manual mode, opening valve value (ranging 0 to 100%) will be key-in manually by the operator via LabView. This signal was sent to control valve, V17. The increment of V17 will result the increment flowrate, F2 at test section. Even though the flowmeter is designed for a span from 0 - 7.5 m³/h, unfortunately due to pump limitation, the flowrate could be increased up to 1.7 m³/h only.

As mentioned earlier, FC02 is designed to control flowrate, F2 consistently at any given temperature. The concept of designing this system is to obtain a consistent flowrate by rejecting any disturbance during the experiment. In automatic mode, the desired value, setpoint of F2 is keyed-in into FC02. The value of setpoint is then compared to the actual value of F2,
which is given by the flowmeter inside the controller, FC02. Any discrepancies or errors which generated from this result, will be corrected using control algorithm that primarily set inside the controller itself. In HiPOR, the controller algorithm used is the convectional PID (Proportional, Integral, Derivative) control. This algorithm will generate the value of controller, manipulated variable and send it to final element. The final element, control valve, V17 will be adjusted accordingly to achieve the desired setpoint, FC02. The loop of this process will be continued until the error between setpoint and process variable is eliminated. Therefore, it is important to understand the total process of the flowrate and final element behaviour before any attempts to design and tune the control algorithm. It is also noted that the flowrate process is a fast process and the noise that associated within the process is inevitable. For that reason, acceptable noise or fluctuations of flowrate needs to take into account before the tuning process begins.

2.5.3.2.2 Experimental procedure

The experiment that has been designed previously for control valve characteristic can be used to find the controller tuning. This experiment also known as an open loop test where the response of flowrate process will be calculated against the controller output. Despite finding the tuning parameters, another experiment was designed to test the controller tuning algorithm. The aims of the experiments are;

- To study the response when changing from a manual mode to automatic mode
- To study the response when FC02 is introduced with a disturbance

The changes of FC02 from manual mode to automatic mode was done when the measured flowrate, F2 is approximately equal to the desired setpoint. The response that was meant to study was the overshoot value, if any, and the fluctuation of process variable, F2. As informed earlier, the noise from the flowrate is inevitable and shall be in the acceptable range. In HiPOR, ±0.1 m³/h is considered an acceptable range of noise that generated from flowrate process.

Secondly, the aim is to study the response towards disturbances. There are two types of disturbance that will be introduced in this experiment. First, setpoint disturbance and second is external disturbance. The setpoint disturbance is the process where the setpoint is changed to a new desired setpoint. As the new setpoint is introduced, the capability of the control algorithm is tested to bring the process variable to the new setpoint from the previous set. The response time and overshoot value will be monitored closely.

Another disturbance is the external disturbance. In this scenario, the oil will be introduced into another loop, annulus test section loop, and decrement of flowrate, F2 is expected. The main idea of this test was to monitor the controller algorithm responding the error in order to bring
back the process variable, which is flowrate, F2 to the original desired setpoint. Both tests are very important to determine a correct control algorithm tuning parameter, PID for FC02.

2.5.3.2.3 Results and discussion

The experiment for control valve was used in this testing to find the important parameters that used for controller tuning. It is noted, the flow process is the process that follows the response of first order plus dead time, FODT. This is shown in figure 2.35.

![Figure 2.33 Experiment to find data for PID tuning](image)

Figure 2.33 displays the response of the open loop reaction rate of PID tuning method. The dead time, $t_d$ is defined as the duration for process variable, F2 movement when the step change is introduced to the controller output or final element, V17. In this case, V17 is increased from 10 % to 20 % the value of $t_d$ can be seen in Figure 2.33. Another important parameter is time constant. This time constant can be obtained by calculating the changes of the process variable, which has changed at 67.2 % of the amount of total F2 changed. All variables that have been mentioned earlier has been shown in table 2.7 and 2.8.

<table>
<thead>
<tr>
<th>Temperature, °C</th>
<th>Valve opening, %</th>
<th>Dead Time, s</th>
<th>PV change, m$^3$/h</th>
<th>67.2 % of PV change</th>
<th>Time constant, s</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>10-20</td>
<td>2</td>
<td>0.23</td>
<td>0.15</td>
<td>13</td>
</tr>
<tr>
<td>20-30</td>
<td>2</td>
<td>0.23</td>
<td>0.15</td>
<td>12</td>
<td></td>
</tr>
<tr>
<td>30-40</td>
<td>3</td>
<td>0.14</td>
<td>0.09</td>
<td>08</td>
<td></td>
</tr>
<tr>
<td>40-50</td>
<td>4</td>
<td>0.07</td>
<td>0.05</td>
<td>11</td>
<td></td>
</tr>
<tr>
<td>50-60</td>
<td>2</td>
<td>0.08</td>
<td>0.05</td>
<td>11</td>
<td></td>
</tr>
<tr>
<td>60-70</td>
<td>6</td>
<td>0.05</td>
<td>0.03</td>
<td>07</td>
<td></td>
</tr>
</tbody>
</table>
Table 2.8 Process parameter obtained from open loop test for FC02

<table>
<thead>
<tr>
<th>Oil Temperature (°C)</th>
<th>Valve opening, %</th>
<th>Process gain</th>
<th>P</th>
<th>I</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>10-20</td>
<td>1.30</td>
<td>5.00</td>
<td>0.11</td>
</tr>
<tr>
<td>20-30</td>
<td>1.30</td>
<td>3.08</td>
<td>0.17</td>
<td></td>
</tr>
<tr>
<td>30-40</td>
<td>0.79</td>
<td>5.06</td>
<td>0.11</td>
<td></td>
</tr>
<tr>
<td>40-50</td>
<td>0.40</td>
<td>6.95</td>
<td>0.22</td>
<td></td>
</tr>
<tr>
<td>50-60</td>
<td>0.45</td>
<td>12.17</td>
<td>0.11</td>
<td></td>
</tr>
<tr>
<td>60-70</td>
<td>0.28</td>
<td>4.13</td>
<td>0.33</td>
<td></td>
</tr>
</tbody>
</table>

Table 2.7 shows the values of P and I algorithm for FC02 based on open loop tuning for oil temperature of 200 °C. The other parameters for different oil temperatures are given in Appendix C. Table 2.8 shows that the process gains are amongst the highest value at lower the valve opening of the FC02. It is expected results as the control valve characteristic showed that the valve has a quick opening characteristic. The rest of process gains are decreasing as the valve opening is increasing. The analysis was done up to 70% of valve opening because the flowrate increment beyond this opening is less significant. As can be observed earlier in figure 2.21, the flowrate difference between 70% and 100 % opening is less than 0.15 m³/h. Therefore, the implication of control system, FC02 is less significant compared with the flowrate at the valve opening less than 70 %.

Figure 2.34 FC02 response when the controller is changed from manual to automatic mode

Figure 2.34 shows the response of the controller, FC02 when it changes from manual to automatic mode. The process variable, F2 is maintained at previous value together with final element, V17. It confirmed that if no disturbance is introduced, the flow control, FC will be
stable as desired. It also confirmed that there was noise associated with a flow process which is between ±0.04 m³/h and the value is within acceptable range.

Figure 2.35 displays the setpoint disturbance which was introduced to FC02. The setpoint was changed from 1.2 m³/h to 1.1 m³/h. The response time to achieve the new setpoint is approximately 15 s and overshoot value is almost negligible. Though, the controller algorithm used for this test was taken from the value that generated from open loop test, but the response time is generally slow but it is still within acceptable value. The most probable reason was the integral time, I is large, 0.11 s. Integral time is the time where it will reset the calculation of the error which generated between the process variable and the new setpoint.

2.5.4 Control System for Heat Input to test section

Heat transfer phenomena research commonly involves heat transfer between the sources to the process fluid. In crude oil refinery, the heat needs to be transferred from the source into the oil. Heat transfer mechanism primarily involves conduction and convection process. It is extremely important to study of the effect when heat source/input is considered in two conditions; uniform wall temperature and uniform heat flux. Both conditions give different effects to the process wall, especially its wall temperature when deposition of fouling starts to occur. Based on this fact, the rig has been designed with its control system to facilitate both situations.

2.5.4.1.1 Design philosophy

Figure 2.36 and Figure 2.37 show a feedback control system and a block diagram which created within LabView interface for constant heat flux, HFC02 respectively. This control system can be
controlled in manual mode and automatic mode. In manual mode, current value (ranging 0 to 1000 A) will be keyed-in manually by the operator via LabView. This signal was sent to power supply unit of main heater, which is connected via copper clamps at both ends of the test section. The increment of current input will result in the increment of power input, therefore the heat flux supplied to the test section will be increased as well. This increment can be seen as the wall temperatures at the outer of the test section and the outlet bulk temperature of the test section, T7. All temperatures can be monitored at the front panel of the monitoring system as well as power supply unit parameters such as current and voltage. The power and heat flux values are the calculated values which are based on the primary measured value and physical plant data like the surface area of heat transfer. The safety features also have been included in order to avoid any incident happen similar to the temperature control system, TC01. In automatic mode, HFC02 is designed to maintain the heat flux at the desired setpoint. This can be done by manipulating the current input at the power supply unit as shown in Figure 2.36 and Figure 2.37.

**Uniform Heat Flux (UHF)**

![Block diagram for heat flux control for tubular test section, HFC02](image)
Uniform Wall Temperature (UWT)

A constant wall temperature control system is one of control features that is designed for heat input at tubular test section as shown in Figure 2.38. In manual mode, the mode of operation is similar to HFC02 which only current input is keyed in into the control system. In automatic mode, the desired wall temperature setpoint will be keyed into the controller and the wall temperature, which is located at the middle of the test section. T6 is used as a process variable. The concepts and working principle of this constant wall temperature control, TC03 is similar to TC01. The safety features are also included in this control system as well. Even though, the heat input control systems are designed for constant wall temperature and constant heat flux, unfortunately these designs cannot be switched between one to another during the experiment run.

2.6 Conclusions

HiPOR has been successful commissioned within the design of operating conditions. A comprehensive engineering approach had been implemented in order to develop a high
standard operating condition, SOP. All documentations include the operation of the rig, training and administration procedures. All important primary measurements had been calibrated with highest precision, Temperature (± 0.01 °C) and Flowrate (± 0.04 m³/h) within the designed range of operating conditions of the rig. A new control systems have been designed and implemented to control the desired operating conditions according to the objectives of the individual control. They are tested and the outcomes were excellent for all experiments which are discussed in chapter 4 and 5.
3 Modelling of the tubular test section

3.1 Introduction

In this chapter a model for the description of the test section in HiPOR is presented. The model builds on the tube model undergoing crude oil fouling by Coletti (2010) (also see Coletti and Macchietto (2011), Coletti et al., 2010 for further details and applications), currently property of Hexxcell Ltd. (Hexxcell Ltd., 2015). The model aims to emulate not only the setup of the rig in term of geometry but also to give a good description of mass and energy transport phenomena. Furthermore, the model must be able to capture the thermal and hydraulic effect of experiment either using non-fouling oil for baseline validation or crude oil for chemical fouling study. It is essential to understand the real setup of HiPOR and consider all external disturbances involved in the experimental setup which include the heat losses toward atmosphere, insulation effect and resistivity of tube metal as function of temperature.

3.1.1 Background: development of mathematical model

Most crude oil fouling models are based in the work by Kern & Seaton (1959), who proposed to describe fouling as the difference between a deposition and a removal mechanism. Fouling models typically attempt to describe deposition based on its thermal effect on the heat transfer surface. Assumptions commonly used in crude oil fouling modelling in heat exchangers are shown in Figure 3-1.

In more than 50 years, development of fouling models has tried to overcome some of those assumptions. Although some of the models have better prediction on data of the literature together with statistical analysis, unfortunately, the description of chemical fouling formation is still in the dark. To date, no model could describe precisely mechanism of chemical fouling formation especially during initialisation process. Most of the findings have assumed that the fouling resistance undergone initialisation process where the fouling resistance was negative until certain period of time, then it will increases linearly/exponentially until it reached the saturated point (asymptotic). The main idea of developing a model for HiPOR, is not only adapting the latest model available, but to overcome most of the limitations that list in figure 3-
Modelling of HiPOR Test Section  

1 and has been validated against experimental and/or industrial plant data. Based on this requirement, a multi scale dynamic and distributed model developed by Coletti (2010) has been used as a basis in this work. The advantages of this model are spatial-temporal effect of thermal and hydraulic over axial, radial of test section, ageing on deposit of fouling, and have an excellent validation against industrial plant data.

<table>
<thead>
<tr>
<th>Assumption</th>
<th>Why used</th>
<th>Limitations</th>
</tr>
</thead>
<tbody>
<tr>
<td>No temperature variations across the HEX</td>
<td>Not integrated with a thermal model</td>
<td>Errors in the calculations of heat transfer coefficients</td>
</tr>
<tr>
<td>Constant physical properties</td>
<td>Variability of crudes compositions</td>
<td>Affects the calculations of the convective heat transfer coefficients and overall heat balance</td>
</tr>
<tr>
<td>Constant heat transfer coefficient</td>
<td>Temperature distribution is not known</td>
<td>Incorrect temperature driving forces being used</td>
</tr>
<tr>
<td>No localized fouling</td>
<td>Temperature distribution is not known</td>
<td>Fouling process not fully described. Affects calculations of the convective heat transfer coefficients</td>
</tr>
<tr>
<td>No shell-side fouling</td>
<td>Removes the need for shell-side calculations</td>
<td>Only valid if tube-side fouling is dominating</td>
</tr>
<tr>
<td>Heat of reaction is negligible</td>
<td>Detailed reaction kinetics not known</td>
<td>Valid in most cases</td>
</tr>
<tr>
<td>Ageing effects neglected</td>
<td>No validated ageing models exists, kinetics of ageing reaction not yet fully understood</td>
<td>Spatial-temporal variation in thermal conductivity is not captured</td>
</tr>
</tbody>
</table>

Figure 3.1 Summary of assumptions commonly made in crude oil fouling model (Coletti, 2010)

3.1.1.1 Background; pro and contra of different model

This review of this report is to understand the previous and current research related to scientific objectives that had been identified. Although, chemical fouling could happen in any types of heat exchanger, but in this study, the focus is on heat exchanger with or associate with tube. It is important to fully understand transport phenomena issue related to this type of heat exchange.

In current practice, designing of heat exchanger or heat exchanger network are done by making a lot of assumptions. These are log mean temperature, steady state, constant physical properties and etc. This will actually contribute towards unknown fouling deposition with no appropriate parameters measurement to be considered. As a result, researchers and industrial players are still exploring the possibility to understand further science of chemical fouling threshold and able to develop robust model prediction control. Therefore, it will increase the equipment availability and keep the operation smooth.
3.1.1.2 Justification of choosing the dynamic, distributed model

Mathematical model is synonym with describing chemical engineering process. The model can be designed to explain scientifically what had happened in macro and/or micro level of equipment or process itself. It has been applied to many types of processes namely reaction, heat transfer, separation, etc.

Several mathematical models on heat transfer have been developed and used to evaluate heat transfer coefficient in heat exchanger. Coletti (2010) has made some review on this study and found out the study concentrated on tube side model and none of study has element of distributed model in their model. Distributed means the calculation of parameters such as temperature, heat transfer coefficient and other depends on 'local' parameter as function of spatial and time. Therefore, the fouling threshold could be scientifically explained with primary measurements of parameters and characterisation of fouling from analysis of sample.

A single tube dynamic and distributed tube model has been successfully developed by Coletti (2010) and segregate them in several domain. This model has taken into account ageing and surface roughness effect which is thoroughly explained later stage of this review. Based on this model, an extended model will be developed to describe the fouling of pilot plant (HIPOR) which has two types of test sections namely tube section and annulus section. Even tough, the test sections represent only one third of actual tube length in industry but it is to use primary measurement to further understand fouling phenomena in industrial application.

3.1.1.3 Correlations for tube-side heat transfer coefficient, friction factors and fouling rates

The main issues for the heat exchanger or heat exchanger network are heat transfer efficiency and high pressure drop. It is crucial to understand concisely the reason of these thermal and hydraulic effects to this equipment. As reviewed by Coletti (2010), crude oil fouling models consider lumped and discrete environment and assume constant physical properties and neglect the effect of local parameters in their calculation. It is understood that during those years, there is limitation of software and processing machine capabilities.

The basis of the model has been made available by Coletti (2010) by taking into consideration dynamic thermal and hydraulic model for single tube in the heat exchanger. In the distributed model, physical properties, tube-side heat transfer coefficient, friction factor and fouling rates, among other variables, are calculated as a function of local conditions. In this section, a survey on available empirical correlations is provided. The correct choice of these correlations is crucial to accurately simulate the thermal and hydraulic responses in HiPOR.

The heat transfer from the tube (or deposit) surface to the fluid is determined by the driving force (temperature difference) and the heat transfer coefficient. The calculation of heat transfer
Modelling of HiPOR Test Section

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coefficient is performed by using correlations for the Nusselt number in circular ducts. Table 3.1 shows different Nusselt number expression used for turbulent flow in tube, which is the main flow regime expected during normal operation in HiPOR. In this study, the Dittus-Boelter equation has been chosen in mathematic model with the option of other expression included as part of selector for this correlation. Therefore, a comparative study can be made with different types of heat transfer coefficient expression (Holman 2001).

### Table 3.1 Nusselt Number for Turbulent Flow in Tubes

<table>
<thead>
<tr>
<th>Authors/ Equation</th>
<th>Nusselt Number</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Saleh et al., (2005)</td>
<td>$Nu = \left( \frac{f}{8} \right) \frac{(Re - 1000)Pr}{1 + 12.7 \left( \frac{f}{8} \right)^2 \left( Pr \frac{2}{3} - 1 \right)} \left( 1 + \left( \frac{D}{L} \right)^{2/3} \right) X \left( \frac{\mu_b}{\mu_w} \right)^{0.14}$</td>
<td>$f = (1.82 \log Re - 1.64)^{-2}$ Use for transition region in constant heat flux condition</td>
</tr>
<tr>
<td>Nu = $\frac{U_0D_h}{\lambda}$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Petukhov Equation</td>
<td>$Nu = 0.125 f Re Pr^{1/3}$</td>
<td>$f = (0.790 \ln Re - 1.64)^{-2}$ 3000 &lt; $Re$ &lt; $5 \times 10^6$ applies to turbulent flow in the smooth tube</td>
</tr>
<tr>
<td>Shah et al., (1988)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Colburn Equation</td>
<td>$Nu = 0.023 Re^{0.8} Pr^{1/3}$</td>
<td>$f = 0.184 Re^{-0.2}$ can be used for smooth and rough tubes</td>
</tr>
<tr>
<td>Shah et al., (1988)</td>
<td>$Re &gt; 10000$</td>
<td></td>
</tr>
<tr>
<td>Dittus – Boelter Equation</td>
<td>$Nu = 0.023 Re^{0.8} Pr^n$</td>
<td>differentiate between heating and cooling application in heat transfer</td>
</tr>
<tr>
<td>n=0.4 (heating), n= 0.3 (cooling)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Incropera et al., (2007)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sieder – Tate equation</td>
<td>$Nu = 0.027 Re^{0.8} Pr^{1/3} \left( \frac{\mu_b}{\mu_s} \right)^{0.14}$</td>
<td>The equation will give errors up to 25%.</td>
</tr>
<tr>
<td>Incropera et al., (2000)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$0.7 &lt; Pr &lt; 16 700$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$Re &gt; 10000$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$\frac{L}{D} \gg 10$</td>
<td></td>
</tr>
<tr>
<td>Second Petukhov Equation</td>
<td>$Nu = \left( \frac{f}{8} \right) RePr$</td>
<td>The refine and complex equations shown below can reduce the error less or below than 10%.*</td>
</tr>
<tr>
<td>Janna, (2009)</td>
<td>$1.07 + 12.7 \left( \frac{f}{8} \right)^{1/2} (Pr^{2/3} - 1)$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$0.5 &lt; Pr &lt; 2000$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$10^4 &lt; Re &lt; 5 \times 10^6$</td>
<td></td>
</tr>
<tr>
<td>Gnieliski Equation</td>
<td>$Nu = \left( \frac{f}{8} \right) \frac{(Re - 1000)Pr}{1 + 12.7 \left( \frac{f}{8} \right)^{1/2} (Pr^{2/3} - 1)}$</td>
<td>Properties referred to bulk mean temperature.*</td>
</tr>
<tr>
<td>Incropera et al., (2007)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$0.5 &lt; Pr &lt; 2000$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$3 \times 10^3 &lt; Re &lt; 5 \times 10^6$</td>
<td></td>
</tr>
</tbody>
</table>

*American expression of friction factor is $\lambda = 4f$
Hydraulic effect is associated with pressure drop. It might be easily identified for different types of flow region. The main issues are the friction factors associated with the fluid region and surface roughness could easily drift the results away from the actual performance. This could happen if a distributed and dynamic model which local parameters are much more important to describe the behaviour of the fouling phenomena. Pressure drop due to friction is calculated by using empirical correlations for the estimation of the friction factor. Among the existing correlations for turbulent flow in circular ducts, three correlations are considered in this thesis (see table below). A comparison of model prediction and experimental results by using each of these correlations is provided in Chapters 4 and 5. These correlations have been chosen for coherence with previous works (Pental, 2011; Coletti 2010).

Table 3.2. Friction Factors

<table>
<thead>
<tr>
<th>Authors</th>
<th>Fouling Model</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wilkes (2005)</td>
<td>[ f = \frac{1}{\sqrt{f}} = -1.737ln \left[ 0.269 \frac{e}{R_{flow}} \right. ] [ - \frac{2.185}{Re} \ln \left( 0.269 \frac{e}{R_{flow}} + \frac{14.5}{Re} \right) ]</td>
<td>Turbulent Regime. Referred to as Friction Factor 1 in this thesis.</td>
</tr>
<tr>
<td>Saunders (1988)</td>
<td>[ f = 0.0035 + 0.264Re^{-0.42} ]</td>
<td>Slightly corroded tube (e = 0.046 mm). Turbulent regime. Referred to as Friction Factor 2 in this thesis.</td>
</tr>
<tr>
<td>Taitel and Duckler</td>
<td>[ f = 0.046Re^{-0.2} ]</td>
<td>Turbulent regime. Referred to as Friction Factor 3 in this thesis.</td>
</tr>
</tbody>
</table>

Fouling model with ageing model has taken into consideration of several factors that affect heat transfer coefficient. This study will concentrate on chemical fouling in crude oil system. Even though Ebert-Panchal model is a most accepted model for crude oil fouling, there are several other fouling models with other process conditions can take into consideration. Study on different fouling models and statistical analysis on average relative error has been made (Nasr and Givi 2006). The main argument, the result is not consistent on the variation of process conditions. Table 3.3 shows the development of rate of fouling for different heat transfer application primarily in crude oil. The main differences between them are the selection between film temperature and wall temperature and rate of suppression expression associated with empirical flow expression of dimensionless number.
Table 3.3. Development of Rate of Fouling for heat transfer application in crude oil

<table>
<thead>
<tr>
<th>Authors</th>
<th>Fouling Model</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Saleh et al (2005)</td>
<td>( \frac{dR_f}{dt} = ab^\beta \omega^\gamma \exp \left( - \frac{E}{RT_f} \right) )</td>
<td>Constant heat flux. Transition region.</td>
</tr>
<tr>
<td>Ebert &amp; Panchal (1995)</td>
<td>( \frac{dR_f}{dt} = a Re^\theta \exp \left( - \frac{E}{RT_f} \right) - \gamma \tau )</td>
<td>Deposition and suppression function. Chemical reaction &amp; film temp. vs. Tube shear stress and fluid velocity.</td>
</tr>
<tr>
<td>Panchal et al. (1997)</td>
<td>( \frac{dR_f}{dt} = aRe^\theta Pr^{-0.33} \exp \left( - \frac{E}{RT_f} \right) - \gamma \tau )</td>
<td>Mechanistic understanding. Correlation on Prandtl number may deviate parameter numerically.</td>
</tr>
<tr>
<td>Polley et al. (2002)</td>
<td>( \frac{dR_f}{dt} = aRe^{-0.8} Pr^{-0.33} \exp \left( - \frac{E}{RT_w} \right) - \gamma Re^{0.8} )</td>
<td>Developed to validate Knudsen's experiment data. Use wall temperature instead. Re - calculate the constant values.</td>
</tr>
<tr>
<td>Nasr and Givi (2006)</td>
<td>( \frac{dR_f}{dt} = aRe^\theta \exp \left( - \frac{E}{RT_f} \right) - \gamma Re^{0.4} )</td>
<td>Independent from Pr number. Re - calculate the constant value on the graphical method.</td>
</tr>
</tbody>
</table>

### 3.1.1.4 Physical properties calculation

Original model physical properties calculation is based on API crude oil relationship and standard chemical and physical properties for non-fouling oil. The calculation of physical properties is based on a function of other main parameters such temperature and pressure which normally can be measured by instrumentation. These can be obtained by looking to other methods such as SAFT, UNIFAC, API, DIPPR, RACKET and etc. The usage of those equations depends on value of other constants associated with them at the process conditions.

In mathematical model, concise and accurate expression and correlation are relatively important to develop a robust predictive model which is able to validate with different types of oil characteristic. A clean experiment with Paratherm will use the physical properties given by its manufacturer in their material safety data sheet (MSDS).

### 3.1.1.5 Aging and Surface roughness

It is relatively important for dynamic and distributed model for chemical fouling in tube system to include ageing and surface roughness. Most of rate of fouling expressions has expression of fouling formation and rate of suppression. As fouling formation is concerned, it has a tendency
to form different layer of deposits ranging from gel type deposit to solid type coke deposit as illustrated in Figure 3.2. Ishiyama et al. (2011) developed several model for aging process for different operating conditions. In their study, aging will be divided into three categories; two layer model (Zeroth order and first order) and distributed model. Each category will be tested under two different operating, UHF and UWT. They have found that the distributed model only can be applied at UHF operation and not in UWT.

![Diagram of deposit changes](image)

Figure 3.2 Schematic diagram of deposit changes which describe the formation, its initial soft layer (gel type) in (a) harder type where aged form as layer grows (b, c). Darkness of shading (c) shows the degree of ageing. (Ishiyama et al. 2011)

This model represents more likely lumped system and discrete and more likely is not suitable to be used for curvature design. Coletti et al. (2010) reported deposit aging gives significant impact on thermal fouling whilst the increment of surface roughness gives an opposite impact. The study has taken into consideration several factors which includes:

- Introduction of suppression term in the model
- Moving boundary problem in cylindrical coordinate
- Layer growth dependent on fouling rate
- Distributed over length and radial coordinate
- Model proposed for roughness dynamics
- Single tube

The rate of suppression expression includes the shear stress factor. As mentioned in previous friction factor to calculate pressure drop, it is important to include the value of surface roughness. Different type of deposit layer will contribute towards different value of surface roughness, hence gives different local value of parameters for thermal and hydraulic effects.
Hewitt et al., (1994) had reported an overview of the fouling phenomenon which can affect the overall heat transfer coefficient for heat transfer process. Amongst points have been addressed are;

- Reduction due to thermal resistance of fouling layer
- Increase due to an increased local velocity
- Change in roughness of heat transfer surface

### 3.1.1.6 Prediction capabilities/Statistical tools

Process Modeller gPROMS environment (PSE 2015) has two powerful statistical tools embedded within. They are parameter estimation and optimisation. These tools can lead into a good experiment design (Franceschini & Macchietto 2008a; Franceschini & Macchietto 2008b); hence an excellent predictive model can be built within this environment.

These statistical tools (parameter estimation and optimisation) played an essential role in validating plant and experimental data. There are few issues need to be resolve before any attempts of using these statistical tools. For instance, in parameter estimation, the understanding of model development and measured variables are relatively crucial in order to eliminate any uncertainties ranging from model structure, measurement errors to model discrimination (if used more than one model).

The development of the model started with the first fundamental chemical principle of chemical engineering which involves mass and energy balance. It is relatively important to understanding the type of process involved for modelling purposes. Mathematical model can be as simple as lumped and algebraic and as complex as distributed, dynamics with differential equation. The parameter estimation in the gPROMS environment has covered the following quite extensively which are commonly discussed in chemical engineering application (Englezos & Kalogerakis 2000):

- Structure of the model or model identifiable
- Objective function
- Solution technique
- Statistical properties of parameter estimations
- Statistical properties of model based calculated value
- Tests for model adequacy
- Tests for model discrimination

As mentioned earlier, model identifiable based on the first principle will indicate the complexity of process by increasing the gathered information. The selection of objective function and statistical tools associated with it which resulted projection differences of the process. This estimation can be divided into two categories; explicit function and implicit function. The best
algorithm depends on availability of information of measured variable and their instrumentation variance.

In many circumstances, a little information is made known about uncertainties of measurement. It is important to identify statistical variance model which has been incorporated within gPROMS. There are three different types of variance model in gPROMS:

- Constant variance; error do not depend on magnitude of measurement
- Constant relative variance; errors are proportional to magnitude of measurement
- Heteroscedastic; general type of variance expression

The general expression of these variance models is shown in equation (3.1);

\[ \sigma^2 = \omega^2 (\bar{z}^2 + \epsilon) \gamma \]

where the value \( \gamma = 0 \) and \( \gamma = 1 \) are for constant variance and constant relative variance respectively.

### 3.1.1.7 Sensitivity analysis

Sensitive analysis normally classified as steps in estimation environment after it has passed the structure and model identifiable process. It also tries to estimate continuous variable which can't be measured in the experiment. This method also can discriminate types of variables which gives significant influence to the performance of the process. It can then identify the most influential parameters which contribute substantially towards output variation. This can be done qualitatively by plotting input vs. output and quantitatively by calculation of correlation coefficient and regression analysis. Uncertainty and sensitivity analysis are the key point of the successful of parameter sensitivity. This process is important to identify in crude oil fouling where several physical properties of oil and operating parameter will be evaluated to see their contributions towards fouling deposition occurrence.

### 3.1.1.8 Model validation/prediction

Model validation in gPROMS environment will involve parameter estimation. In common statistical approach, it is also known as data fitting process. It involves a process of experiment design, experiment execution and statistical analysis. Initially, simulation of model will use literature or previous experimental values to be compared to current experimental values. Discussion will be specifically on the inclusion of experiment design which can generate ‘best’ model selectivity with ‘best’ parameter through the optimisation of objective function based on criterion of optimality. The common criterions used for this process are\(^1\);

\(^1\) gPROMS manual
• A-optimality; minimise the average variances or traces of the parameter estimates.
• D-optimality; minimise the generalised variances and the determinant of variance-covariance matrix
• E-optimality; minimise the largest variance eigenvalue of the variance-covariance matrix

The parameters to be estimated not only to the process parameters (physical properties and/or operating conditions) in the experiment but it can be extended to variance model and physical properties of plant (tube thermal conductivity, resistivity of heater and etc.).

3.1.1.9 CFD coupling with mathematical model

Hybrid modelling in gPROMS environment has been successfully applied for tubular reactor. This method is a combination of computational fluid dynamics (CFD) and mathematic model. Brahim et al. (2003) run a model based on experimental result to understand further the fouling phenomena occurring during crystallisation fouling and found out how important the local physical parameters are which contribute towards this. Strandstrom et al. (2007) discussed the involvement of deposition and removal of ash operation by coupling the model with quantitative parameters obtained from CFD simulation. Stehlik used CFD tool to improve a process of waste to energy conversion and mentioned about integration of heat transfer and heat exchanger for this application (Stehlik 2007b; Stehlik 2007a). Master et al. (2006) discussed on insert of baffle types to increase heat transfer coefficient by reducing fouling at shell side for power plant application.

The discussion of this concept will be extended once the model equation for both test sections has been successfully developed and validated or part of future works that need to be explored.

3.1.1.10 Monitoring process

 Fouling leads to a gradual decrease in thermal and hydraulic performance in pre-heat train heat exchangers in refineries. Thermal problem relates to the decrease of the overall heat transfer coefficient. The hydraulic problem corresponds to an excessive pressure drop as the flow area is reduced by the deposit. Both problems contribute to a substantial cost in operation, capital as well as environmental issues.

Monitoring of heat exchangers hence involves measurement of primary variables that are affected by thermal and hydraulic effects of the fouling phenomenon. These measured variables give indication on the severity of fouling and can be potentially used to characterise and study the relationship between fouling rates and operating conditions.

In the case of HiPOR, bulk and wall temperatures are used to monitor the thermal effects, and pressure drop as an indication of the hydraulic effects. In order to understand the thermal and hydraulic processes under “real” clean conditions, it is important to, initially, perform
experiments with non-fouling oil. This should provide a base-line to eliminate external factors other than oil physical properties. With crude oil, deviations in thermal and hydraulic performance with respect expected behaviour in clean conditions will indicate the formation of fouling deposits and enable the study of fouling rates.

### 3.1.1.11 Control and mitigation

It is important to be able to control the overall process or operational of heat exchanger or their network to fulfil the effectiveness and keep the operational in an optimum condition. There are also studies which have been made specifically on cleaning or scheduling, economic and environmental impact, design modification i.e. insert at tube side, type of baffle, process modification via threshold conditions, chemical injection, crude oil blending, operating parameters and model predictive control or online monitoring, real time optimisation.

Relevant literature on the control and mitigation of heat exchanger or networks is not limited to crude oil fouling but it has been extended to other chemical processes as well. Not all fouling can be eliminated by chemical additives especially severe fouling problems. An integral approaches to deal with it include optimum operating conditions and schedules (Müller-Steinhagen 2000). There is fouling mitigation via design for plate heat exchanger (Thonon and Grillot 1999), food application (Balasubramanian and Puri 2008). A mitigation study via management of cleaning in refinery (Wilson & Vassiliadis 1997). Smaili et al., (2001) discussed on MINLP approached and data reconciliation in fouling evaluation and (Yeap et al., 2004; Yeap et al., 2005) had been looking into the retrofit based on thermo-hydraulic fouling model.


### 3.2 Model for Tubular Test Section

The HIPOR pilot plant comprises two test sections, tubular section and annulus section.

The main purposes of this research are to validate mathematical model of HIPOR plant for the tube test section. A dynamic, distributed model for a single heat exchanger tube undergoing crude oil fouling developed by Coletti (2010) is used as basis for the development of the HiPOR tube. The model is modified to include the insulation layer, losses to the environment, and modification of the tube wall model and inclusion of a Joule heating model.
The test tube model for HIPOR plant can be divided into several sub-models or domains (Figure 3.3):

- Θ_T: Inside tube where the fluid flow through
- Θ_A: Deposit layer which determine the ageing of deposit
- Θ_W: Tube wall of the cylinder piping
- Θ_H: Heat input through Joule Heating
- Θ_I: Insulation layer which contain material as insulator

![Figure 3.3 Schematic diagram of 5 main domains of test tube in HIPOR experiment](image)

The heat balances were developed based on equation 3.2 of energy balance over control volume as illustrated at figure 3.3.

**Equation 3.2**

\[
\left\{ \text{rate of energy accumulation in control volume} \right\}_1 = \left\{ \text{net energy transfer by fluid flow} \right\}_2 - \left\{ \text{net energy transfer by conduction} \right\}_3 + \left\{ \text{rate of internal heat generation} \right\}_4 - \left\{ \text{net work transfer to environment} \right\}_5
\]

### 3.2.1.1 Existing model and improvement

#### 3.2.1.1.1 Tube-side Model

Tube side flow comprises the heat balance for the test fluid (either non-fouling or crude oil), which exchanges heat with the surroundings, and the pressure drop along the tube. The model is distributed in the axial direction of the tube and assesses the heat transfer coefficient, properties of the fluid, and the reduction of flow area due to fouling as function of local conditions.

Tubular test section is defined in axial direction for \( z = [0, L] \) and in the radial direction for \( r = [0, R_{\text{flow}}] \). The development of the heat balance is detailed elsewhere (Coletti 2010). In terms of enthalpies \( e_r \), the heat balance is:
Equation 3.3

\[
\frac{\partial}{\partial t} \left( \rho_T A_{flow} e_T(z) \right) = -\frac{\partial}{\partial z} \left( \rho_T A_{flow} e_T(z) u(z) \right) + \frac{\partial}{\partial z} \left( k_T A_{flow} \frac{\partial T_T}{\partial z} \right) + \\
+ 2\pi R_{flow}(z) h_T(z) (T_L(z) |_{R_{flow}} - T_T(z))
\]

where \( T_T \) is fluid temperature, \( \rho_T \) density, \( k_T \) is the conductivity of the fluid, \( u \) linear velocity, \( h_T \) is the film heat transfer coefficient, \( R_{flow} \) is the radius at the surface of the fouling layer, \( T_L |_{R_{flow}} \) is the temperature at the surface of the deposit, and \( A_{flow} \) represents flow area. The heat transfer coefficient is calculated using the Dittus - Boelter correlation.

The pressure drop for the system depends on friction loss and elevation. The elevation was taken into account because the tubular test section was installed in vertical direction.

Equation 3.4

\[ \Delta P = \Delta P_{friction} + \Delta P_{hydrostatic} \]

The pressure drop by friction, \( \Delta P_{friction} \), is calculated as function of local conditions as follows:

Equation 3.5

\[
\frac{dP_T(z,r)}{dz} = -f(z) \frac{\rho_T(z)}{R_{flow}(z)} (u(z))^2
\]

Equation 3.6

\[ \Delta P_{friction} = P(z) - P_0 \]

where \( f \) is the friction factor, \( P_T \) is local pressure and \( P_0 \) is the pressure at the inlet of the test section. The hydrostatic pressure, \( \Delta P_{hydrostatic} \), is:

Equation 3.7

\[ \Delta P_{hydrostatic} = \rho_{ave} g H \]

where \( H \) is the length of the tube and \( g \) the gravitational constant.

The physical properties of Paratherm follow the correlation given by manufacturer which has been stated in MSDS. Most of properties are a function of temperature of the fluid which the variation depends on local temperature of the process over time and space (radial and axial direction). However, the crude oil physical properties will follow API calculation and correlation (Riazi, 2005). These properties used as input parameters to calculate and/or analysis thermal and hydraulic of this experiment.
3.2.1.1.2 Fouling Layer

It is important to understand the concept of fouling layer formation before it can be translated into mathematical model. In this research, it only considers chemical reaction fouling. The main reason is the type of oils will be used for experiment are non-fouling oil and crude oil collected after desalting process. It is assumed that the crude oil is 'clean' and other types of fouling such as sedimentation; precipitation and etc. have been eliminated.

In the earlier literature, figure 3.2 shows the formation of deposit layer for chemical reaction fouling. This process will initially create a soft gel like layer which has a tendency to undergo suppression process. If the deposition process is continuously taken place, most probably the soft layer undergoes ageing process and the layer will be converted to hard layer. The physical properties of this layer will be more dense compare to soft layer. Coletti (2010) has also shown in figure 3.4 that the deposition rate can be divided into two categories. Figure 3.4a shows the development of thickness deposit based on constant deposition rate and figure 3.4b described the deposition rate with auto-retardation process. The $\delta$ is representing the deposit thickness and $t$ is the time where the occurrence of phenomena.

![Figure 3.4](image)

*Figure 3.4 Development of deposit thickness vs. time (Coletti 2010)*

The chemical reaction fouling for crude oil can be divided into 4 categories. There are no aging or 'clean' process, slow ageing, normal ageing and fast ageing process. The selector will be made available in the model development and the default parameters (shown in Table 3.4) will be used to calculate the rate of deposition, hence, thickness of deposit. However, further investigation shall be made via parameter estimation to estimate the value of pre-exponential and activation energy for respective ageing process.
Table 3.4 Default Parameter used for various types of Aging Process (ESDU 2006)

<table>
<thead>
<tr>
<th>Case</th>
<th>Default Parameter</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Aging</td>
<td>Pre Exponential = 0</td>
<td>s⁻¹</td>
</tr>
<tr>
<td></td>
<td>Activation Energy = 0</td>
<td>J</td>
</tr>
<tr>
<td></td>
<td>Thermal conductivity, ( k_0 = 0.2 )</td>
<td>Wm⁻¹K⁻¹</td>
</tr>
<tr>
<td></td>
<td>Fouling thermal conductivity = ( k_f(0,1) )</td>
<td>Wm⁻¹K⁻¹</td>
</tr>
<tr>
<td>Slow Aging</td>
<td>Pre Exponential = 0.0038/3600*24</td>
<td>s⁻¹</td>
</tr>
<tr>
<td></td>
<td>Activation Energy = 12.8 X 10³</td>
<td>J</td>
</tr>
<tr>
<td></td>
<td>Thermal conductivity, ( k_0 = (k_{coke} *(k_{gel} - k_{coke}) *y) )</td>
<td>Wm⁻¹K⁻¹</td>
</tr>
<tr>
<td></td>
<td>Fouling thermal conductivity = ( k_{gel} )</td>
<td>Wm⁻¹K⁻¹</td>
</tr>
<tr>
<td>Aging</td>
<td>Pre Exponential = 17/3600*24</td>
<td>s⁻¹</td>
</tr>
<tr>
<td></td>
<td>Activation Energy = 42.4 X 10³</td>
<td>J</td>
</tr>
<tr>
<td></td>
<td>Thermal conductivity, ( k_0 = (k_{coke} *(k_{gel} - k_{coke}) *y) )</td>
<td>Wm⁻¹K⁻¹</td>
</tr>
<tr>
<td></td>
<td>Fouling thermal conductivity = ( k_{gel} )</td>
<td>Wm⁻¹K⁻¹</td>
</tr>
<tr>
<td>Fast Aging</td>
<td>Pre Exponential = 2.88 X 10⁶/3600*24</td>
<td>s⁻¹</td>
</tr>
<tr>
<td></td>
<td>Activation Energy = 84.7 X 10³</td>
<td>J</td>
</tr>
<tr>
<td></td>
<td>Thermal conductivity, ( k_0 = (k_{coke} *(k_{gel} - k_{coke}) *y) )</td>
<td>Wm⁻¹K⁻¹</td>
</tr>
<tr>
<td></td>
<td>Fouling thermal conductivity = ( k_{gel} )</td>
<td>Wm⁻¹K⁻¹</td>
</tr>
<tr>
<td>User Specified Aging</td>
<td>Pre Exponential = user value</td>
<td>s⁻¹</td>
</tr>
<tr>
<td></td>
<td>Activation Energy = user value</td>
<td>J</td>
</tr>
<tr>
<td></td>
<td>Thermal conductivity, ( k_0 = (k_{coke} *(k_{gel} - k_{coke}) *y) )</td>
<td>Wm⁻¹K⁻¹</td>
</tr>
<tr>
<td></td>
<td>Fouling thermal conductivity = ( k_{gel} )</td>
<td>Wm⁻¹K⁻¹</td>
</tr>
</tbody>
</table>

It is understandable that the deposition of fouling layer will reduce heat transfer coefficient of the crude oil. The heat transfer would be treated as conduction type as deposit is assumed to be solid phase. The other important factor in this fouling layer model is the moving boundary conditions. In order to eliminate numerical complexity, the radial coordinate has been introduced as dimensionless function where \( r=0 \) for radius at original inner tube radius and \( r=1 \) for radius flow (deposit boundary).

Fouling layer simulates the gradual growth of a low conductive deposit on the inner surface of the tube. The layer is modelled as a solid-type of material which grows due to deposition and undergoes ageing. The domain, \( \Theta_A \), is defined in the axial \( (z = [0, L]) \) and radial direction \( (r = [r_i, R_{flow}]) \). The energy equation 3.2 will be simplified after neglecting second, fourth and fifth term of equations. Assuming negligible heat transfer in the axial direction and symmetrical behaviour in the angular coordinate,\( \Theta \):

**Equation 3.8**

\[ \rho_A c_p A \frac{\partial T_A(z,r)}{\partial t} = \frac{1}{r} \frac{\partial}{\partial r} \left( r k_A \frac{\partial T_A}{\partial r} \right) \]

Thermal conductivity for deposit layer varies over time due to ageing, as discussed later. A dimensionless radial coordinate is introduced to solve the moving boundary as the fouling
deposit grows (flow radius varies over time). This expression follows the ratio of radius at wall to radius at deposit layer.

**Equation 3.9**

\[
\bar{r} = \frac{r - r_i}{R_{flow} - r_i} = -\frac{r - r_i}{\delta}
\]

where \( \bar{r}, r \) and \( \delta \) are new radial coordinate, dimensionless radial coordinate, and deposit thickness, respectively. The dimensionless radial coordinates varies between 0 and 1: \( \bar{r} = 0 \) when \( r = r_i \), this is, the inner radius of the tube. \( \bar{r} = 1 \) when \( r = R_{flow} \), this is, the flow radius or deposit surface.

The heat balance becomes:

**Equation 3.10**

\[
\rho_A c_p A \frac{\partial T_A(z,r)}{\partial t} = \frac{1}{\delta^2(z)(r_i - \delta(z)\bar{r})} \frac{\partial}{\partial \bar{r}} \left( (r_i - \delta(z)\bar{r}) k_A(z,r) \frac{\partial T_A}{\partial \bar{r}} \right)
\]

The heat flux at this domain can be expressed via *Fourier's equation*:

**Equation 3.11**

\[
\delta(z)q(z,r) = k_A(z,r) \frac{dT_A(z,r)}{d\bar{r}}
\]

Equation 3.12 illustrates the formation of youth deposit which is important for further understand the ageing process for fouling phenomena.

**Equation 3.12**

\[
\frac{dy(z,r)}{dt_{age}} = -A_a \exp\left(\frac{-E_a}{RT_A(z,r)}\right) y
\]

**Equation 3.13**

\[
\frac{d\delta(z)}{dt} = \lambda^0 \frac{dR_f}{dt} (z)
\]

The Threshold model by Panchal et al. (1997) is used to calculate the fouling rate as function of local operating conditions:

**Equation 3.14**

\[
\frac{dR_f}{dt} = \alpha Re^{-0.66} Pr^{-0.33} \exp\left(\frac{E}{RT_f}\right) - \gamma r
\]
3.2.1.1.3 Tube Wall

Tube wall of heat exchanger commonly depends on material selection. It would be selected based on the physical properties of fluid, conductivity and their operating conditions. The most common tube materials for heat exchanger are carbon steel and stainless steel. Each material has physical properties which often contribute toward efficiency of heat transfer coefficient. However, economical factor, reliability, maintainability and robustness factor are also take into consideration.

Heat transfer within tube wall is dominated by conduction. The model of tube wall is developed within tube wall domain, \( \Theta_w \) which it is considering the spatial impact at axial position, \( z=[0,L] \) and radial position, \( r= [r_i, r_o] \). The energy balance at control volume as illustrated in equation 3.2 will be simplified where the second term and fifth term are neglected. Given the high conductivity of the metal compared to the other domains (insulation, layer), conduction in the axial domain has also been included in the balance. In addition, a generation term is included, which corresponds to the heat input by Joule heating. Assuming symmetrical behaviour in the angular coordinate:

Equation 3.15

\[
\rho_w c_{pw} \frac{\partial T_w(z,r)}{\partial t} = k_w \left( \frac{1}{r} \frac{\partial T_w(z,r)}{\partial r} + \frac{\partial^2 T_w(z,r)}{\partial r^2} \right) + k_w \frac{\partial^2 T_w(z,r)}{\partial z^2} + q''''
\]

where \( T_w \) is local temperature at specific location \( (z, r) \), \( \rho_w, c_{pw}, k_w \) are physical properties of the metal (density, heat capacity and thermal conductivity respectively), \( q'''' \) is heat source from Joule heating (W/m\(^3\)).

The heat flux at this domain can be expressed via Fourier's equation:

Equation 3.16

\[
q_w(z,r) = -k_w \frac{\partial T_w(z,r)}{\partial r}
\]

3.2.1.4 Joule Heating

Heating process for HIPOR pilot plant is provided by Joule heating which is also known as Ohmic or Joule heating. It is a conversion of current to heat through resistive material. Heating process in this model is a process to setting up thermal operating condition for test tube section. The heat supply can be adjusted into three types of operating conditions. They consist of constant heat flux or uniform heat flux (UHF), constant wall temperature or uniform wall temperature (UWT) and direct joule heating where heat input can be manipulated and/or being introduced as disturbance to the process. Apparently, the concept of Joule heating is the test tube will be directed heated at both end of tube via two clamps that directly connect the source of heating. The current supply is controlled at the power supply.
The UHF is controlled by constant heat supply from the source to test tube. It is assumed the heat flux operation by controlling the amount of current supply to the test tube throughout the experiment. The UWT is currently available through automatic control but yet to be tested and tune. In normal circumstances, the constant wall temperature can be obtained via manipulating power supply to test tube as temperature of the tube as control variable. Temperature must be set as a set point variable before any attempts to control this variable. A simple control system shall be developed to achieve this operating condition. Normally, PID controller should be sufficient to achieve this objective.

The heat generated at the wall is:

\[ q''' = \frac{p}{\pi(r_0^2 - r_i^2)H} \]

The domain for heating, \( \Theta_H \) is model based on Ohm’s Law and Joule’s Law. The relationship between power (P), voltage (V), resistance (R) and current (I) is:

\[ P = \frac{V^2}{R} \]

\[ I = \frac{V}{R} \]

\[ R = \frac{\sigma H}{\pi(r_0^2 - r_i^2)} \]

where \( \sigma \) is material resistivity, \( H \) is the height or length of the tube and \( r_0, r_i \) are radius for outer and inner tube respectively.

3.2.1.1.5 Insulation Layer

The insulation layer installed at test section aims to minimise the external factor influence the study of fouling phenomena in test section. The losses of heat to environment through convection and radiation could possibly reduce the actual heat transfer from sources to fluid, hence, it will deficient heat transfer to the fluid.

The selection of insulation must be carefully studied in order to obtain the purpose of thermal insulation. There are a few factors that influence the selectivity of insulator or lagging material. The common requirements of insulator material are a low thermal conductivity and it has a tendency to suppress convection. Other factors that also can be taken into consideration are economic impact and critical thickness of insulator. Economic impact commonly correlates with deterioration of material through aging and infiltration. Critical thickness is also important as its thickness increases, the possibility of increases of heat losses especially to cylinder pipe.
Figure 3.5 Critical thickness of insulator (Coulson & Richardson Vol. 1)

Figure 3.5 shows the influence of critical thickness of insulator towards heat losses to atmosphere. $Q_0$ is the rate of heat loss without any insulator, $h$ is the outside heat transfer coefficient, $k$ is the thermal conductivity of insulator and $r$ is the thickness of insulator. As shown in Figure 3.5, when $hr/k > 1$, increment of insulator thickness will reduce heat loss compare to $hr/k < 1$, increment of thickness will increase heat loss until it reaches the ratio of $Q/Q_0$ equal to 1. It is relatively important to understand the impact of critical thickness of insulator before any attempts of applying thermal insulation to pipeline.

The model for insulation layer is developed to quantify heat loses to environment during experimental process. The insulator used for this process is mineral wool where the physical properties provided from Material Safety Data Sheet (MSDS).

It is important to realise the important factors affecting the selection of insulator for piping application in general and specifically in this HIPOR in particular. The model for this insulation layer is developed base on energy balance stated at equation 3.2 in the insulation domain, $\Theta_i$ that illustrated in figure 3.3. The energy balance of this domain is define between axial position, $z = [0, L]$ and radius, $r = [r_0, R_0]$ where $L$, $r_0$, $R_0$ represent tube length, outer tube radius and total tube radius (inclusive of insulation thickness). The heat transfer in this domain dominated by conduction and other methods of transfer like convection and radiation are considered negligible. The second term in equation 3.2 is neglected. Heat loss to environment dominated by heat transfer by convection (Newton's Law of Cooling) is captured at the boundary condition, as explained later in this chapter. The final model expression of energy balance can be described as:
Equation 3.21
\[ \rho I c_p I \frac{\partial T_I(z,r)}{\partial t} = k_I \left( \frac{1}{r} \frac{\partial T_I(z,r)}{\partial r} + \frac{\partial^2 T_I(z,r)}{\partial r^2} \right) \]

where \( T_I \) is local temperature at specific location \((z, r)\), \( \rho_I \) \( c_p I \) \( k_I \) are physical properties of insulator (density, heat capacity and thermal conductivity respectively).

The heat flux of insulator can be calculated via Fourier’s equation:

Equation 3.22
\[ q_I(z,r) = -k_I \frac{\partial T_I(z,r)}{\partial r} \]

3.2.1.1.6 Boundary Conditions

The model development with algebraic and differential equations for HIPOR experimental has their boundary and initial conditions which depend on their process conditions.

As mentioned earlier, the losses of heat to environment only considered heat transfer via convection and heat transfers via radiation and conduction are assumed negligible in both operating conditions.

The heat flux between atmosphere and outer thickness of insulator can be expressed as:

Equation 3.23
\[ q_I|_{r=R_o} = h_o(T_I|_{r=R_o} - T_{amb}) \]

where \( h_o \) is air heat transfer coefficient and \( T_{amb} \) is ambient temperature.

The temperature of insulator is expected to be higher than ambient temperature since the internal heat generation taken place at wall of the test tube.

At interface between insulator and wall of outer tube, the temperature and heat flux should have continuity, depends on their operating conditions and can be expressed as:

Equation 3.24
\[ T_I|_{r=r_o} = T_W|_{r=r_o} \]

Equation 3.25
\[ q_I|_{r=r_o} = q_W|_{r=r_o} \]

At interface of inner wall of the tube and deposit layer also have a continuity of heat flux and temperature:

Equation 3.26
\[ T_W|_{r=r_i} = T_d|_{r=r_i} \]
\textbf{Equation 3.27}

\[ q_w|_{r=r_i} = q_A|_{r=r_i} \]

There is a moving boundary between deposit layer and tube wall of flow area and heat flux depending on Fourier’s Law expression;

\textbf{Equation 3.28}

\[ q_A|_{r=r_{\text{flow}}} = h_{\text{fluid}} \left( T_A|_{r=r_{\text{flow}}} - T_{\text{fluid}} \right) \]

\section*{3.2.1.7 Solution Method}

The mathematical model comprises partial, differential and algebraic equations which is implemented in gPROMS. The distributed domains are discretized by using centre finite difference method (CFDM) for the radial domains and backwards finite discretization method (BFDM) for the axial domain. Time integration is performed by the standard DASOLV solver.

\section*{3.3 Conclusions}

Chapter 3 presented the mathematical model that was the adapted model from (Coletti, 2011) and modified to suit with the HiPOR test section. The modifications on the adapted model has been highlighted which include the joule heating and insulation model. There are a few correlations expressions had been presented according to literature which related to heat transfer, fouling model and friction factor. The friction factor correlations captured the differences for validation that explained extensively in chapter 4 and 5.
4 Baseline validation against experimental data on test section

Baseline validation is very important in crude oil fouling study. To the best of the knowledge of the author, none of the research paper on the crude oil study has reported extensively their baseline validation results especially with respect to their experimental rig. Amongst the objectives which will be discussed in this chapter is to highlight the importance of baseline validation, factors that influence validation process, repeatability of primary measurement, heat loss effect on the test section and the thermal and hydraulic impact on the variation of operating conditions.

4.1 Introduction

The experiments have been designed to characterise and understand further the physical rig of HiPOR. These experiments were done with non-fouling oil, Paratherm. The main reason is to obtain the thermal and hydraulic profiles of the test section before any crude oil fouling run is attempted. Furthermore, the data could be compared with simulation results which extensively been modelled as discussed previously in chapter 3. Several operating conditions such as inlet temperature, heat input, flowrate have been varied in order to validate the robustness and reliability of the model.

4.1.1 The importance of baseline validation

Amongst the important factors of the baseline validation is to obtain the thermal and hydraulic effect of the non-fouling oil experiment results, especially involving the primary measurements like temperature and differential pressure. These results are more predictable as non-fouling oil physical properties were supplied by the manufacturer. Thus, it can be compared with the crude oil fouling run to observe the differences. As discussed earlier in chapter 3, the model is designed for both oils; non-fouling oil and crude oil.
4.1.2 Factors influence the validation process

The influential factors for validation process will be divided into two categories; experiment and modelling. In experiment, the primary measurements play very important roles, ranging from the accuracy and precision of instrumentations, reproducibility and sufficient measurement points. For modelling, the physical descriptions of the process need to be correct, correlation used for important calculated parameters, e.g. friction factor, fouling resistance and input parameters.

4.2 Experimental setup and procedure

The test section is the most important section in crude oil fouling investigation. It involves installation of various primary measurements, equipment to ensure all important parameters are controlled and captured the moment of the phenomenon.

Physical plant data for HIPOR have been listed as Table 4.1 for preheater and tubular test sections respectively. It will be used for further analysis to calculate the surface area, flow area, and other pertinent parameters such as heat transfer and pressure drop to create baseline validation purposes. Some properties such as thermal conductivity would vary based on operating temperature and so does the absolute roughness if fouling starts to deposit on the surface area of the inner tubular test section.

Table 4.1 Physical Data for Preheater and Tubular Section

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Preheater</th>
<th>Tubular 3/4”</th>
<th>Tubular 1”</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inner diameter</td>
<td>m</td>
<td>0.01575</td>
<td>0.0196</td>
<td>0.0269</td>
</tr>
<tr>
<td>Outer diameter</td>
<td>m</td>
<td>0.01905</td>
<td>0.0229</td>
<td>0.03016</td>
</tr>
<tr>
<td>Length</td>
<td>m</td>
<td>2.5</td>
<td>1.80</td>
<td>1.74</td>
</tr>
<tr>
<td>Density @25 °C</td>
<td>kg/m³</td>
<td>7900</td>
<td>7850</td>
<td>7850</td>
</tr>
<tr>
<td>Specific heat capacity @25 °C</td>
<td>J/kgK</td>
<td>510</td>
<td>490</td>
<td>490</td>
</tr>
<tr>
<td>Thermal conductivity @25 °C</td>
<td>W/mK</td>
<td>16</td>
<td>36</td>
<td>36</td>
</tr>
<tr>
<td>Resistivity @25 °C</td>
<td>Ohm m</td>
<td>76.0E-8</td>
<td>16.9E-8</td>
<td>16.9E-8</td>
</tr>
<tr>
<td>Absolute roughness</td>
<td>m</td>
<td>45E-5</td>
<td>14E-5</td>
<td>14E-5</td>
</tr>
</tbody>
</table>

Table 4.2 states the physical plant properties of both test sections, the Paratherm properties at 25 °C and joule heating capabilities. The Paratherm properties will be varied with increment of the operating temperature as it follows the expression in Table 4.3.

Table 4.3 presents the correlation of Paratherm physical properties with function of temperature with a high degree of regression value. It is an important expression to take into account when the value will be used in heat transfer calculation at later stages of research.
### Table 4.2 Physical plant properties for test sections

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Tubular 3/4&quot;</th>
<th>Tubular 1&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inner diameter</td>
<td>m</td>
<td>0.0196</td>
<td>0.0269</td>
</tr>
<tr>
<td>Outer diameter</td>
<td>m</td>
<td>0.0229</td>
<td>0.03016</td>
</tr>
<tr>
<td>Length</td>
<td>m</td>
<td>1.80</td>
<td>1.74</td>
</tr>
<tr>
<td>Inlet temperature</td>
<td>°C</td>
<td>30-250</td>
<td>30-250</td>
</tr>
<tr>
<td>Inlet pressure</td>
<td>bar</td>
<td>5-25</td>
<td>5-25</td>
</tr>
<tr>
<td>Inlet volumetric flowrate</td>
<td>m³/hr</td>
<td>0.8-2.2</td>
<td>1.0</td>
</tr>
<tr>
<td>Inlet velocity</td>
<td>m/s</td>
<td>Up to 1.2</td>
<td>0.4887</td>
</tr>
</tbody>
</table>

**Insulator properties**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Property 1</th>
<th>Property 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density @25 °C</td>
<td>kg/m³</td>
<td>2600</td>
<td>2600</td>
</tr>
<tr>
<td>Specific heat capacity @25 °C</td>
<td>J/kgK</td>
<td>1000</td>
<td>1000</td>
</tr>
<tr>
<td>Thermal conductivity @25 °C</td>
<td>W/mK</td>
<td>0.061</td>
<td>0.04</td>
</tr>
<tr>
<td>Thickness</td>
<td>m</td>
<td>0.03</td>
<td>0.01</td>
</tr>
</tbody>
</table>

**Paratherm properties**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Property 1</th>
<th>Property 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density @25 °C</td>
<td>kg/m³</td>
<td>880.385</td>
<td>880.385</td>
</tr>
<tr>
<td>Specific heat capacity @25 °C</td>
<td>J/kgK</td>
<td>1856.7</td>
<td>1856.7</td>
</tr>
<tr>
<td>Thermal conductivity @25 °C</td>
<td>W/mK</td>
<td>0.10425</td>
<td>0.10425</td>
</tr>
<tr>
<td>Viscosity @25 °C</td>
<td>cP</td>
<td>40.09</td>
<td>40.09</td>
</tr>
</tbody>
</table>

**Joule Heating properties**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Property 1</th>
<th>Property 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resistivity @25 °C</td>
<td>Ohm m</td>
<td>16.9E-8</td>
<td>16.9E-8</td>
</tr>
<tr>
<td>Current</td>
<td>Ampere</td>
<td>1000</td>
<td>1000</td>
</tr>
</tbody>
</table>

### Table 4.3 The correlation of Paratherm physical properties to temperature

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Correlation</th>
<th>Regression</th>
<th>Temperature range °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>kg/m³</td>
<td>( y = -0.6578x + 896.84 )</td>
<td>R² = 0.9984</td>
<td>-20 to 343</td>
</tr>
<tr>
<td>Specific heat capacity</td>
<td>J/kgK</td>
<td>( y = 0.0053x + 1.7242 )</td>
<td>R² = 0.9995</td>
<td>-20 to 343</td>
</tr>
<tr>
<td>Thermal conductivity</td>
<td>W/mK</td>
<td>( y = -5e^{-5}x + 0.1055 )</td>
<td>R² = 0.9937</td>
<td>-20 to 343</td>
</tr>
<tr>
<td>Viscosity</td>
<td>cP</td>
<td>( y = 16649x^{-1.873} )</td>
<td>R² = 0.99998</td>
<td>24 to 66</td>
</tr>
<tr>
<td></td>
<td></td>
<td>( y = 8050.9x^{-1.708} )</td>
<td>R² = 0.99771</td>
<td>79 to 191</td>
</tr>
<tr>
<td></td>
<td></td>
<td>( y = 14356x^{-0.014} )</td>
<td>R² = 0.9994</td>
<td>204 to 343</td>
</tr>
</tbody>
</table>
Table 4.4 Summary of Paratherm experiment

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Description</th>
<th>Temperature Range, °C</th>
<th>Pressure Range, bar</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>RUN 235</td>
<td>Cold Run</td>
<td>50 - 70</td>
<td>5-10</td>
<td>Varies the flowrate</td>
</tr>
<tr>
<td>RUN 236</td>
<td>Cold Run</td>
<td>50 - 70</td>
<td>5-10</td>
<td>Repetition of RUN 235</td>
</tr>
<tr>
<td>RUN 259</td>
<td>Hot Temperature without joule heating to test section</td>
<td>50-250</td>
<td>5-10</td>
<td>Designated test at temperature 100 °C, 150 °C, 200 °C and 250 °C</td>
</tr>
<tr>
<td>RUN 260</td>
<td>Hot Temperature with joule heating to test section</td>
<td>50-200</td>
<td>5-10</td>
<td>Test only at 200 °C with different flowrate</td>
</tr>
</tbody>
</table>
| RUN 266    | 24 hours Run                                     | 60-220               | 5-25                | Flowrate = 1.2 m³/h  
Temperature= 220 °C  
Current = 950 A |

4.2.1 Cold run experiment descriptions and objectives

The cold run experiment was part of the commissioning process. It involved in testing the functionality of the equipment like pumps, control valves as well as the instrumentations such as thermocouples, pressure sensor, and etc.

4.2.1.1 Experiment descriptions and objectives

Cold run experiment is run at the minimum temperature. Initially, it is designed to run the experiment at ambient temperature. The main objective of this run is to capture the hydraulic and thermal profile for non-fouling oil at the lowest temperature possible. Unfortunately, this run at ambient temperature couldn’t be done because of the limitation at the pump. At this temperature, the kinematic viscosity is 50 mm²/s which very high and creates high resistance towards motor current. Testing has been done, and motor current run at overload current. For this reason, the cold run for non-fouling oil runs at temperature 70 °C. At this temperature, the oil can be circulated throughout the system with current loading at 90 % of maximum current.

The other objectives of this run are to calculate the heat generated by the main pump, M1 and monitor the repeatability of the primary measurements. The reliability and reproducibility of primary measurements are as important as processing the data itself. The manufacturer of instrumentation normally would supply the instrumentation with specific technical data which includes the precision and accuracy of the instrumentation. However, it is relatively important for the researcher to calibrate the instruments offline and online to countermeasure the actual data given by the supplier.

A recent study (Kim et al., 2013) shows the importance of uncertainty concept for thermophysical properties in experimental measurement which could jeopardise the process validation process. In their example, this propagation of uncertainties could amplify the error of final expression of variable that only can be determined through the calculation process of
measured variables. As mentioned earlier in literature review, most important variables to be monitored in fouling phenomena such as fouling resistance, heat transfer coefficient, dimensionless number, came from the thermophysical properties expression and other physical plant properties via calculation process. Therefore, measurement of parameters is relatively important in order to create a baseline validation for a HIPOR rig for validation purposes. All results for experiment Run 235 and Run 236 will be discussed in detail in section 4.3 of this chapter.

4.2.1.2 Experiment protocol
A cold run experiment generally designed for training an operator. After the preliminary check of the rig, activated the nitrogen generator, chiller for cooling water supplied, CO₂ suppression system, rapid ventilation, then the rig is ready for operation. The start-up procedure is started with activated the LabView interface for monitoring and controlling purposes. Run number will be keyed in as the approved by in the permit to operate. Control valve, V10 is opened manually which allows the cooling water flow from the chiller to the bowman heat exchanger in cooling loop. Then, the cooling pump, M2 was put in operation. This cooling loop is designed to reduce the outlet temperature of testing oil from the test section. This loop also used for shutdown purposes in the hot run experiment. As the protection and safety of the rig, the main pump, M1 cannot put in operation (interlock) without the signal of cooling pump, M2. Subsequently, the main loop which is the test section will put into service. First, the isolation valves, V3 and V22 will be opened. This allows the oil is flowing from the main tank to the test section and return back the main tank. It was the closed loop experiment where both valves were used to isolate the main tank from the circulation loop. The main pump, M2 was used to transport the oil from the tank and return back into the tank. Before M2 is started, control valve, V17 is initially opened at 10 % opening. This allows the oil to be circulated and prevent damage to the pump. The flow is increasing by increases the opening of V17. In this experiment, V17 is opened at intervals of 10 % until it reaches 100 %. The experiment was held for approximately 2 hours before the shutdown procedure has taken place. All thermal and hydraulic parameters are monitored and recorded within LabView environment.

4.2.2 Hot run experiment descriptions and objectives
The hot run experiments can be divided into two categories. First, the experiment was done without any joule heating being introduced to the test section and the other experiment is the introduction of heat input via joule heating at test section.
4.2.2.1 Experiment descriptions and objectives

Hot run experiment without joule heating is an experiment where the inlet temperature is increased to the desired inlet temperate through the heat input at the preheater section. This heat will be supplied until the desired inlet temperature, T5 to the test section is achieved. To maintain the T5, the control system, TC01 will put in automatic mode. RUN 259 is designed especially for this purpose. The main aims of this experiment are to study the thermal and hydraulic effect, control valve characteristic at different inlet temperature, the effect of flowrate variation at constant T5 and verification of the corrected flowrate which previously discussed in chapter 2.

Another hot run experiment is an experiment with joule heating. In this experiment, The T5 is kept constant and heat input via joule heating is introduced in the step change of current at the interval of 100 A. RUN 260 is designed specifically for this purpose. Amongst the aims of this experiment are; to study the thermal and hydraulic effect of the heat input variation and to validate the experimental data against the simulation results.

4.2.2.2 Experiment protocol

The initial start-up of a hot run experiment is similar with a cold run experiment. The only difference was the heat input is introduced via preheater section in order to increase the oil temperature to the desired inlet temperature. In Run 259, the inlet temperature, T5 was increased to 100 °C, 150 °C, 200 °C and 250 °C. At the mentioned temperature, the flowrate were varied by increasing the control valve, V17 opening at the interval of 10 % until it reaches 100 % opening. The analysis of the friction loss and the thermal profiles due to flowrate can be done and discussed in the later section of this chapter. Furthermore, the analysis of the characteristic of the valve at a different inlet temperature also can be performed.

The Run 260 is designed for the hot experiment with joule heating. The inlet temperature, T5 is kept steady at 200 °C. The flowrates were varied at different valve opening; 25 %, 50 %, 75 % and 100 %. At these conditions, the current is introduced at the test section with a step change of 100 A until it reached maximum value of 1000 A. The thermal effect shall be seen in the outlet temperature, T5 as well as the wall temperatures; T20, T12, 6, T21 and T11. The hydraulic effect shall be captured via the differential transmitter, dP2 which measuring the pressure difference between the inlet and outlet of the test section. All relevant primary instrumentations are shown in Figure 4.1.

For both experiments, the shutdown procedure needs to be executed carefully in order to protect the process as well as the equipment. In the hot run experiment, the first step was to isolate the heat input to the rig. In Run 259, the only heat input was in the preheater and Run 260 has two sources of heat input; preheater and main heater (test section). After the power
supply is turned OFF, the process oil temperature will be decreased. The rate of decrement depends on the convection heat transfer via rapid ventilation and copper block heat exchanger which allocated at the return line of the test section. When the oil temperature reaches 150 °C, the oil will be introduced in close step into the cooling loop. The cyclic process is introduced because of the limitation of the compabloc heat exchanger in order to avoid thermal stress.

![Figure 4.1 Schematic diagram of tubular test section and its primary measurements](image)

**4.2.3 Designated run**

The designated run normally was incorporated within the cold and/or hot run experiments. Several runs had been performed during commissioning and data have been analysed accordingly, which were not presented in this thesis. Based on this reason, the designated run has been designed to verify some discrepancies of the data which obtained during commissioning. Amongst the reasons:

- Verification of the corrected flowrate against the pump power
- Effect of magnetic field towards volumetric flowrate
- Heat loss quantification and their effect on thermal profiles

**4.2.3.1 Experiment descriptions and objectives**

For example, Run 259 was done in different inlet temperature, 100 °C, 150 °C, 200 °C and 250 °C. Normally, in each run, some variations of operating condition enabled to study and validate various scopes of baseline validation. In this run, amongst the objectives are to study the control valve characteristic and verification of the corrected flowrate against the valve opening at different constant inlet temperature, T5.
The heat losses also can be quantified in the hot run experiments, Run 259 and Run 260 respectively. The results obtained from the analysis which will be discussed later will lead into the improvement of the HiPOR rig in order to capture more important data, thus all phenomena related to crude oil fouling could be physically and scientifically explained.

4.3 Results and discussions

In this section, all experimental results will be described and analysed according to the objectives stated in section 4.2. The important parameters will be displayed for the entire experiments and some designated objectives which incorporated within the experiment will be thoroughly discussed and analysed. Last part of this section will show the validation process between the experimental results against the simulation results.

4.3.1 Cold run experiment

There are two important objectives of these experiments which were the reproducibility of primary measurements and the heat input for the main pump, M1. Two runs had been performed, Run 235 and Run 236 with the same operating conditions.

Figure 4.2 Flowrate profiles for Run 235

Figure 4.2 shows the flowrate profiles for tubular test section, F2 in Run 235. Initial 35 minutes was the start-up process. In this condition, the rig was kept in standby mode. The only equipment that have been in service are the auxiliary equipment like cooling loop, rapid ventilation system, nitrogen generator and CO2 suppression system. As the main pump, M1 started to operate, the flowrate starts to increase to 0.86 m$^3$/h. The control valve is open in a step change of 10% for every 2 minutes, and the response of flowrate can be seen until it
reaches the maximum flowrate of 2.2 m$^3$/h. As discussed earlier in chapter 2, the measured flowrate in HiPOR needs to be corrected as the flowmeter was calibrated based on crude oil at temperature 300 °C. The corrected flowrate is shown in Figure 4.2 and the value is approximately 84 % from the measured flowrate. The experiment was kept at steady state for 2 hours and shutdown procedure has taken place.

![Figure 4.3 Temperature profile for the main tank, T1](image)

For the first 35 minutes, no oil was flowing through the rig. The tank temperature, T1 dropped due to the effect of rapid ventilation inside the enclosure as shown in Figure 4.3. When the pump, M1 started, T1 was spiked for a few seconds before it starts to decrease. The most probable reason for the spike in temperature was the location of thermocouple T1. T1 was located at the bottom of the tank. As the volume of the tank is approximately 50 litre, the oil temperature at the top level of the tank may be slightly higher than the bottom temperature. When the oil starts to circulate, T1 is dropped nearly 10 °C. The main reason was the oil temperature at the remaining of the rig is at ambient temperature. The amount of this oil approximately 7 litre and it took 12 minutes for the oil temperature to homogenise. At minutes 56, the oil temperature starts to increase. Even though, no heat input is supplied to the rig the oil temperature is increasing with the rate of increment 3 °C/h. The only reason is the heat input, which came from the main pump into the oil. From this result, it has satisfied one of the objectives which is to observe the heat input from the pump.
Baseline validation against experimental data on test section

4.4 Differential Pressure, dP2 [mbar] for tubular test section

Figure 4.4 shows the differential pressure measurement, dP2 for the tubular test section. The expression for pressure drop has been explained in Equation 3.4. The only difference between the expression and measurement was the unit. Equation 3.4 is calculated in Pascal (Pa) and the measured data is in mbar. Since the test section was installed in the vertical arrangement, the pressure drop for the tubular test section consists of the friction and static pressure. The simulation results will be corrected later in mbar for the comparison and validation purposes. From Figure 4.4, there was an increment of dP2 as pump, M1 started to operate. This increment due to the friction pressure as the flowrate starts to increase. It also shows the sudden drop of dP2 before it starts to increase back and reaches the maximum value of this experiment. The sudden drop can be explained as there is a spike of the tank temperature, T1 as shown in Figure 4.3. The spike of T1 generally decreases the static pressure of the test section, though the value was only 2 mbar. It shows that the instrumentation was calibrated precisely and able to capture every movement and disturbance inside the rig. As the experiment is kept steady for 2 hours, there are slight changes of dP2, (-1 mbar). The reason is the increment of oil temperature inside the rig as the results of heat input from the pump, M1, hence decreasing the static pressure for the tubular test section.

4.3.2 Reproducibility of experiment

To test the reproducibility of the measurements, the results of two cold run experiments are examined in this section. The two runs were performed with the exact same schedule of inputs (given above) thus the values of the measured variables are expected to be close. The following shows data for a time slice of 100 min from the same starting time (minute 50) in the two runs.
Baseline validation against experimental data on test section

Figure 4.5 shows that the difference in measured flowrates at the inlet of the annulus (F1), tubular (F2) and cooling sections (F3) between the two runs is within ±0.04 m³/h (2%). Considering the scale and complexity of the equipment, this is an excellent result, indicating that repeated cold runs with non-fouling fluids are reproducible enough to allow the study of the phenomena of interest.

Figure 4.5 Flowrate measurement points are reproducible within an absolute difference of ±0.04 m³/h

The differences in the differential pressure measurements at the tubular test section for the two experimental runs considered also show excellent agreement as shown in Figure 4.6. In this case, the difference within the two runs is ± 2 mbar. The results in Figure 4.5 and Figure 4.6 demonstrate that the control and instrumentation systems on the rig are capable of producing replicable results which can be used with confidence for baseline validation with non-fouling oil.

4.3.3 Hot run experiments

The hot run experiments can be divided into two categories; without and with joule heating being introduced to test section. Both experiments are incorporated with designated experiments which had been explained in section 4.2.
4.3.3.1 Run 259

Run 259 is designed to characterise the HiPOR rig without any joule heating introduced into the test section. The primary measurements for the entire experiment can be seen in figures 4.7, 4.8 and 4.9.

![Inlet temperature profile, T5 for Run 259](image1)

**Figure 4.7** Inlet temperature profile, T5 for Run 259

The inlet temperature, T5 profile is shown in figure 4.9 for the entire experiment of Run 259. In this run, the T5 has been increased at temperature of 100 °C, 150 °C, 200 °C and 250 °C. At these constant inlet temperatures, the flowrate were varied via the opening and closing of control valve, V17 in order to observe the effect of the flowrate on the thermal and hydraulic profiles of the test section.

![Flowrate, F2 profile for measured and corrected flowrate in tubular test section](image2)

**Figure 4.8** Flowrate, F2 profile for measured and corrected flowrate in tubular test section
Figure 4.8 shows the flowrate, F2 profiles for measured and corrected values. As previously discussed in chapter 2, the flowmeter was calibrated based on crude oil at temperature 300 °C. This baseline validation experiment was using non-fouling oil, Paratherm which has a different physical properties from the crude oil. Hence, the corrected factor needs to be applied in order to obtain the correct flowrate for analysis. It shows that the differences of the corrected flowrate and the measured flowrate is much higher at the lower temperature compare with the higher temperature. In fact, at the highest opening of V17 (100 %), the measured flowrate shows the same values and it is proven as previously shown and discussed in figure 2.21. In this figure, it also shows the decrements and increments of F2 at the constant different inlet temperature, T5.

![Figure 4.8](image)

**Figure 4.9 Differential pressure, dP2 for Run 259**

Another important parameter that needs to be captured is a differential pressure of the test section. The differential pressure for the test section, dP2 for Run 259 is shown in figure 4.9. From the graph, the trending of dP2 is decreasing as the inlet temperature, T5 is increasing. This happens because of the static pressure of the test section as the density of the oil is decreased. The density of Paratherm is a function of the temperature as shown in table 4.3. The dP2 also shows some decrements and increments when the variation of the flowrate was imposed in the experiment. The decrements of flowrate will result the decrement of friction loss in the test section.

After 580 minutes, the Run 259 was moved from the experimental phase to the shutdown phase. At this point onwards, T5 is decreasing, resulted the increasing of dP2. When T5 is approximately 150 °C, the cyclic process of cooling via cooling loop is introduced. It can be seen (minute 620) the interruption in F2 as well as dP2 in figure 4.8 and 4.9 respectively. The Run 259 was completed at minute 720.
4.3.3.2 Run 260

Run 260 is an experiment run with joule heating introduced into test section. The experiment was kept constants at 200 °C for the designated tests. Amongst the designated tests are; to study the effect of the heat input on different flowrate of the oil and the observed the effect of joule heating on the flowrate of the oil. These tests performed in 4 stages of flowrate via the desired control valve, V17 opening. An overview of Run 260 is shown through figures 4.10, 4.11 and 4.12.

Figure 4.10 Inlet and outlet temperature, T5 and T7 and current input for Run 260

Figure 4.10 displays the inlet and outlet temperature, T5 and T7 respectively, and the current input to the test section. There are 4 stages of tests with varying the V17 opening which is shown in figure 4.11. At every stage, the joule heating is introduced by increasing the current input in step changes of 100 A until it reached the maximum value of 1000 A as shown in figure 4.10. The Run 260 also undergone three phases which are startup, experiment and shutdown phase. The startup phase can be seen within the first 140 minutes of the experiment where the inlet temperature, T5 is increased until it reaches the desired operating condition, 200 °C. The designated tests took place at minutes 140 until minutes 580. From minutes 580 onwards, the shutdown phase has begun to take place.
Figure 4.11 Corrected flowrate, F2 profile and control valve, V17 opening [%] for Run 260

Figure 4.11 shows the corrected flowrate, F2 profile and control valve, V17 opening for Run 260. Since there were 4 stages of tests in this run, the flowrate, F2 shall correspond to V17 opening between the start of experiments until the time before the shutdown phase took place. Despite 4 steps of V17 opening, the F2 were disturbed, especially when joule heating was introduced. For this reason, an analysis of the effect of joule heating had been made and will be discussed later in this chapter.

Figure 4.12 Differential pressure, dP2 for Run 260

Figure 4.12 presents the differential pressure, dP2 profile for Run 260. Initially the temperature is ramping from startup temperature, 60 °C to the desired inlet temperature, 200 °C, dP2 is linearly decreased. As T5 is controlled at 200 °C, dP2 also reached steady state. There were
slight changes of dP2 (-1 mbar) when joule heating was increased from 100 A to 1000 A. The most probable reason was the outlet temperature, T7 increases as current input increases. The dP2 decreases as current input decreases as well. As the flowrate increases in order to move into another stage, dP2 shows increment. An increment of flowrate resulted the increment of friction pressure loss.

4.3.3.3 Run 266

Run 266 is designed for a long run experiment with total experiment time approximately 24 hours. Amongst the aims are to control the desired flowrate, F2 and inlet temperature to test section, T5 precisely in order to prepare for crude oil fouling. Thermal and hydraulic profiles are closely monitored and analysed. Since no fouling will be occurring during this run, the desired operating temperature was only kept at steady state for only 2 hours and other designated tests were done for the remaining period of the experiment run. For that reason, the results will be displayed for the first 12 hours for Run 266 as shown in figures 4.13 (only for minutes 200 to 600), 4.14 and 4.14.

Figure 4.13 Start-up process of RUN 266 until it reaches steady state at T6= 220 °C

Figure 4.13 shows the initial start-up process of HiPOR. The ramping process of T6 took approximately 220 minutes where the current input was introduced in step change process. When T6 reaches 220 °C, the temperature controller, TC01 was changed from manual to automatic mode with setpoint of 220 °C. As the process was done in a closed system, the oil in the tank is keep warming up until it reaches steady state. At this moment, all temperatures at
strategic locations were measured in the entire rig. In this condition, as described in the law of thermodynamic in conservation energy, the heat input is equal to heat losses. The heat loss could be calculated based on the temperature profile which will be shown in Figure 4.23 later. Figure 4.13 also displays the introduction of joule heating at test section at minutes of 350 with increment of current of 100 A at the interval of 5 minutes. Since the T6 is controlled automatically at setpoint 220 °C, the power input at preheater is decreasing as the joule heating at test section is increasing. The temperature profiles and further explanation of this process will be based on trending in Figure 4.23.

Figure 4.14 displays the inlet and outlet temperature, T5 and T7 respectively, and the current input to the test section. The startup phase can be seen within the first 220 minutes of the experiment where the inlet temperature, T5 is increased until it reaches the desired operating condition, 220 °C. The experiment is maintained at this condition for an hour before the heat input via joule heating had been introduced. The joule heating is introduced by increasing the current input in step changes of 100 A until it reached the maximum value of 1000 A as shown in figure 4.13. As the heat input increases, T7 increases as well. It also shows that T7 steadily maintained after the maximum heat input is reached. After 520 minutes, the first aim of the Run 266 has been fulfilled; to control precisely F2 and T5. The experiment then moves to the next designated test to observe the effect of thermal and hydraulic profiles at inlet temperature of 150 °C.
Figure 4.15 Corrected flowrate, F2 profile and current input [A] for Run 266

Figure 4.15 shows the corrected flowrate, F2 profile and current input for Run 266. This flowrate correction was done in the data analysis rather than inside the LabView interface. Even though F2 was controlled at 1.2 m3/h, the corrected flowrate shows slightly lower compared with the measured value. The measured flowrate has been put in automatic mode at minutes 15 and keep constant until the first objective of the experiment run achieved in minutes 520. But the corrected flowrate is a function of density, which indirectly is a function of temperature. The corrected flowrate has shown that it increases as the temperature increases. The value shows a constant value when the desired inlet temperature for, T5 is achieved and maintained. The analysis of heat input towards the test section will be discussed further later in this chapter.

Figure 4.16 Differential pressure, dP2 for Run 266
Figure 4.16 displays the differential pressure, dP2 profiles for 12 hours in the Run 266. The trending was effected at the beginning of the experiment as the flowrate is increasing to the desired flowrate. Then, dP2 linearly decreases as T5 increase. At this condition, the density of oil is decreasing, thus the static pressure is decreased. At minutes 440, there is some decrement of dP2. The decrement is a result of the changes in flowrate setpoint from 1.2 m$^3$/h to 1.1 m$^3$/h which can be seen in figure 4.14.

### 4.3.4 Effect of inlet temperature

The experimental results had been presented in the previous section which involved the hot experiment runs with and without heat input in the test section. Here, in this section, a few analyses were done to study the effect of variation of inlet temperature, T5.

![Differential pressure vs. flowrate at different inlet temperature](image)

**Figure 4.17 Differential pressure vs. flowrate at different inlet temperature**

Figure 4.17 displays the differential pressure, dP2 between the inlet and outlet of the test section against the flowrate, F2 at different inlet temperature, T5. At the same amount of F2, the dP2 depends on hydrostatic pressure, which is based on the elevation of the test section. This dP2 varies for the different inlet temperature, T5. It is noted that the hydrostatic pressure is a function of density. For Paratherm, the density decreases as the temperature increase. That is the reason dP2 shows differences at different T5. At constant inlet temperature, dP2 increase as F2 increases. This is due to frictional loss occurring between the oil and surface of the test section.

Another analysis of the effect of inlet temperature has been presented previously in chapter 2 for clarification and justification of correcting flowrate from the measured flowrate, which were displayed in figure 2.20 and 2.21. As an extension of this data, the power requirement for the
pump was plotted against the corrected flowrate and shown in figure 4.16 and density correlation in table 4.3.

Figure 4.18 shows the power requirement for the motor pump against the corrected flowrate at different inlet temperature, T5. The motor power is much higher at low inlet temperature compare with higher temperature. It is noted that the density correlation as a function of temperature, which was displayed in table 4.3. This means at higher temperature, the pump required less power to transport the fluid as the density of the oil is lower, and vice versa. The physical properties of Paratherm and crude oil are much dependant on the temperature. The variation of inlet temperatures definitely changes the physical properties of the Paratherm.

4.3.5 Effect of inlet flowrate

The flowrate represents the behaviour of the oil flow. It can be seen through the calculation of Reynolds number. In Run 259, the only significant result of the effect in inlet temperature was the changes in differential pressure, dP2. The trending and analysis has been done in the previous section and shown in figure 4.16.

There are other significant effects of the variation of inlet flowrate, F2 into the test section such as heat transfer coefficient, the temperature profiles in the radial position and the effect on the boundary layer. However, these effects are more significant when the heat input is introduced to the test section and crude oil is performed. In these cases, the analysis will be discussed in the later section of this chapter and the effect of inlet flowrate will be incorporated.

4.3.6 Effect of heat input to test section

The Run 260 is designed specifically to monitor the effect of heat input towards the measured parameters which are installed surrounding the tubular test section as displayed in figure 4.1.
During this run, the temperature is controlled at 200 °C. The effect of heat input is tested at 4 stages of flowrate via a step change of control valve, V17 opening: 25 %, 50 %, 75 % and 100 %. At every stage, the heat input was introduced and all important parameters were monitored and recorded.

Figure 4.19 Study on the effect of dP2 at different delta T of test section

Figure 4.19 displays the effect of dP2 on the different temperature (between outlet temperature, T7 and inlet temperature, T5), delta T at inlet temperature of 200 °C. The tubular test section has been preheated with Joule heating at the similar current input. Theoretically, the effect of delta temperature shouldn't give much effect on the dP2 as the only difference which affected this dP2 is the difference in density. From these graphs, it confirmed that the measured differential pressure, dP2 is not affected from the heat input that had been introduced to the test section.
Figure 4.20 shows that the profile between the amount of heat input, power [kW] against the delta T at inlet temperature, T of 200 °C for different flowrate. The percentage values are representing the control valve, V17 opening. For the same amount of power, e.g. 5 kW, the delta T shows a significant increment for lesser flowrate. V17 opening of 100 % and 75 % [4.2 °C], 50 % [4.4 °C] and 25 % [6.2 °C]. In chapter 2, figure 2.21 showed the corrected flowrate between 70 % to 100 %, V17 opening has the changes less than 0.02 m3/h, therefore the mass flowrate for this range was relatively small compared with the mass flowrate for 50 % and 25 % of V17 opening. If the general assumption that the heat transfer from the heat input, Q will be transferred to the oil via convection as equation 4.1.

**Equation 4.1**

\[ Q = hA (T_w - T_{oil}) \]

Where \( h \) is heat transfer coefficient, \( A \) is the surface area, \( T_w \) is wall temperature and \( T_{oil} \) is the oil temperature.

As the test section was well insulated, it is assumed that the heat loss is relatively small or negligible compared with the heat being supplied. In this condition, the heat input in equation 4.1 is assumed to be equivalent to sensible heat that has been expressed in equation 4.2. The sensible heat, Q expression is given by;

**Equation 4.2**

\[ Q = mc_p \Delta T \]

Where \( m \) and \( c_p \) is representing the mass flowrate and specific capacity respectively.
The heat input, power is constant, the increment of mass flowrate will be inversely proportional to the delta T. From this heat balance, it confirmed the results that have been displayed in figure 4.21.

Figure 4.21 Effect of flowrate reading due to magnetic field

Figure 4.21 illustrates the effect of current that has been introduced at the test section on the oil flowrate, F2. At any given percentage of flowrate, the effect is entirely dependent on the amount of current that has been introduced. Every 100 A amount of current, the value of F2 decreases of amount 0.01 m³/h. This amount of bias shall take into account in the results analysis in order to obtain the correct value of derivative variables such as velocity, mass flowrate, and etc. It can also affect the operating condition if F2 is controlled in automatic mode. The effect can be seen in Figure 4.15 as at the initial stage of introducing the current to test section until it reached maximum value, the corrected flowrate is increased.

Figure 4.22 Response of V17 towards disturbance of F2 via joule heating
Another indication of this joule heating or magnetic effect has an effect towards the flowmeter is the response of control valve, V17 as shown in Figure 4.22. Even though the controller of flowrate, FC02 was put in automatic mode, the controller output which is V17 start to increase as joule heating is introduced. That means the magnetic effect is pushing the flowmeter reading and V17 increases to compensate the error between the setpoint and the measured flowrate.

Figure 4.23 Temperature profiles at important location of the rig when the experiment reaches steady state

Figure 4.23 shows the temperature profiles of oil at different locations of the rig. It also displays the heat input in the preheater and test section. As discussed previously, the preheater is used to obtain the desired inlet temperature and keep it consistent as the experiment is running. The heat input for tubular test section is to provide heat flux in order to induce fouling formation. As heat is supplied to test section, the return temperature, T8 will be increases; hence it will increase the tank temperature, T1 as well. When the system is disturbed, it will take sometimes to reach new equilibrium. This can be shown from minutes 340 to minutes 480. All temperatures profiles were increased until it reaches new equilibrium temperature. Despite of most of temperatures have reached its equilibrium a few minutes after the final power has been finalised at the test section, the tank temperature, T1 still takes some time to reach the equilibrium. Most probable reason, 85% of oil volume is located in the tank and it takes a little longer to stabilise the oil temperature. In these two steady state conditions, the calculation of heat loss can be calculated for tubular test section and the rest of the rig. The results can be
Baseline validation against experimental data on test section

Further validated against simulation results. This is among the key elements for baseline validation study.

![Wall temperature profiles for tubular test section](image)

**Figure 4.24 Wall temperature profiles for tubular test section**

The wall temperature profiles for tubular test section were shown in Figure 4.24 (a) against time and Figure 4.24 (b) against power input, kW. As the power input is introduced in step change of 100 A, the wall temperature increment following this changes. Despite of the constant current increment is introduced, the voltage is increasing as well. The main reason is the resistivity of the metal is a function of temperature as shown in Equation 4.3

Equation 4.3

\[
\beta = \beta_0 (1 + \alpha (T_{wall} - T_{25}))
\]

Where \(\beta\), \(\alpha\), \(T\) is resistivity, temperature coefficient and temperature of the metal respectively. Despite of the current input was constant for all range of inlet temperature, the amount of power increases as inlet temperature increases. The reason was the resistance increase as the wall temperature increases based on the equation 4.3.

**4.4 Model validation against experimental results**

In this section, the model presented in Chapter 3 is used to simulate the crude oil experiments previously described. Given measured inputs, the agreement between model prediction and measurements is tested for a number of outputs. It should be noted that none of the model parameters is fitted to experimental measurements.

**4.4.1 Model Set-up**

Inputs and outputs variables and fixed parameters are defined below.
Baseline validation against experimental data on test section

### Table 4.5 Time-varying model Inputs and outputs

<table>
<thead>
<tr>
<th>Model Inputs</th>
<th>Model Outputs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass flowrate (kg/s)</td>
<td>Outlet bulk T (ºC)</td>
</tr>
<tr>
<td>Inlet bulk T (ºC)</td>
<td>$T_{w,o}$ (ºC) at $z = 0.19, 0.54, 0.89, 1.25$ and $1.6$ m</td>
</tr>
<tr>
<td>Test section current (A)</td>
<td>$\Delta P$ (mbar)</td>
</tr>
<tr>
<td>Test section voltage (V)</td>
<td>Ambien T (ºC)</td>
</tr>
</tbody>
</table>

### Table 4.6 Fixed model parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
<th>Parameter</th>
<th>Value</th>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tube $D_i$ (m)</td>
<td>0.0196</td>
<td>Insulation $\rho$ (kg/m³)</td>
<td>120</td>
<td>Environment $h_o$ (W/m²K)</td>
<td>10</td>
</tr>
<tr>
<td>$D_o$ (m)</td>
<td>0.0229</td>
<td>$C_p$ (J/kg K)</td>
<td>840</td>
<td></td>
<td></td>
</tr>
<tr>
<td>L (m)</td>
<td>1.8</td>
<td>$\lambda$ (W/mK)</td>
<td>$1.46 \times 10^{-4}$ * $T_{ins}$ (K) - $9.28 \times 10^{-3}$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\lambda$ (W/mK)</td>
<td>38</td>
<td>Thickness (m)</td>
<td>0.03</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The start time for simulation is taken once the pump is activated (hence when there is a sufficiently high flowrate to avoid simulation failure). As a result, the start of the simulation is delayed compared to the experimental measurements. The model is initialized at steady state. Consequently, greater error between measured and estimated output variables is expected during the initial stages of the simulation.

The accuracy of the measurement is ±2.48 mbar for pressure drop and ±1.5% (or a minimum absolute error of 1.1ºC). The accuracy of the sensor determines the maximum experimental error. The model is considered to provide good prediction if the error between simulation results and measurements is within the accuracy of the sensor.

### 4.4.2 Run 259

#### Hydraulic Response

The pressure drop was estimated considering 3 correlations for friction coefficient. The comparison between experimental and simulated results is shown in Figure 4.25(a). The absolute error (simulated – measured) is shown in Figure 4.25(a). The best agreement is achieved for friction factor 3, for which the estimation is within the experimental error (±2.48
Baseline validation against experimental data on test section

mbar) for the first part of the experiment (until minute 300). Certain pattern in the error is observed, with a tendency towards greater positive error as temperature increases.

![Figure 4.25 Comparison of Pressure drop measurements and simulation results for Run 259 (a) and absolute error (simulated – measured) (b).](image)

**Thermal Response**

The thermal response of the model is tested at the outlet (bulk temperature) and at the outer surface of the tube. The comparison for outlet temperature is shown in Figure 4.26. The results show good agreement between the experiment and model prediction. The outlet temperature is slightly underestimated at the beginning, with the error gradually moving to positive values as the bulk temperature increases.

![Figure 4.26 Comparison of Paratherm outlet temperature measurements and simulation results for Run 259 (a) and absolute error (simulated – measured) (b).](image)

The wall outer surface temperature profile is shown in Figure 4.27(a). For simplicity, only the temperature for the midpoint of the tube (z=0.89m) is shown in the figure. The absolute error is shown in Figure 4.27(b) for the 5 thermocouples. As in the outlet temperature, the error moves...
from slightly negative values at the beginning (low temperature) to positive values when the highest temperatures are reached. Nonetheless, the wall temperature is estimated with an error within the sensor accuracy.

![Graph showing temperature variation over time](image1)

**Figure 4.27** Comparison of wall outer surface temperature measurements and simulation results at the tube midpoint (a) and absolute error for the 5 measurements (simulated – measured) (b), for Run 259.

### 4.4.3 Run 260

**Hydraulic Response**

The comparison between experimental pressure drop and simulated results is shown in Figure 4.28. As in the previous case, the best agreement considering the entire period is achieved for friction factor 3. In this case, the difference between measurement and simulation exceeds the experimental error (±2.48 mbar) during the body of the experiment, which correspond to the high temperature period. The average error is 5.36, 1.76 and -2.08 mbar for Factor 1, 2, and 3, respectively.

![Graph showing pressure drop variation over time](image2)

**Figure 4.28** Comparison of Pressure drop measurements and simulation results (a) and absolute error (simulated – measured) (b) for Run 260.
Thermal Response
The comparison for outlet temperature is shown in Figure 4.29. The results show very good agreement between experiment and model prediction. The outlet temperature is slightly overestimated (less than 1°C) during the period when oil is enters the test section at 200 °C. As in the previous run, a tendency towards increasing positive errors is observed in the error as temperature increases.

The wall outer surface temperature profile is shown Figure 4.30(a). For simplicity, only the temperature for the midpoint of the tube (z=0.89m) is shown in the figure. The absolute error is shown in Figure 4.30 (b) for the 5 thermocouples. As in the outlet temperature, the error moves from slightly negative values at the beginning (low temperature) to positive values when the highest temperatures are reached. Punctual peaks in the error are observed at some points both in outlet and wall temperature. The timing of these peaks corresponds to the changes in current/voltage input. This is due to a slight difference in the response time of the wall model to fast changes in power input compared to the real system. This affects the outlet temperature. Neglecting those punctual errors, the wall temperature is estimated with a maximum error of 11 to 8 °C for bottom and top thermocouples, respectively.
Baseline validation against experimental data on test section

4.4.4 Run 266

Hydraulic Response

The comparison between experimental and simulated results is shown in Figure 4.31. The pressure drop is overestimated in all cases, with the error systematically exceeding the experimental error (±2.48 mbar).

Thermal Response

The comparison for outlet temperature is shown in Figure 4.32. The results show very good agreement between experiment and model prediction. The outlet temperature is slightly overestimated (about 0.5 to 1 °C).
Baseline validation against experimental data on test section

Figure 4.32 Comparison of Paraterm outlet temperature measurements and simulation results (a) and absolute error (simulated – measured) (b) for Run 266.

The wall outer surface temperature profile is shown in Figure 4.33(a). For simplicity, only the temperature for the midpoint of the tube \((z=0.89\text{m})\) is shown in the figure. The absolute error is shown in Figure 4.33 (b) for the 5 thermocouples.

![Figure 4.33](image)

**Figure 4.33** Comparison of wall outer surface temperature measurements and simulation results at the tube midpoint (a) and absolute error for the 5 measurements (simulated – measured) (b), for Run 266.

### 4.4.5 Discussion

All the experimental runs have been validated against experimental results. For hydraulic response, all data have validated using 3 friction factor correlations which are stated in chapter 3. Based on the observation, the friction factor 3 has an excellent agreement compared to the other two friction factors especially in detecting the step changes of the flowrate. The error of this friction factor started to deviate more to positive error as the bulk temperature increase. Most probably the error has related to the coefficient in terms of hydrostatic pressure and some future works should consider this factor for analysis and/or measurement purposes.
For thermal validations, the outlet temperature shows an excellent agreement against experimental data for all experiments performed. However, the wall and outer wall temperature only shows excellent agreement when the test section is operated without the joule heating.

4.5 Conclusions

It is a huge success of commissioned and tested the HiPOR rig within the desired design parameter. All primary measurements had excellent reproducibility for all range of operating conditions. It is also shown that the heat input at the test section give less impact towards differential pressure compared with the friction loss. All the disturbance towards flowrate measurement such as different type of oil and an effect of magnetic field via joule heating have been detected and corrected for data analysis. A validation against experimental data also presented in this thesis. It showed that the differential pressure, DP [±2 mbar] and differential test section temperature, delta T [± 0.5 °C] give excellent agree, while the wall temperature, Tw only gives fair against the experimental data.
5 Test on crude oils

5.1 Introduction

Experiments of crude oil have been performed in 3 phases as shown in Figure 5.1: i) Cold run, with no heating; ii) Low temperature run, with bulk temperature controlled at 100 ºC and 150 ºC without heating at the test section; iii) Fouling run, with heat flux at the test section.

Table 5.1 Summary of Crude oil experiment

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Description</th>
<th>Temperature Range, ºC</th>
<th>Pressure Range, bar</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>RUN 400</td>
<td>Cold Run</td>
<td>50-70</td>
<td>5-10</td>
<td>Varies the flowrate</td>
</tr>
<tr>
<td>RUN 401</td>
<td>Low Temperature run without joule heating to test section</td>
<td>50-150</td>
<td>5-10</td>
<td>Designated test at temperature 100 ºC, 150 ºC</td>
</tr>
<tr>
<td>RUN 402</td>
<td>Fouling RUN (72 hours)</td>
<td>60-200</td>
<td>5-25</td>
<td>Flowrate = 1.2 m3/h Temperature= 200 ºC Current = 900 A</td>
</tr>
</tbody>
</table>

The crude oil properties had been supplied by BP and their basic properties as shown in Table 5.2

Table 5.2 Basic properties of two crude oils and Paratherm

<table>
<thead>
<tr>
<th></th>
<th>Heavy oil, HIPOR OIL B/1</th>
<th>Light oil, HIPOR OIL B/2</th>
<th>Paratherm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (kg/m3), 15 ºC</td>
<td>974.8</td>
<td>789.1</td>
<td>887</td>
</tr>
<tr>
<td>Density (API)</td>
<td>13.6</td>
<td>47.8</td>
<td></td>
</tr>
<tr>
<td>Dynamic Viscosity, cSt at 20 ºC</td>
<td>46843</td>
<td>2.17</td>
<td>45</td>
</tr>
</tbody>
</table>

Table 5.3 below reports the viscosity of the two crude oils (from BP supplied assays), as well as of Paratherm, up to 100C.
Table 5.3 Kinematic viscosity of oils at various temperatures

<table>
<thead>
<tr>
<th>Temperature, °C</th>
<th>Light oil HIPOR OIL B/2</th>
<th>Viscosity, mm²/s</th>
<th>Heavy oil HIPOR OIL B/1</th>
<th>Paratherm</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>2.17</td>
<td>46843</td>
<td>50</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>1.85</td>
<td>14167</td>
<td>28</td>
<td></td>
</tr>
<tr>
<td>40</td>
<td>1.6</td>
<td>5055.7</td>
<td>18</td>
<td></td>
</tr>
<tr>
<td>50</td>
<td>1.4</td>
<td>2071.6</td>
<td>12</td>
<td></td>
</tr>
<tr>
<td>60</td>
<td>1.24</td>
<td>9534.6</td>
<td>9.1</td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>0.83</td>
<td>99.19</td>
<td>3.7</td>
<td></td>
</tr>
</tbody>
</table>

Table 5.3 displays the kinematic viscosity of oils at various temperatures ranging from 20 °C to 100 °C. Data of crude oils were taken from Material Safety Data Sheet (MSDS) from British Petroleum (BP) and Paratherm Data from its manufacturer.

5.2 Cold run experiment

Based on discussion with industrial research collaborators, the desired blend oil for this experiment is 50:50 by mass fraction. It is assumed that the blended crude oil will have similar behaviour and operating response to Paratherm. As this is the first time, crude oil is tested in the HiPOR, it is safer to test it at low temperature before running the experiment at higher temperature.

5.2.1 Experiment descriptions and objectives

Cold crude oil run experiment is run at the minimum temperature. The initial start-up procedure has a similarity with Paratherm run, Run 235 and Run 236. The main objective of this run is to capture the response of differential pressure, dP2 at various valve openings and thermal behaviour, especially the heat input that generated from the main pump, M1. Referring to Table 5.3, the calculated kinematic viscosity of the crude oil is nearly 50 mm²/s, which was closer to Paratherm at 20 °C. Therefore, the run has been decided to start at the same temperature as Run 235, 70 °C in order to avoid overloading of current for motor M1.

5.2.2 Experiment protocol

A general experimental procedure for cold run has been explained thoroughly in chapter 3. The differences are the oil that being tested and tank pressure, P1. P1 need to be kept higher, so the tank pressure always higher than crude oil vapor pressure. This is to avoid the release of light hydrocarbon into the atmosphere, hence created hazardous environment, which may provoke into fire and/or explosion. The schedule for the experiment was developed according to SOP which essentially starts with a preliminary check for the entire rig. After the preliminary check
of the rig, activated the nitrogen generator, chiller for cooling water supplied, CO\textsubscript{2} suppression system, rapid ventilation, then the rig is ready for operation. The start-up procedure is started with activated the LabView interface for monitoring and controlling purposes. Run number (referring to the first column in Table 5.1) will be keyed in as the approved by in the permit to operate. Control valve, V17 is opened manually as desired opening which allows the cooling water is flowing from the chiller to the bowman heat exchanger in cooling loop. Then, the cooling pump, M2 was put in operation. This cooling loop is designed to reduce the outlet temperature of testing oil from the test section. This loop is also used for shutdown purposes in the hot run experiment. As the protection and safety of the rig, the main pump, M1 cannot put in operation (interlock) without the signal of cooling pump, M2. Subsequently, the main loop which is the test section will put into service. First, the isolation valves, V3 and V22 will be opened. This allows the oil is flowing from the main tank to the test section and return back the main tank. It was the closed loop experiment where both valves were used to isolate the main tank from the circulation loop. The main pump, M2 was used to transport the oil from the tank and return back into the tank. Before M2 is started, control valve, V17 is initially opened at 10% opening. This allows the oil to be circulated and prevent damage to the pump. The flow is increased by increases the opening of V17. In this experiment, V17 is opened at intervals of 10% until it reaches 100%. The experiment was held for approximately 2 hours before the shutdown procedure has taken place. All thermal and hydraulic are monitored and recorded within LabView environment.

5.3 Low temperature run

5.3.1 Experiment descriptions and objectives

The low temperature run experiment for crude oil is crucial as this run could be considered as a commissioning process as well. The experiment is designed without joule heating introduced in the test section. Based on previous experiment runs with Paratherm, it is noted that a systematic monitoring system needs to be introduced for a few critical parameters. One of important parameters needs to be monitored is the tank pressure. Based on ideal gas law, the increment of pressure in the tank shall be proportional to the temperature. Therefore, the ramping of temperature from one to another should be closely monitored. As the tank was blanketed with nitrogen, any abrupt change may be caused by the release of light hydrocarbon from the crude oil.

Amongst the objectives of Run 401 are to see the dP2 profile against temperature and flowrate. The data will be used for validation of the model. Preheater was used to increase the inlet temperature, T5 to the desired temperature and temperature control system, TC02 is used to keep the temperature consistent at the designed test (flow variation).
5.3.2 Experiment protocol
The initial start-up of a low temperature run experiment is similar with a cold run experiment. The only difference was the heat input is introduced via preheater section in order to increase the oil temperature to the desired inlet temperature. In Run 401, the inlet temperature, T5 was increased to 100 °C and 150 °C. At the mentioned temperature, the flowrate were varied by increasing the control valve, V17 opening at the interval of 10 % until it reaches 100 % opening. The analysis of the friction loss and the thermal profiles due to flowrate can be done and discussed in the later section of this chapter. Furthermore, the analysis of the characteristic of the valve at a different inlet temperature also can be performed.

As mentioned earlier in Run 259, 260 and 266, the shutdown procedure needs to be executed carefully in order to protect the process as well as the equipment. The cyclic cooling process can be immediately introduced as the highest temperature of Run 401 is 150 °C. The temperature will steadily decrease and experiment will be stopped once the temperature has decreased to 70 °C.

5.4 Crude oil fouling run
HiPOR is designed for the crude oil fouling study. The operating conditions set for this experiment is based on the recommendation by British Petroleum (BP) and is shown in Table 5.4.

Table 5.4 Operating conditions for crude oil fouling run

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Description</th>
<th>Inlet Temperature, °C</th>
<th>Wall Temperature, °C</th>
<th>Pressure Range, bar</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>RUN 402</td>
<td>72 hours Run</td>
<td>200</td>
<td>250</td>
<td>5-25</td>
<td>Flowrate = 1.2 m3/h</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Temperature= 200 °C</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Current = 950 A</td>
</tr>
</tbody>
</table>

5.4.1 Experiment descriptions and objectives
The crude oil fouling run is the ultimate run in order to test the design of HiPOR. Beside getting fouling deposition, the other objective is able to operate the rig safety and recorded the primary measurements consistently for long hours experiment.

5.4.2 Experiment protocol
The initial start-up of crude oil fouling run experiment is similar to the hot run experiment with joule heating in the test section. As the initial start-up procedures were the same for all experiments, the hot run requires the heat input via preheater section in order to increase the oil temperature to the desired inlet temperature. Since the flowrate, F2 needs to be controlled at 1.2 m3/h, the valve, V17 is opened up to 25 %. Once the measured flowrate reaches the desired F2, the control system for flowrate, FC02 is changed from manual to automatic. In Run 402, the
inlet temperature, T5 was increased to 200 °C gradually and tank pressure is kept higher than crude oil vapour pressure. This can be seen in the results section.

Once T5 reached 200 °C, the temperature control system, TC01 is switched to automatic. It takes some times for the heat input to stabilise because the return temperature is still increasing, hence increase the tank temperature, T1. At one point, T1 is stabilised and heat input at preheater will be stable and consistent. At these conditions, the current is introduced at the test section with a step change of 100 A until it reached the value of 900 A. The thermal effect shall be seen in the outlet temperature, T5 as well as the wall temperatures; T20, T12, 6, T21 and T11. The hydraulic effect shall be captured via the differential transmitter, dP2 which measuring the pressure difference between the inlet and outlet of the test section. All relevant primary instrumentations are shown in figure 4.1.

The shutdown procedure will be the same procedures as described in Run 260.

5.5 Experimental results

5.5.1 Cold Run (400)

- Show temperature profile at the tank at initial start-up until end of experiment
- Explain in details the decrement of temperature and flow increment over time
- Step change of flowrate and other information can be extract such as control valve behaviour (compare to non-fouling run), current motor profile (initial assumption on density of blended oil), increment of temperature due to heat input from the pump, hydraulic profile due to increment of flowrate, the calculation of corrected flowrate for crude oil based on discussion in chapter 2.

Generally the cold run was run as part of commissioning process after several runs of HiPOR rig with non-fouling oil. The blended oil was mixed with mass ratio of 50:50. The main idea was the aim of physical properties of blended oil would be similar with non-fouling oil. Therefore, the physical plant could be practically and ready to operate for blended crude oil at the desired operating conditions which has been proved with non-fouling oil.
Important monitoring parameters for crude oil study (a) Temperature and Pressure (b) Flowrate and Differential Pressure

Figure 5.1 shows the entire process of cold run experiment. The experiment time was only 2 hours. The main aim was to commission the rig with blended crude oil. Part of the commissioning was to test and verify the instrumentations as well as control valve. When the pump, M1 started, T1 was spiked for a few seconds before it starts to decrease. The most probable reason for the spike in temperature was the location of thermocouple T1. T1 was located at the bottom of the tank. It has the same behaviour as shown in cold run experiment of non-fouling oil in Figure 4.3. Figure 5.1 (a) shows slight decrement of temperature at after the spike of temperature. The main reason was initially, the blended oil at tank has temperature of 60 °C compare to other sections of the rig, which has oil temperature at ambient temperature. As shown in Figure 5.1 (a), the tank pressure, P1 is increased in step change of 5 bar. P1 was kept high, to avoid the release of light gases in the blended crude oil. As the pump circulating the blended oil and the entire oil temperature is uniform, the oil temperature starts to increase. Despite no heating is supplied to entire rig, the increment of oil temperature was due to heat input from the main pump that circulated the oil for the rig. Figure 5.1 (b) shows the flowrate and differential pressure when valve opening, V17 was adjusted accordingly.

5.5.2 Low temperature run (401)

This experiment was done purposely to check the safety of the system, to compare with non-fouling run and quantifying heat losses at desired temperature, 100 °C and 150 °C.
Figure 5.2 shows the overview of important parameters for crude oil which had run and controlled at low temperature. In this experiment, the inlet temperature was controlled at 100 °C and 150 °C. At both temperatures, valve opening of V17 was manipulated between 10% to 100%. The aims are to check flow characteristic of blended oil, to test the response of temperature control for a given disturbance and to monitor the temperature profiles of test section at different flowrate. The results will be discussed later in this chapter.

- Show the entire run 401 from the start-up activities until shutdown. How the temperature was controlled and other tests have been carried out within this experiment. The wall temperature, differential pressure and flowrate profiling at different valve opening for both desired inlet temperature.
- Heat losses could be easily quantified at steady state conditions rather than transient conditions. The noise of important controlled parameter could also been observed through this experiment. Estimation of calculation through all measured parameters, heat input. Initial approaches were using a well-known formulation heat balance by identifying the relevant input of output of the entire system.

**Hydraulic profile**

One of the important factor to monitor the formation of fouling deposition is hydraulic profile. Since the test section installed in HiPOR was in vertical position, the hydraulic profile is not only influenced by friction factor, but it is also effected by hydrostatic pressure. Several tests have been performed to validate the measurement of hydraulic profile and their characterisations at different inlet temperature and variation of flowrate.
Tests Run on Crude Oils

Figure 5.3 Differential pressure, dP2 at (a) different valve opening (b) flowrate

Figure 5.3 shows the measured differential pressure for different valve opening and flowrate for inlet temperature of 100 °C and 150 °C. It is noted that there is no heat input for the test section for this experiment. The differential pressure will be corresponded to hydrostatic pressure and the friction pressure. As the valve opening is increased, differential pressure is assumed to be responded to friction pressure as hydrostatic pressure would be the same at constant inlet temperature.

Temperature profile

In this experiment, no joule heating was introduced at test section. Amongst the aims were to validate the hydraulic profile based on friction factor at constant inlet and outlet temperature and estimate the heat losses of temperature to the environment due to rapid ventilation system which has been installed within the rig system to comply with safety regulation and operation. As mentioned earlier, temperature or thermal profiles of the test section are very crucial for crude oil fouling study. They only give indications of heat transfer that has been introduced or removed (based on changes in wall and outlet temperatures), but it can later be used for data analysis on fouling studies. It is very useful to obtain data that quantifying the heat losses without any heating been introduced before the crude oil fouling experiment is being performed.
Tests Run on Crude Oils

Figure 5.4 Temperature profiles for Run 401 (a) inlet temperature, T5 (b) wall temperature, T11, T20, T6, T12 and T20

Figure 5.4 shows the temperature profiles for (a) inlet temperature, (b) wall temperature, T11, T20, T6, T12, and T20. Wall temperature profiles amongst important parameters need to be monitored and captured for crude oil fouling study. These thermal profiles give indication of heat transfer occurrence from the source (joule heating) to the oil inside the test section. In theory, any deviations of thermal profiles may give indication of the phenomena of fouling. As discussed before, lack of primary measurements measuring every moment of the process would bring difficulties to identify the stages of fouling formation starting from induction, transportation, initial deposition i.e. gel formation and ageing process. Most papers only discuss the negative fouling period at initial stage of fouling study and subsequently the fouling rate has increased exponentially until it reaches equilibrium/ asymptotic.

5.5.3 Fouling run (402)

An experimental run has been designed in order to study crude oil fouling. The crude oil properties and desired operating temperature had been shown in Table 5.4. The overall results will be presented in Figure 5.5, Figure 5.6, Figure 5.7 and Figure 5.8.
Figure 5.5 Temperature profiles of wall temperature, $T_w$ for (a) start-up phase (b) introduction of heat input at test section

Figure 5.5 shows the temperature profiles for crude oil Run 402 at start-up phase (a) and the introduction of heat input at test section (b). The wall temperature increase as heat input increase. Even though the current input in the intervals of 100 A, the amount of heat input, kW is a square function of current value.

Figure 5.6 Details of $T_w$ profile for crude oil run

Figure 5.6 displays the details wall temperature profiles at maximum heat input for approximately 66 hours of experiment. No significant changes towards the behaviour of these temperature.
Figure 5.7 Differential pressure, dP2 profiles for Run 402

Figure 5.7 shows the differential pressure, dP2 for Run 402 for the entire experiment. At maximum heat input, there is no significant changes in dP2. Since no change in wall temperature and differential pressure, it gives the strong indications that no fouling deposition occurring during experiment.

Figure 5.8 Temperature profiles for inlet, T5 and outlet, T7

Figure 5.8 shows the temperature inlet, T5 and outlet, T7 for the entire experiment run. The only changes can be seen was when the test section being introduced to joule heating until it reached maximum value.
5.6 Model validation against experimental results

In this section, the model presented in Chapter 3 is used to simulate the crude oil experiments previously described. Given measured inputs, the agreement between model prediction and measurements is tested for a number of outputs. It should be noted that none of the model parameters is fitted to experimental measurements. The hydraulic, differential pressure of test section and thermal effects, outlet temperature and outer surface temperature will be presented.

5.6.1 Model Set-up

Inputs and outputs variables and fixed parameters are defined below.

Table 5.5 Time-varying model Inputs and outputs

<table>
<thead>
<tr>
<th>Model Inputs</th>
<th>Model Outputs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass flowrate (kg/s)</td>
<td>Outlet bulk T (ºC)</td>
</tr>
<tr>
<td>Inlet bulk T (ºC)</td>
<td>T_{w,o}(ºC) (at z = 0.19, 0.54, 0.89, 1.25 and 1.6 m)</td>
</tr>
<tr>
<td>Test section current (A)</td>
<td>Δ P (mbar)</td>
</tr>
<tr>
<td>Test section voltage (V)</td>
<td>T_{in,c,l}(ºC) (at z = 0.19, 0.89 and 1.6 m)</td>
</tr>
<tr>
<td>Ambien T (ºC)</td>
<td></td>
</tr>
</tbody>
</table>

Table 5.6 Fixed model parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
<th>Parameter</th>
<th>Value</th>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
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<td></td>
<td>Insulation</td>
<td>Crude Oil Properties</td>
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<td></td>
</tr>
<tr>
<td>Tube D_i (m)</td>
<td>0.0196</td>
<td>ρ (kg/m³)</td>
<td>120</td>
<td>API</td>
<td>30.55</td>
</tr>
<tr>
<td>D_o (m)</td>
<td>0.0229</td>
<td>C_p (J/kg K)</td>
<td>840</td>
<td>MeABP (ºC)</td>
<td>350</td>
</tr>
<tr>
<td>L (m)</td>
<td>1.8</td>
<td>λ (W/mK)</td>
<td>1.46 x 10^{-4} *</td>
<td>T_{ins} (K) - 9.28 x 10^{-3}</td>
<td>T_{38ºC} (cSt)</td>
</tr>
<tr>
<td>λ (W/mK)</td>
<td>38</td>
<td>Thickness (m)</td>
<td>0.03</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Environment</td>
<td></td>
<td>l_h (W/m²K)</td>
<td>10</td>
</tr>
</tbody>
</table>

The start time for simulation is taken once the pump is activated (hence when there is a sufficiently high flowrate to avoid simulation failure). As a result, the start of the simulation is delayed compared to the experimental measurements. The model is initialized at steady state. Consequently, greater error between measured and estimated output variables is expected during the initial stages of the simulation. In addition to the measurements in the experiments in Chapter 4, 3 thermocouples where installed to measure the outer surface temperature of the
insulation. These measurements are here used to provide additional validation of the thermal model.

As discussed in the previous chapter, the accuracy of the measurement is ±2.48 mbar for pressure drop and ±1.5% (or a minimum absolute error of 1.1°C). The accuracy of the sensor determines the maximum experimental error. The model is considered to provide good prediction if the error between simulation results and measurements is within the accuracy of the sensor.

As commented in the previous section, no indication of fouling was observed in any of the runs, both in the measurement trends or in observed status of the tube inner surface after the hot run (Run 402). As a result, no fouling is considered in the simulations.

5.6.2 Cold Run (400)

Hydraulic Response

The pressure drop was estimated considering 3 correlations for friction coefficient, as in Chapter 4. The comparison between experimental and simulated results is shown in Figure 5.9(a). The absolute error (simulated – measured) is shown in Figure 5.9(b). In this case, the best agreement is achieved for friction factor 2, for which the estimation is within the experimental error (±2.48 mbar) for most of the period. Good agreement is also achieved for Friction Factor 1, but significant deviation is observed for Friction Factor 3.

![Figure 5.9 Comparison of Pressure drop measurements and simulation results for Cold Run (a) and absolute error (simulated – measured) (b).](image)

Thermal Response

The thermal response of the model is tested at the crude oil outlet (bulk temperature) and at the outer surface of the tube. As discussed earlier, these two are the main thermal variables used for fouling monitoring. As a double check, the temperature at the surface of the insulation is also
compared. The experimental error for the thermocouples is ±1.5% (or a minimum absolute error of 1.1°C).

The comparison for outlet temperature is shown in Figure 5.10. The results good agreement between experiment and model prediction. The outlet temperature is slightly underestimated over the entire period, but well within the experimental error.

![Comparison of Crude oil outlet temperature measurements and simulation results for Cold Run](a) and absolute error (simulated – measured) (b).

The wall outer surface temperature profile is shown Figure 5.11(a). For simplicity, only the temperature for the midpoint of the tube (z=0.89m) is shown in the figure. The absolute error is shown in Figure 5.11(b) for the 5 thermocouples. The greater errors are shown at the beginning of the simulation, which could be due to a wrong choice of initial conditions for the simulation. Once the system stabilizes, the temperatures are systematically underestimated within the experimental error.

![Comparison of wall outer surface temperature measurements and simulation results at the tube midpoint (a) and absolute error for the 5 measurements (simulated – measured) (b), for Cold Run.](a)
The insulation outer surface temperature profile is shown Figure 5.12(a). For simplicity, only the temperature for the midpoint of the tube (z=0.89m) is shown in the figure. The absolute error is shown in Figure 5.11(b) for the 3 thermocouples located at 0.19, 0.89 and 1.6 m from the tube entrance, respectively. The insulation temperature shows more noise than the temperature measurements. These measurements are exposed to the rapid ventilation of the enclosure, and therefore this will affect the readings.

The results show agreement within the experimental error for the measurement at the midpoint. The deviation is greater for the top and bottom measurements. However, given the noise in the measurements and the lack of a pattern in the temperatures, these results are considered to be good enough. Further research on the definition of the outer heat transfer coefficient and air distribution in the enclosure could help to improve the results.

Figure 5.12 Comparison of insulation outer surface temperature measurements and simulation results at the tube midpoint (a) and absolute error for the 5 measurements (simulated – measured) (b), for Cold Run.

5.6.3 Low Temperature Run (401)

Hydraulic Response

The comparison between experimental and simulated results is shown in Figure 5.13. As in the previous case, the best agreement considering the entire period is achieved for friction factor 2. In this case, the difference between measurement and simulation exceeds the experimental error (+2.48 mbar) during some stages of the simulation. With Friction Factor 1 the pressure drop is overestimated through the experiment. On the other hand, Friction Factor 3 shows good agreement after 100 min. The average error is 5.36, 1.76 and -2.08 mbar for Factor 1, 2, and 3, respectively.
Tests Run on Crude Oils

Thermal Response

The comparison for outlet temperature is shown in Figure 5.14. The results show very good agreement between experiment and model prediction. The outlet temperature is slightly underestimated (less than 0.5 ºC) for oil temperatures of 100ºC, very slightly overestimated for higher temperatures.

The wall outer surface temperature profile is shown in Figure 5.15(a). For simplicity, only the temperature for the midpoint of the tube (z=0.89m) is shown in the figure. The absolute error is shown in Figure 5.15 (b) for the 5 thermocouples. As in the cold run, once the system stabilizes the temperatures are predicted within the experimental error. A pattern in the error is observed, with similar time profile as the error for the outlet bulk temperature.
Figure 5.15 Comparison of wall outer surface temperature measurements and simulation results at the tube midpoint (a) and absolute error for the 5 measurements (simulated – measured) (b), for Low Temperature Run.

The insulation outer surface temperature profile is shown in Figure 5.16(a). For simplicity, only the temperature for the midpoint of the tube (z=0.89m) is shown in the figure. The absolute error is shown in Figure 5.16 (b) for the 3 thermocouples located at 0.19, 0.89 and 1.6 m from the tube entrance, respectively. As in the cold run, the results show agreement within the experimental error for the measurement at the midpoint and the deviation is greater for the top and bottom measurements.

Figure 5.16 Comparison of insulation outer surface temperature measurements and simulation results at the tube midpoint (a) and absolute error for the 5 measurements (simulated – measured) (b), for Low Temperature Run.

5.6.4 Hot Run (402)

Hydraulic Response

The comparison between experimental and simulated results is shown in Figure 5.17. The pressure drop is overestimated in all cases, with the error systematically exceeding the
experimental error (±2.48 mbar). The average error is 7.77, 5.26 and 3.47 mbar for Factor 1, 2, and 3, respectively.

**Thermal Response**

The comparison for outlet temperature is shown in Figure 5.18. The results show very good agreement between experiment and model prediction. The outlet temperature is slightly overestimated (about 0.5 ºC).

The wall outer surface temperature profile is shown Figure 5.19(a). For simplicity, only the temperature for the midpoint of the tube (z=0.89m) is shown in the figure. The absolute error is shown in Figure 5.19 (b) for the 5 thermocouples. The temperatures are predicted within the
experimental error, although with values close to the lower limit (temperature underestimated) for all measurements but that at the bottom.

![Figure 5.19](image1)

**Figure 5.19** Comparison of wall outer surface temperature measurements and simulation results at the tube midpoint (a) and absolute error for the 5 measurements (simulated – measured) (b), for Hot Run.

The insulation outer surface temperature profile is shown Figure 5.20(a). For simplicity, only the temperature for the midpoint of the tube (z=0.89m) is shown in the figure. The absolute error is shown in Figure 5.20(b) for the 3 thermocouples located at 0.19, 0.89 and 1.6 m from the tube entrance, respectively. As in the other runs, the results show agreement within the experimental error for the measurement at the midpoint and the deviation is greater for the top and bottom measurements.

![Figure 5.20](image2)

**Figure 5.20** Comparison of insulation outer surface temperature measurements and simulation results at the tube midpoint (a) and absolute error for the 3 measurements (simulated – measured) (b), for Hot Run.

### 5.6.5 Discussion

All the experimental runs have been validated against experimental results. For hydraulic response, all data have validated using 3 friction factor correlations which stated in chapter 3.
Based on the observation, the second friction factor has an excellent agreement compared to the other two friction factors especially in detecting the step changes of the flowrate. The error of this friction factor started to deviate more to positive error as the bulk temperature increase. Most probably the error is related to the coefficient in terms of hydrostatic pressure and has the similarity responses on Paratherm runs. Some future works should consider this factor for analysis and/or measurement purposes.

For thermal validations, the outlet temperature shows an excellent agreement against experimental data for all experiments performed. However, the wall and outer wall temperature only shows excellent agreement when the test section is operated without the joule heating.

5.7 Impact of fouling on experimental measurements

As discussed in previous section, the variables used to monitor fouling were observed to stay undisturbed along the Hot Run (Run 402). This run was carried out under operating conditions for which fouling was expected, but the stability of the measurements indicated that fouling was not occurring or, at least, that the extent of fouling was not enough to impact the measured variables. In fact, no fouling was visually observed on the tube inner surface after the experiment. This could be due to:

a) Crude oil fouling slow under operating conditions to significantly affect the measurable variables.

b) Existence of an initiation period that delays the appearance of fouling.

c) Fouling conditions on the No Fouling side of the Threshold.

In order to bring insight into the above hypotheses and guide future experiments, in this section the model for the test section of HiPOR is run considering fouling in order to assess the expected impact of the deposition on the measurements for the timeframe of Run 402. The results are compared to the simulation results without fouling. The model is run considering the actual inputs of the experiments. The inputs were only modified during the emergency shutdown after 12h of experiments, which may cause numerical instability. Additionally, the fouling model in Chapter 3 is extended to include an initiation period based on models in literature. Results using these models are also included in the comparison.

5.7.1 Hot Run Considering Fouling

Typical fouling parameters, obtained based on Refinery plant data in a previous work (Coletti and Macchietto 2011), are assumed. Ageing was neglected.
Table 5.7 Fouling parameters (Coletti and Macchietto, 2011)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha$ (m$^2$ K$^{-1}$)</td>
<td>$1.65 \cdot 10^{-3}$</td>
</tr>
<tr>
<td>$\gamma$ (m$^4$ K$^{-1}$ N$^{-1}$)</td>
<td>$9.28 \cdot 10^{-13}$</td>
</tr>
<tr>
<td>$E_f$ (kJ mol$^{-1}$)</td>
<td>28.5</td>
</tr>
</tbody>
</table>

Simulations were run neglecting and considering fouling and using the actual inputs from Run 402 (Hot Run).

The average fouling resistance over time and thickness at various locations is shown in Figure 5.21. After 72h, and according to the assumed fouling model, a thickness of 35μm would be expected leading to a maximum fouling resistance of 0.175 m$^2$K/kW. It should be noted that an initial value of $10^{-6}$m was assumed for the deposit thickness for initialization purposes. This thickness was checked to be small enough so as not to affect the simulation results.

The effect of the growing fouling film is discussed for the three monitoring variables in HiPOR: pressure drop, wall temperature, and outlet bulk temperature.

The comparison for pressure drop is shown in Figure 5.22. Fouling leads to a gradual deviation in pressure drop. However, the deviation is very small (maximum deviation of 0.24 mbar in the 72 h experiment), and therefore undetectable for the current accuracy of the pressure drop sensor.
Figure 5.22 Comparison of Pressure drop simulation results neglecting and considering fouling (a) and absolute error (Fouling - No Fouling) (b) for Hot Run.

The outlet temperature slightly affected by fouling, with a decrease of less than 0.01ºC. This is expected, since the heating section in HiPOR is designed to work in uniform heat flux conditions. For such design, the thermal effect of fouling is expected to be reflected on the wall temperature. The comparison for the wall outer surface temperature is shown in Figure 5.23.

Figure 5.23 Wall outer surface temperature simulation results at the tube midpoint (a) and absolute error for the 5 measurements (Fouling - No Fouling) (b), neglecting and considering fouling for Hot Run.

The results show how that if fouling had developed as described by the model, it would have been possible to detect such deposition in the wall temperature at least during the second half of the experiment. The total deviation reaches about 7ºC which is more than double the temperature sensor accuracy.

As a result, it can be concluded that the fouling model does not capture the deposition phenomena during the initial stages. Of course, this depends on the fouling parameters used in the simulation.
However, the non-development of a fouling layer agrees with the visual observation of the tube after the experiment, and also with the fouling behaviour observed in other experimental works in literature that report the existence of an initiation period. This is explored in the next section.

5.7.2 Hot Run Considering Fouling with Induction Period

In the previous section it was shown how for typical fouling parameters the impact of fouling should have been noticed within the 72 h experiment. In that simulation fouling is assumed to start building-up as soon as hot crude oil fouling flows through the tube. However, a period during which fouling is not observed is reported by many authors. This is interpreted as an “initiation” period during which the heat transfer surface is conditioned (e.g. formation of a monolayer) prior to fouling build up.

Here, the model by Yang et al. (2012) is adapted to the distributed model in Chapter 3. In that paper the authors proposed a model for the induction (or initiation or pre-conditioning) period by defining the fractional coverage of the heat transfer surface, $\theta$. The fouling rate in terms of fouling resistance is calculated as follows:

\begin{equation}
\frac{dR_f}{dt} = \theta \dot{R}_f
\end{equation}

Where $\dot{R}_f$ is the fouling rate given by empirical expressions such as that by Panchal et al. (1997). In reality, and considering local effects, the layer starts growing immediately after the surface is covered. The model attempts to capture the overall growth, by considering that the average fouling rate is very slow until most of the surface is covered. The fractional coverage varies over time according to the following kinetics, which considers a growth and a removal term:

\begin{equation}
\frac{d\theta}{dt} = k_1 \theta(1 - \theta) - k_2 \theta
\end{equation}

In their paper, Yang et al. (2012) neglect the removal term (second term on the right hand side) and fit the model to crude oil fouling experimental data for several experiments at various temperatures. As commented by the authors, an initial very small but non-zero coverage is required, which can be interpreted as few active spots that are instantaneously covered by the foulant. The reported experimental parameters are shown in the table below.
Tests Run on Crude Oils

Table 5.8 Initiation parameters Yang et al. (2012)

<table>
<thead>
<tr>
<th>Experiment</th>
<th>T_s (°C)</th>
<th>c</th>
<th>k_1 (h⁻¹)</th>
<th>k_2 (h⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>376</td>
<td>8800</td>
<td>6.03</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>385</td>
<td>8800</td>
<td>6.41</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>411</td>
<td>8800</td>
<td>11.9</td>
<td>0</td>
</tr>
</tbody>
</table>

T_s is the surface temperature and the parameter c is an integration constant which can be related to the initial coverage θ₀ of the surface as follows:

Equation 5.3

\[ \ln \left( \frac{k_1 - k_2}{k_1} \right) = \ln(c) \]

Here, the expressions above are adapted to the distributed model in Chapter 3. The fouling rate, in terms of variation of deposit thickness is:

Equation 5.4

\[ \frac{d\delta(z)}{dt} = \theta(z) \lambda L_{0} \frac{dR_f}{dt}(z) \]

Where \( \frac{dR_f}{dt} \) is given by the correlation by Panchal et al. (1997). Assuming negligible removal term in Eqn. (5.2), the rate of coverage is:

Equation 5.5

\[ \frac{d\theta(z)}{dt} = k_1(z)\theta(z)(1 - \theta(z)) \]

Finally, by using the data in the previous table, an expression for the kinetic constant as function of the local wall temperature can be devised:

Equation 5.6

\[ \ln(k_1(z)) = 15.709 - \frac{9064.6}{T_s(z)} \]

where \( k_1 \) is in h⁻¹ and \( T_s \) in K.

The simulation in the previous section were run again but this time including the effect of surface coverage. The fouling resistance and deposit thickness are shown in Figure 5.24, where the results are compared to the previous simulation without initiation. The results show that, for the conditions of Run 402, the experiment is not long enough to reach the end of the
initiation period and therefore to develop a noticeable fouling layer. The results show how the deposit thickness starts growing just at the end of the experiment. This is explained by the fraction of coverage, shown for various location in Figure 5.25. The fractional coverage starts growing significantly at the end of the experiment, reaching values of 0.24 at the end of the tube.

![Figure 5.24 Simulated tube average fouling resistance (a) and deposit thickness (b) for Hot Run conditions, considering fouling (no initiation) and fouling with initiation period.](image)

![Figure 5.25 Simulated fractional coverage over time for Hot Run.](image)

With initiation no deviation is observed in the pressure drop nor in the bulk outlet temperature. The deviation in the wall temperature is negligible even at the end of the period when fouling starts building up, as shown below.
The results considering initiation agree with the experimental observations. The question is whether, under the operating conditions in Run 402 and assuming that the initiation model is correct, it would have been possible to observe fouling if the experiment had been run for a longer time period. In order to address that question a modified Run 402 (here called Sim 403) is simulated with the maximum heat flux period lasting double the time of that in the original experiment, this is, Joule heating with 900A and 5.28V for 132h instead of the original 66h.

The simulation results for average fouling resistance, deposit thickness and fractional coverage are shown below. The results show that the fractional coverage quickly changes after 4000 min, reaching values near 1 after 7000 min. The deposit thickness grows in the last part of the experiment to values between 20 and 30μm, leading to an overall fouling resistance of 0.125 m2K/kW at the end of the experiment.
Figure 5.28 Simulated fractional coverage over time for Hot Run with Joule heating for 132h (Sim 403).

Figure 5.29 Wall outer surface temperature simulation results at the tube midpoint (a) and absolute error for the 5 measurements (Fouling – No Fouling) (b), considering fouling with initiation for Hot Run with Joule heating for 132h (Sim 403).

The deviation in pressure drop is very small, with a maximum value of 0.17 mbar and, therefore, undetectable. On the other hand, the development of the fouling layer leads to a deviation in the wall temperature which exceeds the sensor accuracy after 7000 s at the top of the tube.

As a result, for the initiation model and fouling parameters assumed in this case study, the results show that an experiment such as Sim 403 could lead to noticeable deposition if the Joule heating period is extended from 66 to 132h. Fouling could be potentially detected based on wall temperature.

Although the above proposed prolonged experiment could be used to as a proof of concept for HiPOR to be used as a fouling rig, it would have limited application to extract information about the fouling behaviour of the system, other than the initiation period. According to the models,
higher surface temperatures could accelerate the initiation and fouling processes, hence promoting deposition and potentially enabling the study of fouling.

An alternative experiment at higher temperature (here called Sim 404) is simulated to investigate such effects. The operating conditions are:

- Inlet bulk temperature: 220°C.
- Maximum current: 950 A.
- Maximum voltage: 5.94 V.
- Mass flowrate: same as Run 402.

These conditions are similar to Run 266 (Chapter 4) and are practical for the current configuration of HiPOR since they would permit controlling the inlet bulk temperature to the set-point. In this simulation, the inputs in Sim 403 are modified, increasing inlet oil temperature, current and voltage but keeping the noise in the original experiment to produce more realistic results.

The simulation results for average fouling resistance, deposit thickness and fractional coverage are shown below. The vertical line in the figures indicates the time at which a 72h experiment (such as Run 402) would have finished (4500 min). The results show that the fractional coverage quickly changes after 2000 min, reaching values near 1 after 3300 min. The thickness of the layer reaches values of about 20 μm after 4500 min and 60 μm at the end of the experiment, leading to overall fouling resistances of 0.1 and 0.3 m²K/kW, respectively.

![Figure 5.30 Simulated tube average fouling resistance (a) and deposit thickness (b) for Sim 404.](image-url)
Tests Run on Crude Oils

Figure 5.31 Simulated fractional coverage over time for Sim 404.

The deviation in pressure drop is greater than in Sim 403, but still too small to be detectable (0.39 mbar at the end of the experiment). The deviation in the wall temperature is significant, exceeding the error given by the accuracy of the sensor before 4000 min, as shown in Figure 5.32. A 72h experiment would be enough to observe the impact of fouling on wall temperature, but too short to study the fouling rate. An extended experiment as Sim 403, conversely, could be used to develop the fouling layer further and therefore would be more useful to eventually extract information on the fouling behaviour.

Figure 5.32 Wall outer surface temperature simulation results at the tube midpoint (a) and absolute error for the 5 measurements (Fouling – No Fouling) (b), considering fouling with initiation for Sim 404.

5.8 Conclusions

HiPOR was able to be operated with blended crude oil at the desired operating conditions; cold, low and hot temperature. The rig was operated successfully for more than 72 hours inclusive the start-up and shutdown phase. All experimental important parameters were presented. Based on the observation, no obvious changes happened during the 72 hours. The experimental
data have been validated with the model without the fouling model. All parameters; differential pressure, dP, differential temperature, delta T, outer insulation surface temperature, $T_{w,o}$ and wall temperature, $T_w$ show an excellent agreement against experimental data. These experimental data also has been validated with the model with fouling. The simulation obtained shows there will be a deviation of wall temperature after 60 hours of experiment. It confirmed that the model without a fouling shall be used to demonstrate the Run 402. An induction model has been introduced within the fouling model. The simulation shows that the coverage or formation of fouling can only be seen after 4000 minutes of the experiments and $T_w$ started to show some deviations. However, if the rig is to be operated with the operating conditions of Run 266, the simulation results show that the induction period is reduced to 2000 minutes and the fouling deposition will start to occur.
6 Conclusions and Future Work

6.1 Summary and contributions
This thesis had explained extensively on the novelty and development of the pilot scale experimental rig, HiPOR in terms of safety, operation and analysis. HiPOR has been built since 2008 and this is the first time the comprehensive experimental results and analyses have been reported. Chapter 2 had been elaborated in details with all necessaries prerequisite knowledge and processes before attempting an operation/experiment. These knowledge include the development of safety information and awareness, commissioning the rig and designing the control system. The control systems have been tested and successfully controlled the desired operating parameters and the results had been presented in chapter 2, 4 and 5.

Chapter 3 was the adapted model from (Coletti, 2011) and modified to suit with the HiPOR test section. The modifications on the adapted model has been highlighted which include the joule heating and insulation model. Furthermore, it also has the case structure to choose between non fouling oil (Paratherm) and crude oil; Paratherm (manufacturer data) and crude oil (API correlation). There are a few correlations expressions had been presented according to literature which related to heat transfer, fouling model and friction factor. The friction factor correlations captured the differences for validation that explained extensively in chapter 4 and 5.

Chapter 4 highlighted the experimental data with non fouling oil, Paratherm. All the primary measurements that had been calibrated and discussed in chapter 2 showed an excellent reproducibility within the design parameters of HiPOR. The experimental results with a difference of operating conditions also presented in this chapter. A validation against experimental data also presented in this thesis. It showed that the differential pressure, DP [±2 mbar] and differential test section temperature, delta T [± 0.5 °C] give excellent agree, while the wall temperature, T_w only gives fair against the experimental data.

In the final chapter, which was chapter 5, the HiPOR was able to be operated with blended crude oil at the desired operating conditions; cold, low and hot temperature. The rig was operated
successfully for more than 72 hours inclusive the start-up and shutdown phase. All experimental important parameters were presented. Based on the observation, no obvious changes happened during the 72 hours. The experimental data have been validated with the model without the fouling model. All parameters; differential pressure, dP, differential temperature, delta T, outer insulation surface temperature, $T_{w,o}$ and wall temperature, $T_w$ show an excellent agreement against experimental data. These experimental data also has been validated with the model with fouling. The simulation obtained shows there will be a deviation of wall temperature after 60 hours of experiment. It confirmed that the model without a fouling shall be used to demonstrate the Run 402. An induction model has been introduced within the fouling model. The simulation shows that the coverage or formation of fouling can only be seen after 4000 minutes of the experiments and $T_w$ started to show some deviations. However, if the rig is to be operated with the operating conditions of Run 266, the simulation results show that the induction period is reduced to 2000 minutes and the fouling deposition will start to occur.

6.2 Future works

The HiPOR rig has been successfully commissioned and tested for a long hours experimental run. That means the rig is ready to be tested for crude oil fouling. Based on the simulation results presented in this thesis, the rig needs to be validated at various operating conditions in order to obtain fouling deposition. The future works can be extended as follows;

- Run the experiment with the operating conditions based on simulation results which show indications of fouling deposition through the primary measurements; $T_w$ and dP
- The existing flowrate can only give a maximum velocity of 1.5 m/s. The change of pump could possibly increase the range of velocity up to 3.0 m/s. The main reason is the velocity of the preheat heat exchanger is ranging between 1.0 m/s to 3.0 m/s.
- Modification of cooling capacity; currently the cooling capacity was not able to cooling down the outlet temperature as desired, especially when the desired inlet temperature less than 230 °C. A high return temperature resulted the increment of desired inlet temperature of preheater, hence the inlet temperature to the test section cannot be controlled.
- Run more experiments with different physical properties. The validation processes will justify the robustness and reliability of the mathematic model.

6.3 Dissemination of results

Some of the results which reported in this thesis have been disseminated at various conferences and publications.


Appendix A: Standard Operating Procedure v1.2

1. INVENTORY/EQUIPMENT

1.1. The HIPOR facility consists of an Oil Rig within an enclosure (Tank, Pipework, Preheater, Control and Isolation Valves, Tubular Test Section, Flow meters, Heat Exchangers, Main and cooling loop pumps, Pressure Relief Valves, Various type of sensors e.g. pressure transducers, differential pressure transducers and thermocouples,) , Main Control Panel ( Display main variables of the process and contain programmable controllers and programmable Logic Controller PLC, HiPOR Workstation Computer with installed LabView 2013 Control & Data Acquisition Software, HiPOR Station Desk with two LCD monitors, 7 day programmer and temperature controller for immersion heater within Tank, Preheater Power Cabinet and Lambda Power Supplies for Preheater, Nitrogen Generator and its Air Drying Unit, Nitrogen cylinder and Water Chiller, Crowcon GasMaster Gas Detection System (CH₄, H₂S and Oxygen Level), Rapid Ventilation System and related control Panel for it, Carbon Dioxide Fire Suppression System interlocked with Ventilation system, Power supplies for Test Section. The layout of all main components is illustrated in Appendix 8.1. The Flow sheet of the facility is available in Appendix 8.2.

1.2. This facility is controlled by LabView 2013 (National Instruments) Software via USB controlled modules (DAQ, Current Loop Control, Analogue Control, Digital IN/OUT) , installed on the HiPOR Workstation Computer located in 111a at HiPOR

1.3. A Nitrogen Generator located on the right side of the facility is connected to the departmental compressed airline supply.

1.4. Copies of the Operating Manuals are kept in lab 111a while originals are stored in the office C409 and a Soft copy is stored in the Safety Departmental Folder.

2. MAIN SAFETY ASPECTS

2.1. Handling certain chemicals during some operations might require additional PPE in accordance to the COSHH assessment (such as personal H2S monitors during handling of crude oil samples). When performing experiments with Paratherm NF it is best to maintain similar safety measures although some aspects of the safety precaution system might be redundant (H2S, CH4 detectors, O₂ monitoring in Lab).

2.2. All chemicals should be always handled in accordance with the COSHH Assessment and it should be checked that a printed copy of the MSDS is present in the laboratory.

2.3. The Main Control Panel allows the separate monitoring of some of the process conditions (indicated with appropriate labels on the main panel). It allows the operation of the pumps M1 and M2 and for their emergency shutdown (emergency stop buttons). Power supplies dedicated to the Test Section(s) and Preheater have to be enabled from the Main Control Panel before use. The Main Control Panel is equipped with a Display/Programmable Controller and PLC (Programmable Logic Controller) and its actions in case of failures are tabulated in Appendix 8.6.

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2 Standard Operating Procedure has been improved continuously since the establishment of HiPOR. The first version of SOP created by Dr. Jerzy Pental, follows by Mr. Zulhafiz Tajudin. The latest version of this SOP updated by Mr Pawel Orlowski
2.4. The Crowcon GasMaster Gas Detection System should be checked on weekly basis between the runs and once per shift when in continuous operation. This can be achieved by pressing the Test button located on the traffic light system mounted on the wall close to N2 generator. Similar panel is installed in the laboratory next door (111b). Sensor readouts can be monitored either from the LabView Software Virtual Instrument or on the main GasMaster control panel located next to the main door leading to the laboratory. During normal operation, there is no exposure to H2S. Under no circumstances should this system be switched off, or gas detectors tempered with. Enclosure panels should be mounted for (operation above 100°C) and cannot be taken off when in operation or during the absence of supervision after a run has been finished. Alarm levels for each of the gas sensors are available in Appendix 8.7.

2.5. The Dominick Hunter N2 generator should be checked on a shift basis (the green light indicating satisfactory functioning should be “on” on the unit’s main display). In addition, the Air Drying Unit should be similarly checked on the shift basis– the gauge (on air drying unit) should be located either on green or blue level, indicating allowable moisture levels.

2.6. The External Thermal Exchange (chiller unit) should be monitored on shift basis (the level of water in the tank can be checked on the back of the unit within marked). Tubing leading to and from the HiPOR facility should be checked for leaks at the same time.

2.7. The Dedicated Rapid Ventilation System has two extraction lines and one supply line that are connected to the facility. The Rapid Ventilation System Control Panel is located above the power supplies dedicated to the Preheater on the left hand side of the facility next to fire engine garage. This system is interlocked with the CO2 Fire Suppression System. The CO2 Fire Suppression System is the Master Control for ventilation. It is standalone system assuring safety protection during operation and where risk of fire exists.

2.8. The CO2 Fire Suppression System should be turned on put into automatic mode when experiments are performed. This System is designed to operate as a double knock system (i.e. it detects two and reacts to two temperature levels detected within enclosure). The first level of temperature (71°C) activates a ringing bell, the second level of temperature (88°C) switches off the Rapid Ventilation System, and initiates emergency CO2 release and flooding of the enclosure within 25 seconds. This system should be regular maintained by engineers from Universal Fire Ltd, according to the maintenance schedule.

2.9. For 24h or longer experiments the overnight form with necessary directions should be issued and placed in an easily accessible place. The form should include a description of the experiment, associated hazards together with directions what to do with the equipment in case of an emergency. At least one operator from the night shift needs to have First Aid Training. (ideally both operators).

2.10. In the case of emergency (dependent on the scenario) the HIPOR facility should be shut down according to procedures 4.5.4 (Scenario A – Major Hazard) or 4.5.5 (Scenario B – Minor Hazard).(In all emergencies power supplies to the Test Section, Preheater and Immersion Heater should be interrupted) and the space evacuated. Both pumps M1 & M2 should be stopped if the emergency situation is serious (Fire, major leak, critical failure of equipment.) In a minor hazard situation the pumps may be kept running to speed up process of cooling down the system.
3. MAINTENANCE

3.1. Scheduled maintenance inspections will be carried out by trained engineers from adequate companies.

3.2. Any maintenance work carried out on the facility, or its components must be done in accordance with the User Manuals.

3.3. The Workshop Team will ensure the facility is in working order before experiments are carried out.

3.4. All activities will be listed in Permit to Operate document which need to be signed and approved by Academic Owner. This document will be uploaded into departmental safety folder for future reference.

4. WORKING PROCEDURE

4.1 Preliminary Inspection (Mandatory before any planned experimental or calibration runs)
   4.1.1 Make sure the following valves remain OPEN: V-1, 5, 6, 7, 11, 14, 16, 18, 20
   4.1.2 Make sure the following valves remain CLOSE & PLUGGED: V-39, 40, 36, 29, 37, 38, 35
   4.1.3 Make sure the Nitrogen Relief Valve V-27 remain CLOSE.
   4.1.4 Make visual inspection of all flanges and threaded connections. Check for right bolt torque.
   4.1.5 Check the Chiller connection and ensure that, all connection and verify the hoses are running freely with no sharp bends. Make sure the level of cooling water is within marks range on chiller tank.
   4.1.6 Carry out visual inspections of electrical connection. All connection need to be sound and tight.
   4.1.7 Check for sufficient pressure of Nitrogen in High Pressure Cylinder (At least 80 bars)
   4.1.8 Examine the status of HiPOR Workstation Computer, which is hosting all DAQ and Control USB modules. Use Instacal software to verify correct configuration for every DAQ/Control boards connected to RIG. Upon validation, save the configuration file and close software.
   4.1.9 Start LabView software and load main Virtual Instruments, which is use for data acquisition on control the facility. The name of Project is HiPOR Control, main Vi HiPOR Control.
   4.1.10 Make sure the thermal insulation is in good and sound condition.
   4.1.11 Make sure all Flange Guards are on place and correctly installed.

4.2 If experiment to be performed needs the tank content need to be changed / filled or drained go to point 4.3. If system is already filled and sealed, no need of change content go to point 4.4

4.3. Loading/Unloading procedure:

4.3.1. Loading Procedure
   4.3.1.1. Test gas alarm (as per point 2.3/2.4)
   4.3.1.2. Depressurise main system (P1 sensor should indicate 0 [bar] – on the Main Control Panel/LabView and on Mechanical Gauge)
4.3.1.3. Check temperature of the system (temperature sensors T1-T12 should not exceed ambient temperature levels).
4.3.1.4. Ensure there is no supply to electrical heaters. Press emergency switch dedicated to the power supplies on the main panel - isolate both supplies.
4.3.1.5. Close valve V1 (manual valve).
4.3.1.6. Close valves V3 and V22 (automated). Valves V3 and V22 are closed through LabView control program.
4.3.1.7. Activate Rapid Ventilation System in Override Mode available from Ventilation Control Panel (Turn Key Left)
4.3.1.8. Unplug the Valves V-39 (Oil Refill Port) and V-40 (Vacuum Port)
4.3.1.9 Connect vacuum pump to valve V-40.
4.3.1.10 Prepare known amount of Paratherm/Crude Oil (25l Steel Drums) weight / volume
4.3.1.11 Connect extension hose to V-39. Another end of hose place in Drum with sample which will be loaded.
4.3.1.12 Start Vacuum pump to create negative pressure in Tank and open V-39.
4.3.1.13 Stop the vacuum pump as soon as drum will be empty.
4.3.1.14 Wait few minutes before you close V-39 to allowed remove small amount of oil. Which can still be in the pipeline into drum.
4.3.1.15 if you wish load more oil according to experiment detail please repeat steps from point 4.3.1.11. If no further load necessary proceed to the next step.
4.3.1.16 Close valves V-39, V-40. Disconnect vacuum Pump and extension hose.
4.3.2. Draining (Unloading) Procedure

4.3.2.1 Test gas alarm (as per point 2.3/2.4)
4.3.2.2 Check depressurisation of the main system (P1 sensor should indicate 0 [bar] – on the Main Control Panel/LabView and on Mechanical Gauge)
4.3.2.3 Check temperature of the system (temperature sensors T1-T12 should not exceed ambient temperature levels).
4.3.2.4 Ensure there is no supply to electrical heaters. Press emergency switch dedicated to the power supplies on the main panel - isolate both supplies.
4.3.2.5 Make sure valve V1 is in OPEN (manual valve).
4.3.2.6 Open valves V3 and V22 (automated). Valves V3 and V22 are controlled via LabView.
4.3.2.7 Prepare appropriate size container / barrel which will be able to accommodate larger volume than planned draining.
4.3.2.8 Container should be placed on spill pallet if possible.
4.3.2.9 Test personal H2S monitors.
4.3.2.10 Unplug valves V-36 (Drain point Valve)
4.3.2.11 Connect extension hose to V-36 and secure another end in unload barrel.
4.3.2.12 Allow for a natural flow from Tank to barrel.
4.3.2.13 Monitor level in Tank by using HF Level Meter.
4.3.2.14 When Tank will be empty stop nitrogen supply isolate valve V-36
4.3.2.15 Put small spill tray or cuvette under connection hose and V-36 as there may remain small amount of oil in hose and it can spill when disconnect from valve.
4.3.2.16 Spill in tray need to be a drain into main unload barrel and valve V-36 Plugged.
4.3.2.17 If RIG need refilling by new oil go to point 4.3.1 (Loading Procedure)
4.3.1.18 In other case re-instate plug on V-40.

4.4 Preliminary Operations (Check and Active of all Auxiliary Systems)

4.4.1 Auxiliary system need to be activate.
4.4.1.1 Power Immersion Heater need to be active certain amount of time before planned experiment. Variables need to be taken into account. (60 litres volume of oil in system climb with a speed of 8°C/per hour) The system needs to be programmed such way and in advance by using seven days programmer and temperature controller located on HiPOR Desk Station to be able to achieve desired initial operating condition which are 650°C inside Tank.
4.4.1.2 Nitrogen Generator need to be started at least 1.5hrs before planned experiment. This is a necessary time for self-purging of this system.
4.4.1.3 TURN ON main Power Switch for HiPOR & Control Panel & Reset by-pass button.
4.4.1.4 Start Chiller (Rotating gear on Chiller Panel) and Push ON Button.
4.4.1.5 Activate CO2 Fire Suppression System (Turn key to automatic ARMED Position)
4.4.1.6 Activate Rapid Ventilation System in Override Mode (Full safe mode require closing all enclosure (Installation of Metal Panels)
4.4.1.7 Verify Crowcon Gas Sensors Functionality.
4.4.1.8 Run main Virtual Instruments “HiPOR Control” under LabView Software
4.4.1.9 Double check as all items from module 4.4.1 are ticked on the relevant check form.

4.4.2 Check for correct functionality off all activated systems.
4.4.2.1 Verify reading displayed on Front Control Panel (all temperature in ambient range, except Tank, Pressure P1 0.0 [bar], Flow meters showing 0.0 Flow, Pressure P2 in range 0.2-0.5 [bar])
4.4.2.2 Verify is Chiller indication lit indication is GREEN and reading on all pressures gauges on chiller panel differs from zero. Make sure the valve V-28 [5%] is in the open position to allowed water circulation from chiller and via Bowman heat Exchanger.
4.4.2.3 Check is Nitrogen Generator reach desired oxygen free level which is below 0.5%
Check is Nitrogen line is free from the leaks.
4.4.2.4 CO2 Fire Suppression system need to be in ARMED Position
4.4.2.5 Check the Health functionality of Rapid Ventilation system which is indicated on this auxiliary system panel.
4.4.2.6 Verify of operation Immersion Heater. Desired temperature should be achieved just now which is 650°C. The temperature is indicate on Front Control panel, Temperature Controller (pre-set for max 650°C) just below LCD’s Monitor and in LabView Software.
4.4.2.7 Verify consistency of all reading in LabView software (All temperature in ambient temperature ranges except Tank which should indicate 600°C +/-70°C, Pressure Tank in range P1<1 [bar], Flow meter reading are 0.0, Valve position – Closed except V-28, Pump M1&M2 not operate, Heaters for Test Section and Preheater are OFF.
4.4.2.8 Check is the reading in Crowcon Gas Monitoring System are within safe range and the reading from LabView and Crowcon panel are within the same range.
4.4.2.9 If any of the above conditions are not met – DO NOT ATTEMPT TO START EXPERIMENT.

4.5 Main Operations – Experiment Phase

4.5.1 Start-Up RIG (Purpose: to reach desired experimental condition; pressure 0[bar]>>5[bar]>>30[bar] and temperature 65[°C]>>100[°C]>>240-260[°C])

4.5.1.1 Purging Gases in Tank by cyclic pressurize and depressurize the Tank by pure nitrogen. 0[bar]>>5[bar]>>0[bar]. Cycle need to be repeated at least three times [3x].

4.5.1.2 Pressurize Tank to 5[bar] using pure nitrogen by gently control of valve V-23

4.5.1.3 Start Pump M2 (Cooling Loop Circulation) – Start button on Front Control panel (make sure the emergency push button is depressed). Please ensure the manual valve V-34 is fully open and cooling loop pressure, P2 is less than 0.5 [bar]. P2 can be release via manual valve, V-32 which is located on the top of Paratherm expansion vessel. This activity need to be done before start cooling loop pump, M2 at initial stage of experiment. DO NOT releases any pressure of P2 during normal operation / experiment.

4.5.1.4 Open Valve V-3 and V-22 from LabView. Make sure they are open by visual inspection of their rotating indicator and green light on front control panel.

4.5.1.5 Open valves V-10 (5%) and V-17 (10%) from LabView to allow flow via cooling loop Compablock Heat exchanger and test section.

4.5.1.6 Check temperature of oil in Pressure Vessel (need to be 65°C to reach minimum viscosity parameter to allow operation of pump M1)

4.4.2.9 Start On Power supplies for preheater and test section. (The emergency button need to be depressed and safety interlock activated)

4.5.1.7 Start pump M1 (main loop circulation) – start button on Front Control Panel (need to be pushed together with by-pass reset button to allow ARM of PLC interlock)

4.5.1.8 Verify flow of fluid through the test section and cooling loop (flow meters, DP sensor, temperature change along all installation)

4.5.1.9 Turn ON Preheater from LabView to allow flow of sufficient power input, to compensate heat dissipation via cooling loop and allow system climb temperature in a safe range that is 60°C/per hour. (Equipment limitation: Compablock heat exchanger related to heat stress in material) This heat input for preheater is controlled in manual / automatic mode via LabView.

4.5.1.10 Rise flow via cooling loop by meaning of opening valve V-10 to 10[%]

4.5.1.11 Adjust the power input of Preheater to compensate energy taken by cooling loop to maintain the temperature rise at the rate not exceeding 60°C/per hour.

4.5.1.12 The maximum permissible temperature rise is 60°C/per hour. The limitation derived from the specification of Compablock heat exchanger, and it is determined by this element.
4.5.1.13 As soon as temperature reaches 100°C Rig enclosure need to be closed and sealed. After sealing the enclosure, the rig can be monitored via Glass Visor and Light provided.

4.5.1.14 Rapid Ventilation system need to be switched from override position to Full automated control feature (Turn control key to the RIGHT).

4.5.1.15 As soon as temperature reach final operational temperature which is 240-260°C, heat input from preheater should only maintain the equilibrium between energy taken by cooling loop.

4.5.1.16 At this stage, the operator need to increase pressure in intervals 10 minutes with increments rate of 5 [bar] by using high pressure nitrogen cylinder via opening valve V-23.

4.5.1.17 Operational condition are achieved, (these operating conditions are set in the experiment design for each run). The maximum allowable pressure and temperature for this rig are 30 [bar] and 270°C respectively.

4.5.1.18 Check visual Rig for possible leaks, fumes within enclosure, unusual pressure changes, flow or temperature.

4.5.1.19 If no deviation from expected condition Rig is ready for next Phase 4.5.2.

4.5.1.20 If any deviation detected proceed Normal Shut Down procedure as in 4.5.3.

– DO NOT ATTEMPT TO CONTINUE OF EXPERIMENT

4.5.2 Test/Calibration/Experimental Run (Important: experiment need to be design in great details and approved after deep analysis by academic owner of the RIG; Information in this module are indicative only and aren’t part of design of experiment)

4.5.2.1 Initial condition of this stage are as in point 4.5.1.17

4.5.2.2 Operate Tubular test section (Flow rate, temperature, heat flux)

4.5.2.3 Maintain flow through cooling section to balance heat flux input in test section.

4.5.2.4 Cooling rate can be controlled direct via V-10 or indirect via V-28/Chiller

4.5.2.5 Make sure flow via test section when power supplies to test section activated.

4.5.2.6 Increase Joule’s heating setting to achieve desired heat flux rate in test section

4.5.2.7 Balance cooling rate as mentioned in point 4.5.2.4

4.5.2.8 To check for experimental equilibrium, observe and determine the following:

- Temperature
- Flow Rate
- Pressure
- Heat Flux
- Time of experiment

4.5.2.9 If any deviation detected from designed experiment, minor leak, or equipment malfunction proceed to Normal Shut Down procedure as in 4.5.3.

– DO NOT ATTEMPT TO CONTINUE OF EXPERIMENT

4.5.3 Normal Shutdown Procedure

4.5.3.1 Turn OFF all heating source: Power supplies (test section, preheater from LabView),
Immersion Heater from programmer/controller under LCD Screen.

4.5.3.2 Reduce Flow in Test Sections to 10% using V-13 and V-17
4.5.3.3 Maintain Flow via cooling loop and monitor cooling rate which should not exceed 60°C/hour.
4.5.3.4 To keep desired cooling rate you may need adjust flow rate via cooling loop, and indirect variables as V-28 and chiller settings.
4.5.3.5 As soon as temperature of system drop down to the level of 60°C [approximately. 3.5hr] switch OFF the Main Pump M1.
4.5.3.6 Shutdown Cooling Loop pump M2
4.5.3.7 Decrease pressure in system from High pressure (less than 30 bar as system is cooled down) to 0 [bar] in rate of 5 [bar]/per 10 minutes.
4.5.3.8 Isolate valves V-3, V-22 from Labview and verify by visual indicator as in 4.5.1.4.
4.5.3.9 Turn Off Chiller
4.5.3.10 Close valve V-28 from LabView
4.5.3.11 Close and finalize HiPOR Control Program in LabVIEW to finish the process of DAQ.
4.5.3.12 Shutdown Nitrogen Generator
4.5.3.13 Make sure high pressure nitrogen cylinder is properly closed and isolated.
4.5.3.14 Enclosure can be re-open at this stage.
4.5.3.15 Rapid ventilation system need to be switched into override mode to keep further cooling of the system until it reaches ambient condition.
4.5.3.16 De-activate CO2 Fire Suppression System (Switch into Manual Mode)
4.5.3.17 Copy all data acquired during experiment and place them on secure departmental hard drive.
4.5.3.18 When system reaches ambient temperature ventilation need to be switch OFF.

4.5.4 Emergency Shutdown – Scenario A - Major Hazard (major leak, critical failure of equipment, sudden drop of pressure, fire, Workstation Computer Failure, etc.)

4.5.4.1 Cut OFF Power Supplies (cut power by pushing emergency button heaters) (preheater, test section) and immersion heater (programmer and temperature controller on HiPOR Desk)
4.5.4.2 Cut OFF Power to Pumps M1 & M2 (emergency buttons for M1 & M2)
4.5.4.3 Shot valves V-3, V-22 from LabView
4.5.4.4 Keep running Rapid Ventilation System and CO2 Fire Suppression System Armed
4.5.4.5 In case of fire ventilation system will be de-activated and air intake damped by CO2 Fire Suppression system.
4.5.4.6 After 25 seconds from detection of fire, CO2 Fire Suppression system release extinguishing
4.5.4.7 As soon as operator notice constant alarm signal from fire suppression system and flashing lights he must push all three emergency buttons, shut valve V-3, V-22 and switch off immersion heater than need immediately to evacuate themselves from hazard area and activate College Fire Alarm outside Lab111.
4.5.4.8 When fire will be eliminated here will be an option to start ventilation to remove fumes and CO2 from the enclosure.
4.5.4.9 In this scenario you need to be aware as the cooling loop is de-activated and RIG may need more time to dissipate heat by natural convection and forced ventilation.
4.5.4.10 Rapid Ventilation System will help to keep safe level of hydrocarbons in air. (Out of combustible range.)

4.5.4.11 Closed isolation valves V-3, V-22 will keep pressure vessel TANK isolated from system. In case over pressure of vessel when system is divided in two parts only the gas will be released to K/O Tank. If pipework/instrumentation system will over pressurized PRV (set to lift at 32[barg]) valve released relatively small amount of fluid (total volume of pipework and instrumentation is no more than 6 litres) into K/O Tank. It will be very limited volume of fluid.

4.5.4.12 Under no circumstances enter into the lab after activation of College Central Alarm.

4.5.5 Emergency Shut Down – Scenario B – Minor Hazard

(minor leaks, negligible leak shows only sign of fumes from the affected area, non-critical failure (loss of sensor reading), etc.)

4.5.5.1 Try as much practicable/reasonable use Normal Shutdown procedure point 4.5.3

This is the fastest way to cool down system to a safe range.

4.5.5.2 If situation change to serious immediately follow emergency shut down procedure described in point 4.5.4.

5.0 Results

The results will be stored on the computer, and secure network drive. This will be done either by laboratory PC, or by USB memory stick.

6.0 Records

A printed version of the manual is available in 111a and an electronic version on a secure network drive.

7.0 Contacts

EMERGENCY CONTACT NO - INTERNAL 4444 OR 020 7589 1000
ICT - INTERNAL 49049 OR 020 7594 9049
Paweł Orzowski (Research Technician) - 07919604384 office C409
Dr Iveline Valkov (PDRA) - 07731504179 office C409
Zulhafiz bin Tajudin (Asset Manager) - 07909045453 office C409
Office C409 telephone: 02075946626
8.0 Appendices

8.1 Spatial plan of the main components of the HiPOR facility.

A. HiPOR facility
B. Main Control Panel
C. HiPOR Station Desk with 2 No LCD monitors and Workstation Computer
D. Nitrogen generator
E. Nitrogen storage vessel 6 [barg]
F. $N_2$ Air Dryer
G. CO$_2$ Fire Suppression System Control panel
H. Power Supply for Test Section
I. Chiller
J. Rapid Ventilation Control Panel
K. Power Supply for preheater
L. High Pressure Nitrogen Bottle
M. 50kg CO$_2$ Cylinder for fire suppression system
N. Reserve High Pressure Nitrogen Bottle
8.2 HiPOR Flowsheet Diagram v2.0 (Should be studied in native size which is A3)
# 8.3 Equipment and valve list

<table>
<thead>
<tr>
<th>Equipment / Valve</th>
<th>Tag number</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Manual Valve</td>
<td>V1</td>
<td>Outlet of main tank</td>
</tr>
<tr>
<td></td>
<td>V4</td>
<td>Inlet to pump M1</td>
</tr>
<tr>
<td></td>
<td>V5</td>
<td>2nd line inlet to pump M1</td>
</tr>
<tr>
<td></td>
<td>V6</td>
<td>Outlet from preheater</td>
</tr>
<tr>
<td></td>
<td>V8</td>
<td>Inlet to sample tank</td>
</tr>
<tr>
<td></td>
<td>V9</td>
<td>Outlet of sample tank</td>
</tr>
<tr>
<td></td>
<td>V11</td>
<td>Outlet of compabloc return line</td>
</tr>
<tr>
<td></td>
<td>V14</td>
<td>Inlet to annulus test section</td>
</tr>
<tr>
<td></td>
<td>V16</td>
<td>Outlet of annulus test section</td>
</tr>
<tr>
<td></td>
<td>V18</td>
<td>Inlet to tubular test section</td>
</tr>
<tr>
<td></td>
<td>V20</td>
<td>Outlet of tubular test section</td>
</tr>
<tr>
<td></td>
<td>V25</td>
<td>Compressed nitrogen inlet for generator/ cylinder to main tank</td>
</tr>
<tr>
<td></td>
<td>V27</td>
<td>Air venting from main tank</td>
</tr>
<tr>
<td></td>
<td>V29</td>
<td>Drain for cooling section</td>
</tr>
<tr>
<td></td>
<td>V30</td>
<td>Inlet of cooling fluid to cooling circuit</td>
</tr>
<tr>
<td></td>
<td>V32</td>
<td>Air venting for cooling fluid's holding tank</td>
</tr>
<tr>
<td></td>
<td>V33</td>
<td>Inlet to pump M2</td>
</tr>
<tr>
<td></td>
<td>V34</td>
<td>Outlet of pump M2</td>
</tr>
<tr>
<td></td>
<td>V35</td>
<td>Drain for cooling loop - Paratherm</td>
</tr>
<tr>
<td></td>
<td>V36</td>
<td>Drain for the main tank</td>
</tr>
<tr>
<td></td>
<td>V37</td>
<td>Drain for the annulus section</td>
</tr>
<tr>
<td></td>
<td>V38</td>
<td>Drain for the tubular section</td>
</tr>
<tr>
<td></td>
<td>V39</td>
<td>Oil refill port</td>
</tr>
<tr>
<td></td>
<td>V40</td>
<td>Vacuum port</td>
</tr>
<tr>
<td>Check Valve</td>
<td>V2</td>
<td>Outlet of main tank</td>
</tr>
<tr>
<td></td>
<td>V12</td>
<td>Outlet of compabloc at cooling loop</td>
</tr>
<tr>
<td></td>
<td>V15</td>
<td>Inlet to annulus test section</td>
</tr>
<tr>
<td></td>
<td>V19</td>
<td>Inlet to tubular test section</td>
</tr>
<tr>
<td></td>
<td>V21</td>
<td>Return from test section</td>
</tr>
<tr>
<td></td>
<td>V26</td>
<td>After flowmeter, F3</td>
</tr>
<tr>
<td></td>
<td>V31</td>
<td>Cooling medium fluid loading</td>
</tr>
<tr>
<td>Control Valve</td>
<td>V10</td>
<td>Control valve to cooling loop</td>
</tr>
<tr>
<td></td>
<td>V13</td>
<td>Control valve to annulus section</td>
</tr>
<tr>
<td></td>
<td>V17</td>
<td>Control valve to tubular section</td>
</tr>
<tr>
<td></td>
<td>V28</td>
<td>Control valve of cooling water to EX2</td>
</tr>
<tr>
<td>Relief Valve</td>
<td>V23</td>
<td>Return line to venting system</td>
</tr>
<tr>
<td></td>
<td>V24</td>
<td>Main tank to venting system</td>
</tr>
<tr>
<td>Isolation Valve</td>
<td>V3</td>
<td>Valve after main tank</td>
</tr>
<tr>
<td></td>
<td>V22</td>
<td>Valve before main tank (return line)</td>
</tr>
<tr>
<td>Strainer</td>
<td>S1</td>
<td>Strainer before pump M1</td>
</tr>
<tr>
<td></td>
<td>S2</td>
<td>Bypass strainer before pump M1</td>
</tr>
<tr>
<td></td>
<td>S3</td>
<td>Strainer before pump M2</td>
</tr>
<tr>
<td>Pump</td>
<td>M1</td>
<td>Main circulation pump</td>
</tr>
<tr>
<td></td>
<td>M2</td>
<td>Cooling circuit pump</td>
</tr>
<tr>
<td>Heat Exchanger</td>
<td>EX1</td>
<td>Compabloc at cooling loop</td>
</tr>
<tr>
<td></td>
<td>EX2</td>
<td>Cooling water at cooling circuit</td>
</tr>
</tbody>
</table>
8.4 Instrumentation list

<table>
<thead>
<tr>
<th>Location</th>
<th>Tag number</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tank</td>
<td>T1</td>
<td>°C</td>
</tr>
<tr>
<td>Annulus inlet</td>
<td>T2</td>
<td>°C</td>
</tr>
<tr>
<td>Annulus RET</td>
<td>T3</td>
<td>°C</td>
</tr>
<tr>
<td>Annulus outlet</td>
<td>T4</td>
<td>°C</td>
</tr>
<tr>
<td>Tube inlet</td>
<td>T5</td>
<td>°C</td>
</tr>
<tr>
<td>Wall Tube middle (3/5)</td>
<td>T6</td>
<td>°C</td>
</tr>
<tr>
<td>Tube out</td>
<td>T7</td>
<td>°C</td>
</tr>
<tr>
<td>Test line return</td>
<td>T8</td>
<td>°C</td>
</tr>
<tr>
<td>Compabloc return</td>
<td>T9</td>
<td>°C</td>
</tr>
<tr>
<td>Paratherm Loop</td>
<td>T10</td>
<td>°C</td>
</tr>
<tr>
<td>Wall Tube bottom (1/5)</td>
<td>T11</td>
<td>°C</td>
</tr>
<tr>
<td>Wall Tube top (5/5)</td>
<td>T12</td>
<td>°C</td>
</tr>
<tr>
<td>Pre-heater bulk</td>
<td>T16</td>
<td>°C</td>
</tr>
<tr>
<td>Wall of preheater (downstream)</td>
<td>T17</td>
<td>°C</td>
</tr>
<tr>
<td>Wall of preheater (middle)</td>
<td>T18</td>
<td>°C</td>
</tr>
<tr>
<td>Cooling water return to the chiller</td>
<td>T19</td>
<td>°C</td>
</tr>
<tr>
<td>Wall Tube top (5/5)</td>
<td>T20</td>
<td>°C</td>
</tr>
<tr>
<td>Wall Tube bottom (2/5)</td>
<td>T21</td>
<td>°C</td>
</tr>
<tr>
<td>Pressure</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tank</td>
<td>P1</td>
<td>bar</td>
</tr>
<tr>
<td>Paratherm Cooling</td>
<td>P2</td>
<td>bar</td>
</tr>
<tr>
<td>Differential pressure</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Annulus delta P</td>
<td>dP1</td>
<td>mbar</td>
</tr>
<tr>
<td>Tube delta P</td>
<td>dP2</td>
<td>mbar</td>
</tr>
<tr>
<td>Power supply</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Current, preheater</td>
<td>IR1</td>
<td>Ampere</td>
</tr>
<tr>
<td>Current, test section (ANNULUS)</td>
<td>IR2</td>
<td>Ampere</td>
</tr>
<tr>
<td>Current, test section (TUBULAR)</td>
<td>IR3</td>
<td>Ampere</td>
</tr>
<tr>
<td>Resistance preheater</td>
<td>ER1</td>
<td>Volt</td>
</tr>
<tr>
<td>Resistance test section (ANNULUS)</td>
<td>ER2</td>
<td>Volt</td>
</tr>
<tr>
<td>Resistance test section (TUBULAR)</td>
<td>ER3</td>
<td>Volt</td>
</tr>
<tr>
<td>Flow</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Annulus Flowrate</td>
<td>F1</td>
<td>kl/h</td>
</tr>
<tr>
<td>Tubular Flowrate</td>
<td>F2</td>
<td>kl/h</td>
</tr>
<tr>
<td>Cooling section Flowrate</td>
<td>F3</td>
<td>kl/h</td>
</tr>
</tbody>
</table>

8.5 Data Acquisition Board list and status

Board 0, ID #35, Voltage DAQ (USB 1208SF)

<table>
<thead>
<tr>
<th>Channel</th>
<th>Item</th>
<th>Tag number</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Annulus volumetric flowrate</td>
<td>F1</td>
</tr>
<tr>
<td>1</td>
<td>Tube volumetric flowrate</td>
<td>F2</td>
</tr>
<tr>
<td>2</td>
<td>Cooling section flowrate</td>
<td>F3</td>
</tr>
<tr>
<td>3</td>
<td>Tank level</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Tank pressure</td>
<td>P1</td>
</tr>
<tr>
<td>5</td>
<td>Annulus differential pressure</td>
<td>dP1</td>
</tr>
<tr>
<td>6</td>
<td>Tube differential pressure</td>
<td>dP2</td>
</tr>
<tr>
<td>7</td>
<td>Cooling loop pressure</td>
<td>P2</td>
</tr>
<tr>
<td>Output 1: Digital Output</td>
<td>WatchDog Output</td>
<td>B (0) C (1)</td>
</tr>
<tr>
<td>Output 2: Digital Input</td>
<td>Status Control Alarm Boolean</td>
<td>B (0) C (2)</td>
</tr>
<tr>
<td>Port Number 1</td>
<td>Initialise : Digital Out</td>
<td></td>
</tr>
<tr>
<td>Port Number 2</td>
<td>Initialise : Digital In</td>
<td></td>
</tr>
</tbody>
</table>

Board 1, ID #96, TC DAQ (USB – TC)

<table>
<thead>
<tr>
<th>Channel</th>
<th>Item</th>
<th>Tag number</th>
</tr>
</thead>
</table>
Appendix A: Standard Operating Procedure v1.2

<table>
<thead>
<tr>
<th>Channel</th>
<th>Item</th>
<th>Tag number</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Test line return</td>
<td>T8</td>
</tr>
<tr>
<td>1</td>
<td>Compabloc return</td>
<td>T9</td>
</tr>
<tr>
<td>2</td>
<td>Cooling loop</td>
<td>T10</td>
</tr>
<tr>
<td>3</td>
<td>Tube wall top (4/5)</td>
<td>T12</td>
</tr>
<tr>
<td>4</td>
<td>{Annulus outlet}</td>
<td>{T4}</td>
</tr>
<tr>
<td>5</td>
<td>{Annulus inlet}</td>
<td>{T2}</td>
</tr>
<tr>
<td>6</td>
<td>T enclosure</td>
<td>T11</td>
</tr>
<tr>
<td>7</td>
<td>T system</td>
<td>T12</td>
</tr>
</tbody>
</table>

Board 2, ID #97, TC DAQ (USB - TC)

<table>
<thead>
<tr>
<th>Channel</th>
<th>Item</th>
<th>Tag number</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>[reserved]</td>
<td>[]</td>
</tr>
<tr>
<td>1</td>
<td>[reserved]</td>
<td>[]</td>
</tr>
<tr>
<td>2</td>
<td>Tube wall bottom (2/5)</td>
<td>T21</td>
</tr>
<tr>
<td>3</td>
<td>Tube wall middle (3/5)</td>
<td>T6</td>
</tr>
<tr>
<td>4</td>
<td>Tube outlet</td>
<td>T7</td>
</tr>
<tr>
<td>5</td>
<td>{Radiant annulus}</td>
<td>{T3}</td>
</tr>
<tr>
<td>6</td>
<td>Tube inlet</td>
<td>T5</td>
</tr>
<tr>
<td>7</td>
<td>Tube wall bottom (1/5)</td>
<td>T11</td>
</tr>
</tbody>
</table>

Board 3, ID #202, Voltage DAQ, analogue out USB 3104; 8 Channel, 16 bit Analog voltage/ Current Output device

<table>
<thead>
<tr>
<th>Channel</th>
<th>Item</th>
<th>Tag number</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>CV to cooling loop (AOut)</td>
<td>V10</td>
</tr>
<tr>
<td>1</td>
<td>CV to annulus loop (AOut)</td>
<td>V13</td>
</tr>
<tr>
<td>2</td>
<td>CV to tubular loop (AOut)</td>
<td>V17</td>
</tr>
<tr>
<td>3</td>
<td>CV to HEX2 loop (AOut)</td>
<td>V28</td>
</tr>
<tr>
<td>4</td>
<td>Annulus temperature setpoint (AOut)</td>
<td></td>
</tr>
</tbody>
</table>

Board 4, ID #115, Voltage DAQ (Gas Analyser) USB 1280 F; 8 analogue input, 2 Digital/ analogue output

<table>
<thead>
<tr>
<th>Channel</th>
<th>Item</th>
<th>Tag number</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Methane , CH4</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>O2 outside</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>H2S</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>O2 inside</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Output 1</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Output 2</td>
<td></td>
</tr>
</tbody>
</table>

Board 5, ID #255, Voltage DAQ USB 1208SF

<table>
<thead>
<tr>
<th>Channel</th>
<th>Item</th>
<th>Tag number</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Preheater Voltage</td>
<td>ER1</td>
</tr>
<tr>
<td>1</td>
<td>Annulus Voltage</td>
<td>ER2</td>
</tr>
<tr>
<td>2</td>
<td>Tubular Voltage</td>
<td>ER3</td>
</tr>
<tr>
<td>3</td>
<td>Main PSU current (Annulus)</td>
<td>IR2</td>
</tr>
<tr>
<td>4</td>
<td>Preheater current</td>
<td>IR1</td>
</tr>
<tr>
<td>5</td>
<td>Main PSU current (Annulus)</td>
<td>IR3</td>
</tr>
</tbody>
</table>
### Output 1

<table>
<thead>
<tr>
<th>Channel</th>
<th>Item</th>
<th>Tag number</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>T bulk temperature</td>
<td>T16</td>
</tr>
<tr>
<td>1</td>
<td>T wall temperature</td>
<td>T17</td>
</tr>
<tr>
<td>2</td>
<td>T wall temperature</td>
<td>T18</td>
</tr>
<tr>
<td>3</td>
<td>T chiller return</td>
<td>T19</td>
</tr>
<tr>
<td>4</td>
<td>Tube wall top (5/5)</td>
<td>T20</td>
</tr>
<tr>
<td>5</td>
<td>Tank</td>
<td>T1</td>
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<td>6</td>
<td>[reserved]</td>
<td>[]</td>
</tr>
<tr>
<td>7</td>
<td>[reserved]</td>
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</table>

### Output 2

### Board 6, ID #68, TC DAQ (USB – TC)

<table>
<thead>
<tr>
<th>Channel</th>
<th>Item</th>
<th>Tag number</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Signal for main pump operate</td>
<td>M1 Operate</td>
</tr>
<tr>
<td>1</td>
<td>Signal for cooling pump operate</td>
<td>M2 Operate</td>
</tr>
<tr>
<td>2</td>
<td>[reserved]</td>
<td>[]</td>
</tr>
</tbody>
</table>
### 8.6 PLC actions to be taken in case of failures

<table>
<thead>
<tr>
<th>Possible Failures</th>
<th>Item</th>
<th>Action - PLC</th>
<th>General</th>
</tr>
</thead>
</table>
| Temp exceeds upper limit | 1. Tank Heater (T1a-1b)  
2. Annulus Heater (T5 & T7) and Tube Heater (T2 & T4) | 1-2. Cut power to tank, annulus and pipe heaters. | Keep motors M1 and M2 running to keep cooling |
| Pressure exceeds upper limit | 5. Tank Pressure (P1)  
6. Annulus and Tube Pressure (DP1 – DP2) | 5-6. Cut power to tank ,annulus and pipe heaters, and motor M1 | Keep motor M2 running to keep cooling |
| Flow rate exceeds upper limit | 10. Flow meters (F1 and F2) | 10. Cut power to tank, annulus and pipe heaters, and motor M1. | Keep motor M2 running to keep cooling. Go into Fail Safe Mode |
| Power failure to any part of the rig | 14. Heaters (tank, annulus, tube), or motors (M1, M2) or PC | 14. Cut power to tank, annulus and pipe heaters, motor M1 and M2. | Go into ‘Fail Safe’ mode |
| PC crashes | 15. PC | 15. Cut power to tank, annulus and pipe heaters. | Keep motors M1 and M2 running to keep cooling |
| N2 generator fails | 17. N2 Generator | 17. Cut power to tank, annulus and pipe heaters. | Keep motors M1 and M2 running to keep cooling – Open N2 cylinder |
| Extract fails (need to avoid filling the lab with N2 - asphyxiation) | 18. External N2 detector | 18. Cut power to tank, annulus and pipe heaters, and N2 generator | Keep motors M1 and M2 running to keep cooling |
Appendix B: Uncertainties and Propagation of Errors

8.7 Alarm levels for HIPOR gas detection system.

<table>
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<tr>
<th>Detector Type</th>
<th>Methane</th>
<th>Oxygen</th>
<th>Hydrogen Sulfide</th>
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</thead>
<tbody>
<tr>
<td>Direction leading to alarm</td>
<td>rising</td>
<td>falling</td>
<td>rising</td>
</tr>
<tr>
<td>Range</td>
<td>100 % LEL *</td>
<td>25 % VOL</td>
<td>25 ppm</td>
</tr>
<tr>
<td>Alarm one – level off</td>
<td>19</td>
<td>19.5</td>
<td>4.5</td>
</tr>
<tr>
<td>Alarm one – level on</td>
<td>20</td>
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<td>5</td>
</tr>
<tr>
<td>Alarm two – level on</td>
<td>40</td>
<td>17</td>
<td>10</td>
</tr>
</tbody>
</table>

*100 % LEL of methane is equal to 5 % by volume

******* The End
Appendix B: Uncertainties and Propagation of Errors

Introduction
Uncertainty is defined in terms of English vocabulary, science, mathematics (statistical) and engineering approach. The elaboration should include how relevant how important this study towards the works that has been carried out. The uncertainties is not only involved the physical plant properties (measurement and geometry) but it also involved the physical properties of oil used for research purposes. In industrial application where primary measurements of important parameters are restricted and/or not available as discussed in previous chapter (mentioned which), furthermore the errors and uncertainties of these readings have been compromised for evaluation purposes. Hence, the prediction of fouling phenomena is always deviated from simulation of the model (references with graph).

The structure of this chapter will discuss the relevant information on this data and result analysis pertaining to uncertainties and errors of propagation which a continuity from the previous chapter (reference) that discuss on the superficial of errors based on primary measurements. In addition, the methodology and approaches of these issues will be discussed thoroughly from primary measured variables to the calculated variables.

The impact of uncertainties and errors of propagation are not only quantified in terms of value and numbers, but it also led into mislead of mitigation strategies and false signal of fouling formation. Hence, it is extremely important to monitor several aspects of significant signals especially on thermal and hydraulic effect before any vital decision will be made to the industrial plant.

Uncertainties philosophy
There are several approaches to oversee this problem on heat transfer problem. Mean temperature difference (Bowman, 1940) to overcome uncertainties in heat exchanger design? Please check the statement.

Study of uncertainty analysis of heat exchange on thermal hydraulic design (Cho, 1987), discussed and divided the area of thermal/hydraulic design parameters as illustrated in table 7.1. Cho (1987) highlighted the consideration to finalise exchanger design by considering the calculated performance analysis with statistical analysis using Gaussian rules. In addition, other factors which could affect the performance exchanges also need to take into account. Tatara (2011) used temperature measure uncertainty by combining the temporal and spatial uncertainties to evaluate independent parameters error of propagation. As a results, individual of uncertainties could be quantified, hence the finalise propagation of errors of heat duty can be determined. Unfortunately, the other parameters which are not related explicitly or implicitly with temperature are not taking into consideration. Uhia (2013) reported uncertainty analysis for experimental heat transfer using Wilson plot method. The authors have emphasized on the effect of operating conditions and geometry of physical rig could contribute towards propagation of errors.

Clarke et.al (2001) studied on sensitivity and uncertainty analysis of heat exchanger due to physical properties variation. Monte Carlo method is used instead of the convectional Taylor's series of expansion. No overall sensitivity weightage is reported in order to identify the most influence thermophysical property that most influence to this propagation of errors.
Moffat (1982) evaluate uncertainty based on the sensitivity coefficient $\delta R/\delta x$, contribution $(\delta R/\delta x)/\delta x$, relative uncertainty $\delta R/R$ and absolute uncertainty $\delta R$ on single sample (theoretical). Moffat (1985) suggested the uncertainty analysis should be included in the planning of experiment by indicated the importance of replication process and identified the desired experiment in steady state method as well as transient method for validation purposes. The author also suggested on how to describe the uncertainties in the experimental results should be reported and further results has been seen that the standard has been development to international standard (ISO reference).

Table A2.1 Thermal and hydraulic design parameters (Cho, 1987)

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<tr>
<th>Items</th>
<th>Variables</th>
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<td>Thermal</td>
<td>Heat transfer coefficient, fluid temperature (inlet &amp; outlet), wall temperature of test section</td>
</tr>
<tr>
<td>Hydraulic</td>
<td>Flowrate, friction factors, pressure different</td>
</tr>
<tr>
<td>Geometry</td>
<td>Tube dimension (outer diameter, wall thickness, length), Insulation thickness</td>
</tr>
<tr>
<td>Thermophysical</td>
<td>Density, viscosity, thermal conductivity, specific heat capacity</td>
</tr>
</tbody>
</table>

Table A2.2 General expression of uncertainties and errors of propagation for different functions (Reference)

<table>
<thead>
<tr>
<th>Function</th>
<th>Formula</th>
<th>Uncertainties</th>
</tr>
</thead>
<tbody>
<tr>
<td>Addition and subtraction</td>
<td>$f = x \pm y$</td>
<td>$\sigma_f = \sqrt{\sigma_x^2 + \sigma_y^2}$</td>
</tr>
<tr>
<td>Multiplication and division</td>
<td>$f = xy$ or $f = x/y$</td>
<td>$\frac{\sigma_f^2}{f^2} = \frac{\sigma_x^2}{x^2} + \frac{\sigma_y^2}{y^2}$</td>
</tr>
<tr>
<td>Powers</td>
<td>$f = x^y$</td>
<td>$\sigma_f = f \cdot y \cdot \sigma_x$</td>
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<tr>
<td></td>
<td>$f = e^{\pm x}$</td>
<td>$\sigma_f = f \cdot \sigma_x$</td>
</tr>
<tr>
<td>Exponential</td>
<td>$f = 10^{\pm x}$</td>
<td>$\sigma_f = f \cdot \ln 10 \cdot \sigma_x$</td>
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<tr>
<td></td>
<td>$f = \log_e \pm x$</td>
<td>$\sigma_f = \frac{\sigma_x}{\log_e x}$</td>
</tr>
<tr>
<td></td>
<td>$f = \log_{10} \pm x$</td>
<td>$\sigma_f = \frac{\sigma_x}{\log_{10} 10 \cdot x}$</td>
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</table>

#explanation of table 7.2

Table A2.3 Uncertainty Analysis in Experimentation (Coleman & Steele, 1998)

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<th>Uses of uncertainty analysis</th>
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<td>General</td>
<td>Choose experiment to answer question; preliminary design</td>
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</table>


### Uncertainties of calculated values

The specific methods to approach the uncertainty of calculated value variables in this experiment by referring to table 7.1. The example derivation of uncertainties is shown below and further explain in Appendix x.

### Effect on modelling environments

The evaluation of computing errors has been described depending on sparse and round-off value which normally set in modelling environment. These phenomena could jeopardise the final value of desired calculations. (Find references).

### Misjudge of mitigation and control

#### Conclusions

- Highlighted the important contributions on this area which normally overlook by the researchers. Elaborate how scientifically; the uncertainties are important factors contribute to other area in technical and science area.
- Most of calculated variables in fouling phenomena, authorities and verification can be misjudged scientifically if the understandings of errors of propagation are not being considered in this decision.
- Simulation of sensitivity of parameters towards fouling phenomena for the HiPOR rig will be shown to verify the importance of uncertainties quantification. Furthermore, the boundary of fouling threshold can be developed accurately rather than being misleading by error of propagation of primary measurement.
Appendix C: Flow Control Tuning Parameters

Cold
Appendix C: Flow Control Tuning Parameters

100 C

[Graphs showing flow rate over time for different conditions]
Appendix C: Flow Control Tuning Parameters

150
Appendix C: Flow Control Tuning Parameters

250°C
<table>
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### Appendix C: Flow Control Tuning Parameters

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References


PSE, 2015. gPROMS. Available at: http://www.psenterprise.com/gproms.html.


