

INVESTIGATION ON INDUSTRIAL HEAT TRANSFER FLUIDS  
DEGRADATION AND PREDICTION OF THEIR PHYSICAL  
PROPERTIES USING A SMART TECHNIQUE

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CHEMICAL ENGINEERING  
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**Investigation on Industrial Heat Transfer Fluids Degradation and Prediction of  
Their Physical Properties Using a Smart Technique**

by

Muhammad Farid bin Shahman

15566

Dissertation submitted in partial fulfilment of  
the requirements for the  
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SEPTEMBER 2015

Universiti Teknologi PETRONAS,

32610, Bandar Seri Iskandar,

Perak

CERTIFICATION OF APPROVAL

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(Dr Abbas Azarpour)

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September 2015

## CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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MUHAMMAD FARID BIN SHAHMAN

## **ABSTRACT**

Heat Transfer Fluids (HTFs) are extensively utilized in industry where direct heating by a naked flame is not practical. Historically, steam has been favoured method because its main advantages are the absence of environmental issues and the low cost of water. However, the steam utilization demands the complex system such as the use of chemical additions, demineralised water, safety valves, drains, traps and blow downs. If the HTF systems are designed properly, it is safer and less problematic. Moreover, HTFs are far better than steam as they are able to operate at high temperature and do not require high pressure system. Since the majority of the industrial processes are operated under high temperature, it signifies the importance of the utilization of HTFs in the industrial applications. In this project, the main objectives are to investigate the parameters affecting the degradation of the HTFs, which are commonly used in industry, to predict the physical properties of the fluids using a smart technique as artificial neural network (ANN), to conclude the degradation rate of one type of HTF, and to give some suggestions to lengthen the lifetime of the commonly used HTFs in industry based on the analysis of the results and the investigation fulfilled. ANN is an efficient tool which is widely utilized to analyse the process systems. The results imply that the causes of HTFs degradation are thermal cracking, oxidation and contamination. The results also reveal that ANN has been successfully able to predict HTFs properties with relative percent error as much as 0.262%. Moreover, the degradation rate derived can be utilized to analyse the performance of the HTF system for the next period of operation, and the suggestions provided can be effectively applied for the industrial systems to improve their efficiency and to increase the HTFs lifetime.

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## **LIST OF ABBREVIATIONS**

HTF	Heat Transfer Fluids
ANN	Artificial Neural Network
MWCNT	Multi-Wall Carbon Nano-Tubes
EG	Ethylene Glycol
AIT	Auto Ignition Temperature
WHRU	Waste Heat Recovery Unit
LM	Levenberg-Marquardt
TAN	Total Acid Number

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# CHAPTER 1

## INTRODUCTION

As for the introduction of this project, this part is going to start with the background of study. Furthermore, clear problem statements are to be defined. Next, objectives of the project and the scope of the study are also being described in this part.

### 1.1 Background of Study

The use of thermal fluid system first stated at the end of 1930s. Significant advancement in the technology has been made since the launch of thermal fluid system. Today, thermal fluid systems are much more thermally stable, nontoxic and able to create higher temperature at atmospheric pressure. The degradation of HTFs take place over the course of time, which depends on several factors. It is very important to slow down the degradation of HTFs in order to maintain higher efficiency of thermal fluid system.

The application of HTFs has been widely increased compared to the steam utilization as heat transfer medium in industry. Because thermal fluid system has more advantages, especially in term of high temperature operation. The decision to use HTF as heat transfer medium can be based on many reasons, but one of the major incentives is the use of non-pressurized system.

HTFs are widely used to carry thermal energy in machine cooling applications and process heating. HTFs are also commonly used in industries where high temperatures are required such as oil and gas, aerospace, marine, automotive, and military applications. HTF can also be used in food, beverage and pharmaceutical applications.

Mineral oil, pressure-less synthetic oil and pressurized synthetic oil are common heat transfer mediums used in industry [1]. Hot oils or mineral oils come from the lube cut in the refinery of crude oil. On the other hand, synthetics which are also referred as aromatics, are man-made fluids. They consist of benzene-based structures and include diphenyl oxide or biphenyl fluid, diphenylethanes, dibenzyltoluenes and terphenyls. Figure 1.1 shows the main HTFs and their temperature operating ranges.

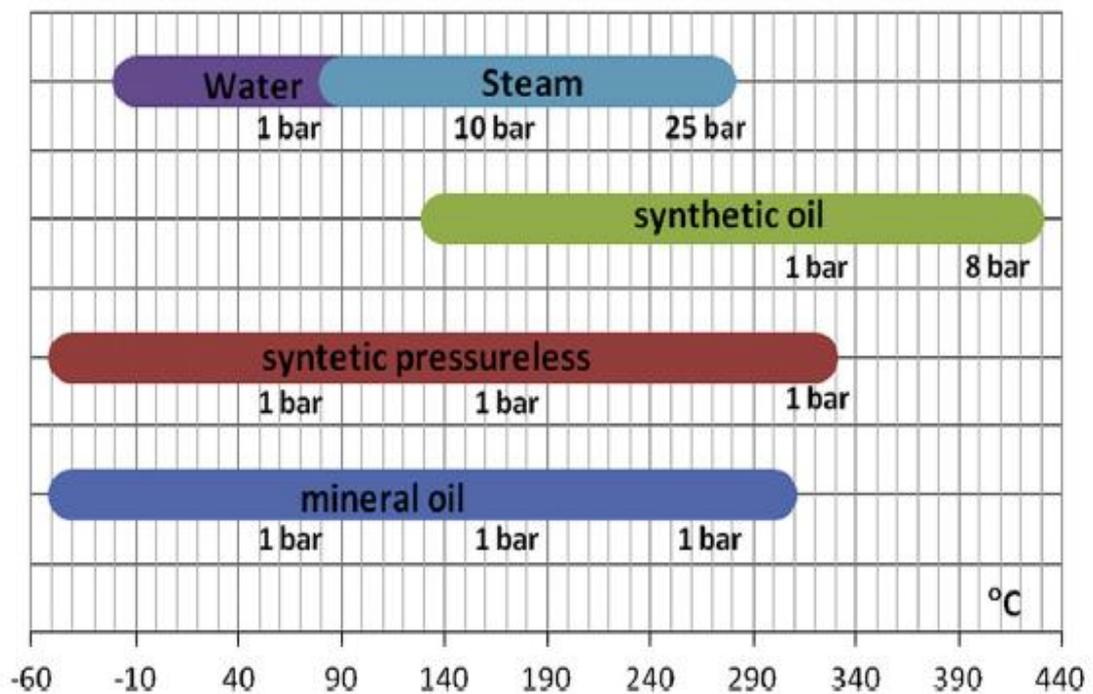


Figure 1.1 HTFs with operating temperature ranges

Days by days, more researches are being carried out to enhance the properties of HTFs. Researches on the use of nanoparticles in HTF have been ongoing for over 15 years [2]. The nanoparticles increase the thermal conductivity of HTF. For example, the addition of copper nanoparticles with a diameter about 10 nm improves the thermal conductivity of ethylene glycol by 40% [3].

Moreover, the HTFs employed in industry have different applications based on the process needs. The HTFs are chosen relying on the operating conditions, especially the temperature. In other words, temperature is the most critical parameter, which determines the efficient usage of the HTFs and their lifetime.

## **1.2 Problem Statement**

The HTFs play an important role in the processes that they are used. Since they provide the high temperature operating conditions for the process, it is very crucial to maintain the proper circumstances related to their operation. Most of them are heated in a furnace which is solely provided to meet the required temperature. The proper control of their condition helps improve their usage for a longer time since the most concerning issue of HTFs is to lengthen their lifetime. Therefore, it is very important to analyse the HTFs conditions, which can be done via the analysis of their physical properties. The knowledge on the physical properties determines the degree of the HTF degradation. Therefore, it is quite helpful to employ a technique which is able to predict their physical properties. Furthermore, the design of the systems which use such oils is not properly carried out. This insufficiency results in the shorter lifetime and the difficulty in the operation of the production process. To sum up, the accurate prediction of the physical properties and the efficient design of the HTFs systems can help to considerably decrease the problems related to their operation.

## **1.3 Objectives**

The objectives of this project are as follows:

1. To investigate the parameters affecting the HTFs degradation
2. To analyse the HTFs rate of degradation and to predict their physical properties using a smart technique as artificial neural network (ANN)
3. To give some suggestions to improve the operation of the industrial HTFs systems

## **1.4 Scope of Study**

Basically, the scope of this project is in the form of analysis of experimented results by using the regression analysis and the ANN modelling. The project covers the properties of a few HTFs, which are broadly used in industry. Nanofluids are a part of study since they have special features, which make them very useful for many engineering application. Besides, the HTFs operation system is one of our concerns since the precision of the system design and its proper control affect the lifetime of HTFs.

## **CHAPTER 2**

### **LITERATURE REVIEW**

The literature is going to be covered with previous studies fulfilled by the researchers. Basically, monitoring the HTFs performance is crucial to the operation in the industry. It can be done with the prediction of the HTFs properties. Using a smart technique such as Artificial Neural Network helps to predict the properties of HTFs with an acceptable accuracy. Besides, the researchers also focus on the effort to improve the properties of HTFs by adding nanoparticles to the base fluids.

#### **2.1 Artificial Neural Network**

In recent decades, artificial neural networks (ANNs) have significantly grabbed the attention of researchers in different scientific fields. The paramount benefits of these networks, compared with prior common methods, are their high speed and ability to solve complicated equations [4]. The thermal conductivity of nanofluids is undoubtedly an important feature that should be measured. Thus, many researchers have conducted different studies on the thermal conductivity of nanofluids [5-12].

In recent years, many researchers have used ANNs to model and simulate the process systems. Kurt and Kayfeci [13] developed an ANN model to predict the thermal conductivity ratio of water/EG based on experimental data. Hojjat et al. [14] conducted an investigation using an ANN ability to propose a thermal conductivity model as a function of temperature, solid concentration, and the thermal conductivity of the nanoparticles. In another study, Papari et al. [15] used an ANN model to predict the thermal conductivity of nanofluid containing multi-wall carbon nano-tubes (MWCNT) in oil, water, and ethylene glycol (EG).

Hemmat et al. [16] conducted an experimental investigation on the thermal conductivity of Magnesium Oxide/Ethylene Glycol. They proposed a thermal conductivity enhancement model in terms of temperature, solid concentration, and particle size using an ANN model, based on the experimental data. Furthermore, in another investigation and based on the experimental data, they proposed two correlations that show the relationships between viscosity, solid concentration, and the nanofluids temperature [17]. Longo et al. [18] used three-input and four-input artificial neural networks to predict the thermal conductivity ratio of oxide-water nanofluid. They employed the temperature, solid concentration, and thermal conductivity ratio of the nanofluid as the three inputs for both of the networks. Moreover, they analysed the effect of the nanoparticle clusters' average size via employing the four-input network. Bhoopal et al. [19] investigated the applicability of ANNs for predicting the thermal conductivity of highly porous metal foams. Modelling of thermal conductivity of Zinc Oxide/Ethylene Glycol using experimental data and ANN method has been carried out by Hemmat et al. [20].

The literature review shows that there is a need to employ a tool which can be effectively used for the prediction of the HTFs physical properties. This tool should be able to predict the essential properties of the HTFs over the wide range of the temperature, which is the most important factor influencing their efficient performance.

## **2.2 Heat Transfer Fluid Properties**

There are some properties that help determine the viability of a heat transfer fluid in a particular application. The most important properties, which are viscosity, pumpability, thermal stability, oxidative stability, carbon residue, and to name a few, are discussed below.

### **2.2.1 Viscosity**

Viscosity is important in the determination of Reynolds and Prandtl numbers for heat transfer systems to estimate fluid turbulence, heat transfer coefficients, and heat flow [21]. Viscosity can be measured using ASTM D445: Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids. The method is carried

out under an accurately reproducible driving head and at a closely controlled temperature. The time for a fixed volume of the liquid to flow through the capillary viscometer is measured. The kinematic viscosity can be calculated from the measured flow time and the calibration constant of the viscometer.

### **2.2.2 Pumpability**

Pumpability refers to the ease of pumping a fluid in a heat transfer system. According to ASTM D 5372, if the fluid viscosity is greater than 200 cS, it is difficult to pump [21]. If a heat transfer fluid is subjected to temperatures below its minimum pumping temperature when not in use, the system should be heat-traced to warm the start-up fluid. The pump and system design determine the viscosity limit required for pumping, and therefore the minimum pumping temperature. The construction of a viscosity versus temperature curve using measured viscosities in the temperature range of interest can be used to estimate minimum pumping temperature.

### **2.2.3 Thermal Stability**

Thermal stability is defined as the resistance of a heat transfer fluid to permanent physical changes caused by exposure to heat. Depending on the composition of the oil, these changes can become noticeable at temperatures as low as 250°C while thermal cracking begins around 350°C [21]. These changes include formation of coke and light ends. If there are substantial amounts of asphaltenes and polar residues in the fluid, the coke may adhere to the heater surfaces, causing fouling, which in turn reduces heat transfer rates. The light ends will cause the overall system pressure to increase, and may result in vaporous cavitation at the pump section. These components will also tend to depress the flash point of the fluid. Thermal cracking is often accompanied by colour change. However, colour change is more common when oxidation is present. While higher viscosity oils are generally more susceptible to cracking than lower viscosity ones, the main cause is due to overheating. Overheating typically results from flame impingement or low velocity through the tubes of fired heaters.

#### **2.2.4 Oxidative Stability**

Oxidation occurs when the hot fluid comes into contact with air. Oxidation rates double every 10°C increasing in temperature [21]. The principal oxidation by-products are organic acids which can undergo further reaction to form extremely high molecular weight components that are soluble in the fluid and increase the viscosity of the fluid. Above 200°C these components will precipitate out on hot surfaces and become insoluble. Insoluble by-products will lead to fouling of heater surfaces, filter plugging, and sticking valves. Any increase in the amount of air contact such as foaming air entrainment and fluid turbulence will also affect the rate of oxidation.

Paraffinic oils have better oxidative stability than naphthenic oils. However, they have lesser solvency, which means poorer ability to dissolve by-products. The vulnerability of petroleum oil to oxidation can be increased by the presence of alkyl aromatic derivatives and some polar components.

#### **2.2.5 Carbon Residue**

Carbon residue is defined as the residue formed by evaporation and thermal degradation of a carbon containing material, which provides an indication of the fluids tendency to form carbonaceous deposits. One test that is used to determine carbonaceous residues is Test Method D 189: the Conradson Carbon Residue. In this test, a sample is added to a crucible and subjected to critical distillation [21]. The residue formed undergoes cracking and coking during the fixed period of heating. At the conclusion of the test, the crucible is cooled and weighed. The Conradson Carbon Residue is the percentage of residue remaining at the end of the test. This test provides a rough approximation of the tendency of the heat transfer fluid to form deposits when heated, and the formation of volatile by-products is possible.

There are a couple of tests which are used to measure carbon residue. One of them, which is frequently used to determine the tendency for a heat transfer fluid to form carbonaceous residues, is Test Method D 524: the Ramsbottom Carbon Residue test. In this test, the sample is weighed into a special glass bulb and placed in a metal furnace where it is heated quickly to 550°C. All volatile material is evaporated out of the bulb, with or without decomposition. The remaining residue undergoes cracking

and coking reactions. After the test, the bulb is cooled and weighed. The residue remaining is reported as the percentage of original sample, and is called the Ramsbottom Carbon Residue.

### **2.2.6 Specific Gravity**

The specific gravity of petroleum and its products is necessary to be determined. The method is carried out in such a way that the sample is transferred to a hydrometer cylinder that has the same temperature. The hydrometer is lowered into the sample and allowed to settle. The hydrometer scale is read after the temperature equilibrium has been reached. By referring to the Petroleum Measurement Tables, the observed hydrometer reading is reduced to the reference temperature. To avoid excessive temperature variation during the test, the hydrometer cylinder and its contents are placed in a constant temperature bath.

### **2.2.7 Metal Content**

Transition metals act as a catalyst for hydrocarbon oxidation. The free radical processes are strongly affected by temperature. Transition metals can act as oxidation initiator or oxidation inhibitor. This depends on their oxidation state. They act as oxidation initiator if they aid in the formation of radicals. On the other side, if they remove the free radicals they will act as oxidation inhibitor.

### **2.2.8 Thermal Properties**

Thermal conductivity and specific heat tests are difficult to carry out. Moreover, facilities for performing them are few, and the precision data is yet to be established. One of the standard tests which able to determine the thermal conductivity is ASTM D 2717: Standard Test Method for Thermal of Liquids [21]. For specific heat, it can be tested using ASTM D 2766: Standard Test Method for Specific Heat of Liquids and Solids. Since thermal conductivity and heat capacity are temperature dependent, to be of use to the designer, they must be measured at different temperatures. Values can be estimated for design use from the general chemical composition. The values for thermal conductivity and specific heat may be available from the fluid supplier.

### **2.2.9 Auto Ignition Temperature**

It is important to note that while HTFs are routinely used above their flash and fire points, they should never be used above their auto ignition temperature (AIT) [21]. AIT is the temperature at which the fluid will ignite spontaneously in contact with air. It is recommended that the fluid supplier be consulted for standard recommended safe practices. The AIT of a heat transfer fluid is determined according to ASTM D 659.

#### **2.2.10 Rubber or Elastomeric Seal**

Heat exchange equipment typically utilizes mechanical seals fabricated from steel or other metal. If elastomeric seals are present, it is desirable that they exhibit rubber swelling in the range of 1%-5% to prevent leakage owing to the poor seal contact [21]. Seals may degrade in some fluids. As oil deteriorates in service, additional tests may be required to assure that seals remain compatible with the altered oil. The temperature ranges of the tests should correspond to the temperatures to which seals will be exposed in service, and the tests should be conducted for at least 1000 h as per ASTM D 471.

### **2.3 Heat Transfer Fluid System**

According to Mitra [22] a thermal fluid system in general is a closed loop heating arrangement with a heat source, which is typically a fired heater or some kind of Waste Heat Recovery Unit (WHRU). The HTF is filled up in the system by a make-up pump through a normally no flow line from the storage tank. To avoid contact with oxygen, which eventually deteriorates HTF quality, the tank is kept under nitrogen blanket. The expansion vessel is usually installed at the highest point of the system to vent any trapped gas as per Figure 2.1. Stable level in the expansion vessel confirms complete filling of the loop.

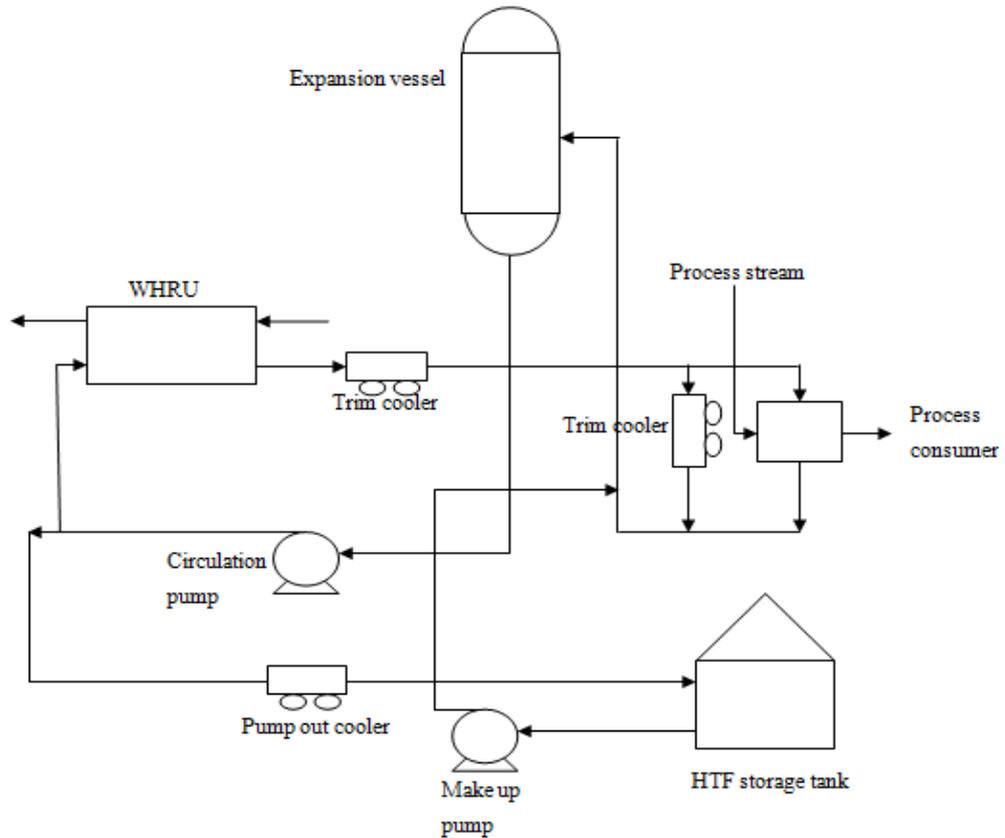


Figure 2.1 Closed loop arrangement of thermal fluid system

HTF is circulated by the circulation pump through WHRU, and heat is supplied to all process consumers. After heat exchange, HTF is returned to the suction of circulation pump. The HTF supply temperature is controlled by a temperature controller at the outlet of trim air cooler which operates on the both main line and bypass line control valve through a split range control mechanism. Temperatures of the process streams are maintained by controlling the HTF flow rates.

Process consumers can be completely bypassed through the full flow bypass line during start up and partially bypassed by sensing the pressure differential through the spill over bypass line when plant runs under turned down condition. Under these circumstances, WHRU is dissipated in the trim cooler on the full bypass line. Volume expansion or contraction of thermal fluid system is accommodated in the expansion vessel. During maintenance of the system or any connected equipment in the loop, HTFs are drained into the storage tank through the pump out cooler.

During maintenance, HTFs are collected from the low point drains of the closed loop piping. Then, it is collected to an underground draining vessel through the dedicated draining network system. The same vessel can be used for system filling purpose using the drain pump. Hot oil drums can be emptied into this vessel through a filling connection. For complete cleaning of this drain vessel, a vacuum truck connection is provided.

Outlet temperature of the fired heater is controlled by a temperature controller which controls the fuel gas flow and HTFs flow to the heater. Due to the reduced demand of HTFs during the plant turndown, pressure of the system increases. The pressure controller senses reduction of flow rate through pressure rise and bypasses the unused HTFs flow through the HTF trim cooler. Temperature at the downstream of trim cooler is controlled by manipulating the motor speed.

#### **2.4 Advancement in Heat Transfer Fluids**

Generally, the era of technical development started in the 21st century, and a lot of changes happened in every industry which includes nanotechnology. A physicist Richard Feynman, Nobel Prize winning in 1959 proposed the concept of micromachines. Then, the term nanotechnology was used for the first time by Norio Taniguchi in 1974. Scientist Choi has marked the starting point for the nanofluids in 1995 when he successfully prepared the first nanofluids [23]. After that, nanofluids have become one of the attentions for many researchers around the world.

A nanofluid can be defined as a conventional base fluid which contains nanoparticles [24]. Basically there are two types of nanofluids, which are metallic nanofluids and non-metallic nanofluids. Aluminium, copper, and nickel are some nanoparticles of metals that are used to prepare metallic nanofluids. On the other hand, non-metal such as metal oxides and various allotropes of carbon are dispersed into the base fluid to synthesize the non-metallic nanofluids. Wei et al. have successfully demonstrated that nanofluids have higher thermal conductivity than the base fluids [25].

Nanofluids have a few of extraordinary properties that allow nanofluids to be used for different engineering applications. Some of the uncommon qualities of nanofluids are:

- Higher thermal conductivity than the base fluids
- Ability to transfer heat faster
- Good stability than normal HTF
- Less erosion and clogging in micro channels
- Less pumping power is needed
- Less friction coefficient
- Better lubrication

Nanofluids can be synthesized by either one-step method or two-step method. In the one-step method, making and dispersing nanoparticles are simultaneous process. For the two-step method, nanoparticles are first generated and then dispersed into the base fluids.

Advantage of one-step synthesis method is that nanoparticles agglomeration is minimized. However, the main problem is the process only applicable for low vapor pressure fluids. One-step preparation process of nanofluids is given in the Figure 2.2.

Two-step method is expansively used in the preparation of nanofluids by mixing the base fluids with existing nanoparticles. Nanoparticles can be obtained through a process such as grinding, milling, and vapor phase methods. Generally, nanoparticles are stirred with the base fluids using high shear mixing device or ultrasonic vibrator. This is carried out to reduce the particles agglomeration. There were three experiments reported using two-step method to produce alumina nanofluids which are Eastman et al. [26], Lee et al. [27], and Wang et al. [28]. Besides, Murshed et al. [29] also used the same method to synthesize titanium dioxide-water nanofluids. Eastman et al. [30] mentioned that preparation of nanofluids containing oxide nanoparticles are more suitable to use the two-step method compared to metallic nanofluids.

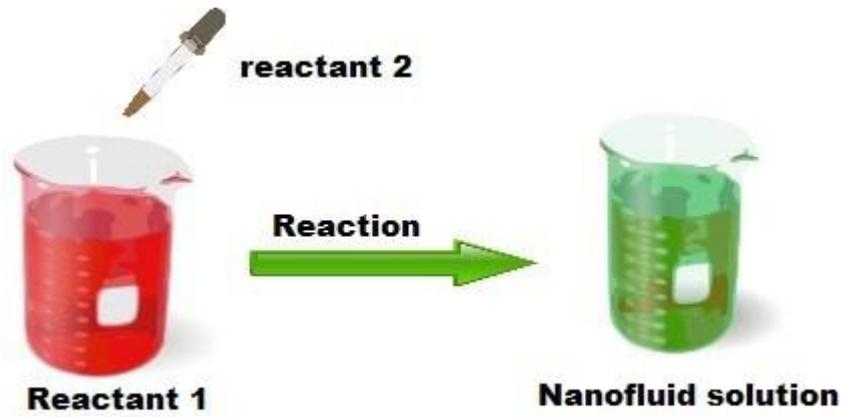


Figure 2.2 One-step preparation process of nanofluids

Stability is a common issue that related to the HTFs synthesis since the nanoparticles easily accumulate due to strong Van Der Waals force among nanoparticles. Despite this disadvantage, this process is still accepted as the most feasible process for nanofluids preparation. The most common two-step method is shown in Figure 2.3.

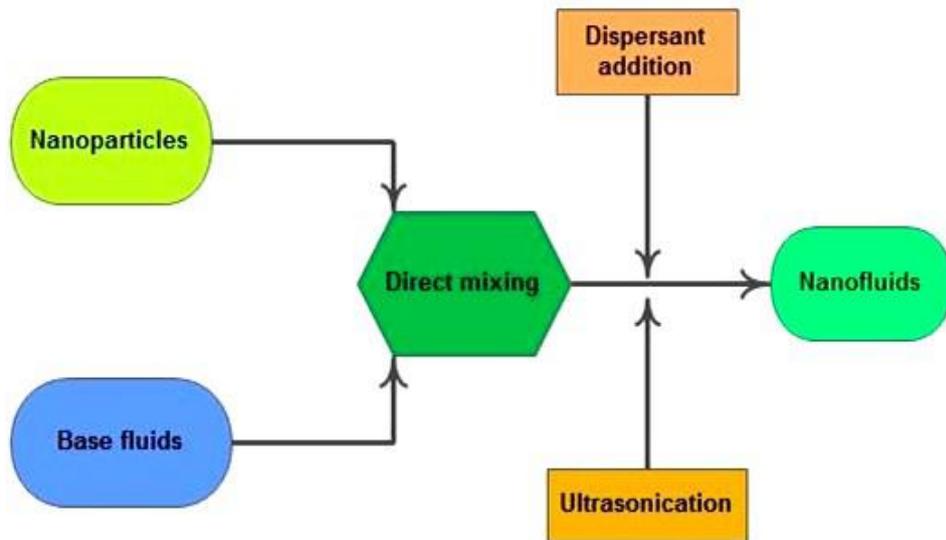


Figure 2.3 Two-step preparation process of nanofluids

## CHAPTER 3

### METHODOLOGY

#### 3.1 Project Flowchart

##### Problem Statement and Objectives

- The problem related to the HTFs degradation, their physical properties prediction, and the improvement of the HTFs system are addressed. Accordingly, the objectives of the project are defined.

##### Literature review

- The HTFs degradation, their physical properties prediction, and the techniques used to predict them will be analysed through the in-detail review in the studies fulfilled up to now.

##### Methodology

- To carry out the ANN technique to predict the properties of HTFs.
- To execute the analysis on the degradation rate of HTFs.
- To propose suggestions to lengthen the lifetime of HTFs.
- To perform the regression analysis on the particle concentration

##### Data Collection and Analysis

- Data collection from the literature
- Analysis of the data and the discussion on the results

##### Conclusion

- To conclude the project
- To prepare the report

### 3.2 Gantt Chart and Key Milestones

No	Detailed work	1	2	3	4	5	6	7	8	9	10	11	12	13	14	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of project topic	█	█																										
2	Preliminary research work		█	█	█	█	█																						
3	Submission of extended proposal							●																					
4	Perform a study on the factors that affect thermal conductivity of nanofluids.								█	█																			
5	Perform the regression analysis on the particle concentration										█	█																	
6	Submission of interim draft report												●																
7	Submission of interim report													●															
8	Carry out the ANN modelling on the properties of HTFs														█	█	█												
9	The factors affecting HTFs degradation															█	█												
10	Propose suggestions to lengthen the lifetime of HTFs																█	█	█										
12	Progress report																						●						
14	Pre-SEDEX																						█	█	●				
15	Dissertation write up																								█	█	█		
16	Viva presentation																											█	█
17	Submission of dissertation																												●

### **3.3 Regression Model**

There is a statistical process for estimating the relationships among variables which called the regression analysis. This technique mainly focus on the relationship between a dependent variable and one or more independent variables. In this project, the regression strategy will be applied to study the effect of particle concentration on thermal conductivity of specified nanofluids.

#### **3.3.1 Regression Analysis**

Specifically, regression analysis related on how the representative value of the dependent variable changes when any one of the independent variables is varied, while the other independent variables are held fixed. Most commonly, regression analysis estimates the conditional expectation of the dependent variable given the independent variables. In all cases, the estimation target is a function of the independent variables called the regression function. In regression analysis, it is also relevant to characterize the variation of the dependent variable around the regression function which can be described by a probability distribution.

Many techniques for carrying out regression analysis have been developed. Linear regression and ordinary least squares regression are some establish familiar methods. The performance of regression analysis methods depends on the form of the data generating process. Besides, it is also relates to the regression approach being used. Since the true form of the data-generating process is generally not known, regression analysis often depends to some extent on making assumptions about this process. These assumptions are sometimes acceptable if a sufficient quantity of data is available.

#### **3.3.2 Regression Validation**

Regression validation is the process of determining whether the numerical results are acceptable as descriptions of the data. The validation process is to analyse the accuracy of the regression, checking whether the model's predictive performance deteriorates substantially when applied to data that were not used in model estimation. Usually, the regression validation used  $R^2$  value.

### **3.4 Artificial Neural Network**

ANNs are computational models of the biological brain. Like the brain, a neural network comprises a large number of interconnected neurons. Each neuron is capable of performing only simple computation. The architecture of an artificial neuron is simpler than a biological neuron. The work flow for the neural network design process has five primary steps which are going to be described in this part.

#### **3.4.1 Collecting the Data**

ANN determines an empirical relationship between the inputs and outputs of a given system. Where the inputs of the system are the independent variables, and the outputs are the dependent variables. Most of neural networks incorporate an input layer, at least one hidden layer, and an output layer. In this project, the input will be the temperature of HTFs while the outputs are the HTFs properties.

#### **3.4.2 Creating the Network**

Figure 3.1 shows that the neural network has three neurons in the hidden layer and three neurons in the output layer with a log-sigmoidal and linear transfer functions in the hidden and the output layers, respectively. The input P matrix to train the neural network is formed by physical parameters and functional groups interactions information of a given binary mixture system. The variables  $w$ 's are the weight matrices,  $b$ 's are the bias vectors,  $n$ 's are the net input, and  $a$ 's is the output of the neural network.

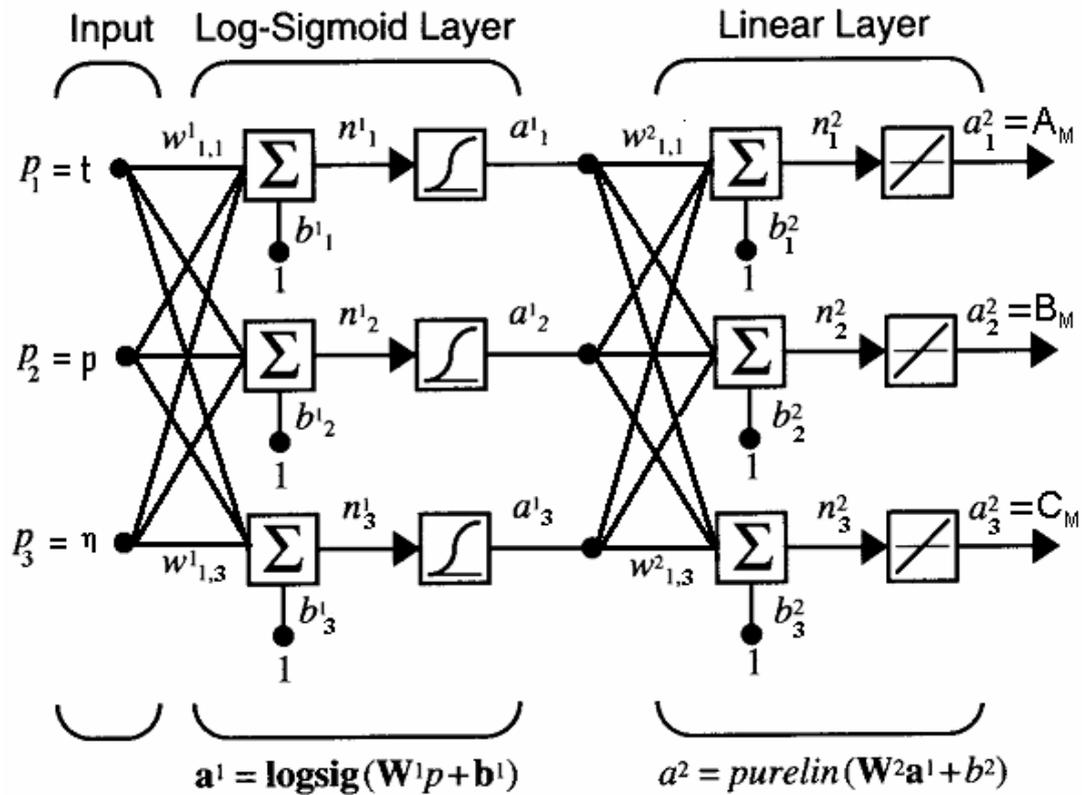


Figure 3.1 Neural network

In this project, different number of neurons will be tested. This is to find the best number of neurons which suits with the ANN modelling resulting the minimum error.

### 3.4.3 Training the Network

It is very difficult to know which training algorithm will be the fastest for a given problem. It depends on many factors, including the complexity of the problem, the number of data points in the training set, the number of weights and biases in the network, the error goal, and whether the network is being used for pattern recognition or function approximation. This section compares the various training algorithms. The following Table 3.1 lists the algorithms that are tested and the acronyms used to identify them.

Table 3.1 List of training algorithms

Acronym	Algorithm	Description
LM	trainlm	Levenberg-Marquardt
BFG	trainbfg	BFGS Quasi-Newton
RP	trainrp	Resilient Backpropagation
SCG	trainscg	Scaled Conjugate Gradient
CGB	traincgb	Conjugate Gradient with Powell/Beale Restarts

#### 3.4.4 Validating and Testing the Network

The next step are validating and testing the network. This step is find the relationship between the outputs of the network and the targets. If the training was perfect, the network outputs and the targets would be exactly equal. But usually the relationship is rarely perfect in practice.

#### 3.4.5 Using the Network

After the ANN modelling has the best performance, it can be used to predict the properties of HTFs. In this project, ANN is being used to predict the properties of commercial HTFs which are Therminol 66, Therminol 72, and Jarytherm.

## **CHAPTER 4**

### **RESULTS AND DISCUSSION**

Throughout this part, results for physical properties prediction are going to be discussed. Besides, factors that affect the HTFs degradation will be briefly explained. Furthermore, the prediction of the degradation rate of one type of HTFs also is provided here. The regression analysis on the effect of particle concentration towards the thermal conductivity of specified nanofluids also has been provided.

It is to be mentioned that the prediction of HTFs physical properties was fulfilled through coding into Matlab software environment, and also via Matlab software ANN toolbox. The data used for the devised model and the results of the ANN model are depicted in Appendices.

#### **4.1 Physical Properties Prediction**

Through coding into Matlab software environment and providing different training algorithms, the best training algorithm has been chosen. The results have been reported in Table 4.1. Levenberg-Marquardt (LM) is the best training algorithm for the network considering the performance values.

Table 4.1 Performance of different training algorithms

Training Algorithms	Performance value
<b>Levenberg-Marquardt</b>	$4.41 \times 10^{-10}$
<b>BFGS Quasi-Newton</b>	$1.10 \times 10^3$
<b>Resilient Backpropagation</b>	$6.58 \times 10^2$
<b>Scaled Conjugate Gradient</b>	$9.57 \times 10^2$
<b>Conjugate Gradient with Powell/Beale Restarts</b>	$8.08 \times 10^2$

To choose the best network to predict the physical properties of the HTFs, different neurons have been considered in the hidden layer. The result for each neuron number is tabulated in Table 4.2. Based on the results, the best neuron number is 27.

Table 4.2 Performance of different neuron numbers

Neuron numbers	Performance Value
<b>23</b>	$1.94 \times 10^{-7}$
<b>25</b>	$2.15 \times 10^{-8}$
<b>27</b>	$5.93 \times 10^{-9}$
<b>30</b>	$7.76 \times 10^{-8}$
<b>33</b>	$8.60 \times 10^{-6}$

Based on the results, the best network has been chosen to produce the results. The best network was trained with trainlm (Levenberg-Marquardt training algorithm) with 27 neurons in the hidden layer. The result is compared with the experimental value. Figure 4.1 shows the prediction of the viscosity of Therminol 66, which is commonly used in industry. As it can be seen, the ANN model outputs and the experimental data are very close to each other implying the good agreement between the model outputs with the experimental data.

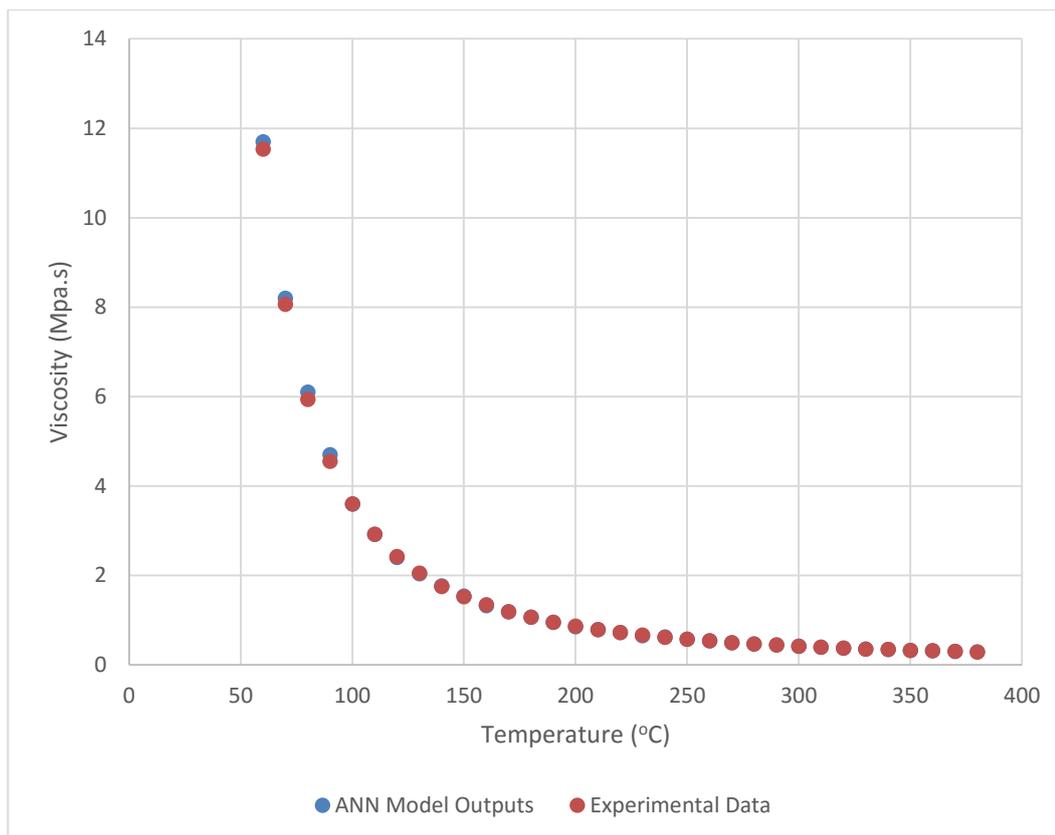


Figure 4.1 The comparison between the ANN model outputs with the experimental data for the prediction of Therminol 66 viscosity

Figure 4.2 represents the comparison of the density values predicted by the model with the experimental data for Therminol 66. As it can be figured out, the model reasonably predicts the physical property.

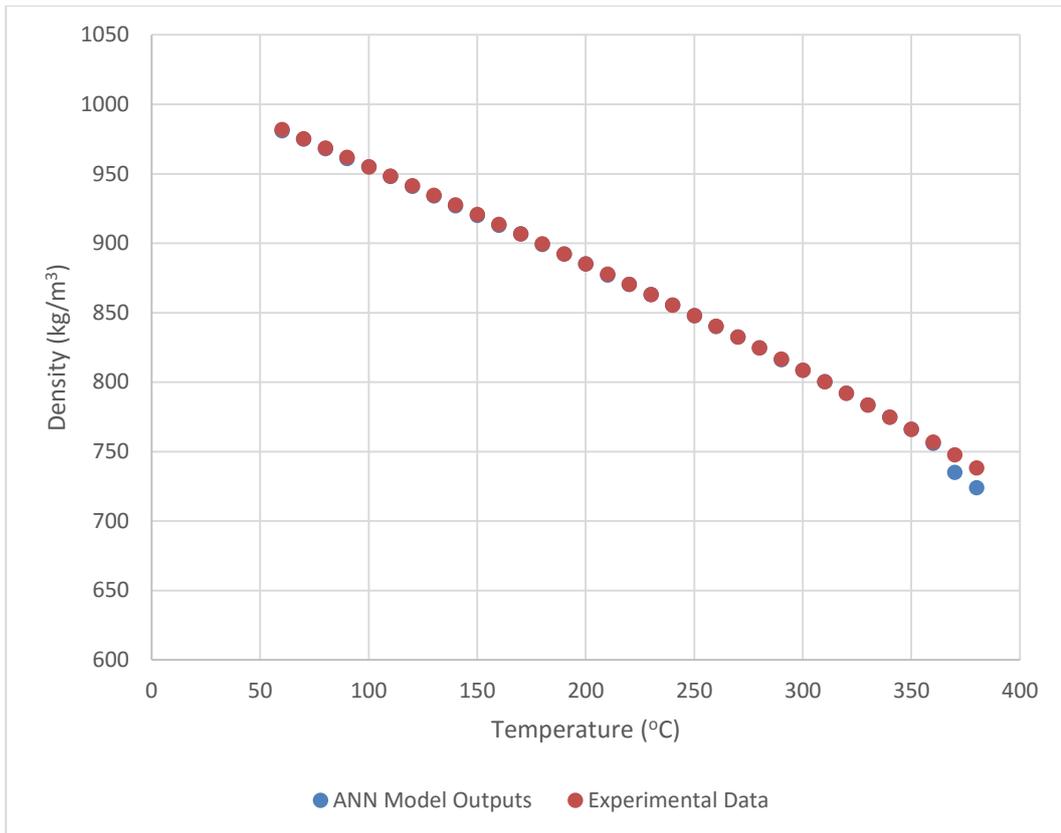


Figure 4.2 The comparison between the ANN model outputs with the experimental data for the prediction of Therminol 66 density

Figure 4.3 shows the comparison of thermal conductivity values predicted by the model with the experimental data for Therminol 66. As it can be implied, the model performance is acceptable.

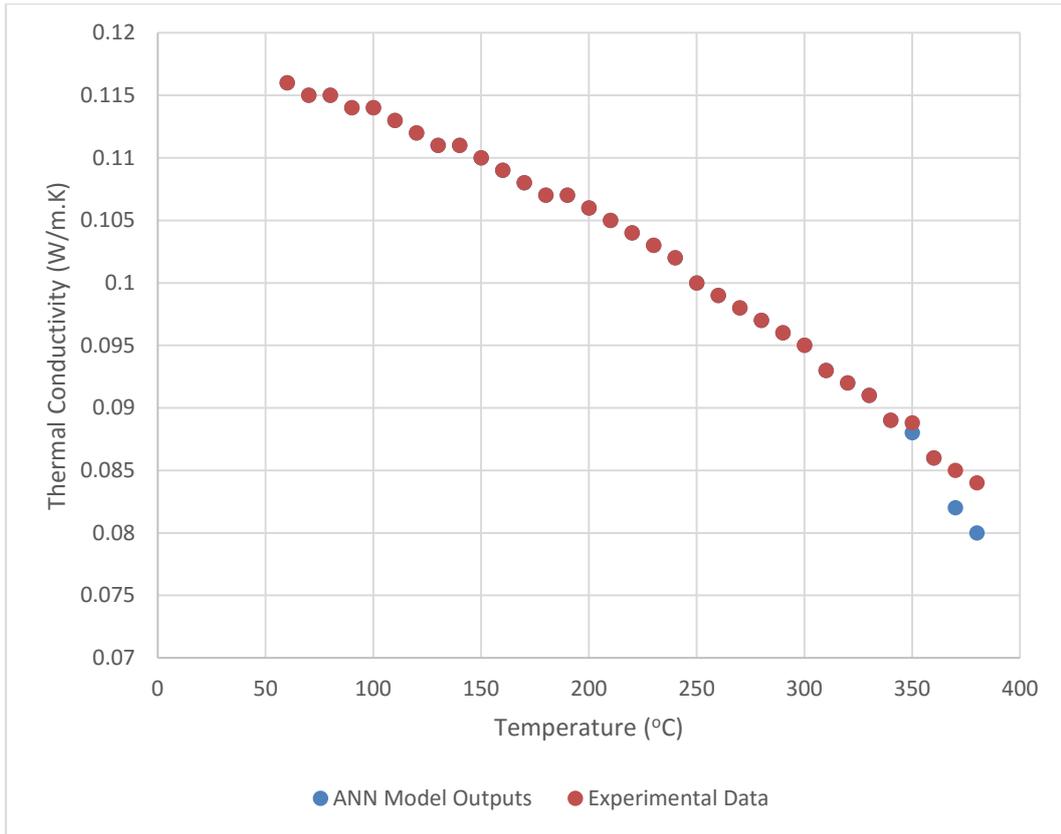


Figure 4.3 The comparison between the ANN model outputs with the experimental data for the prediction of Therminol 66 thermal conductivity

Figure 4.4 illustrates the comparison of heat capacity amounts predicted by the model with the experimental data for Therminol 66. As we can see, there is only one point of ANN output model which different from the experimental data. The point is 230°C.

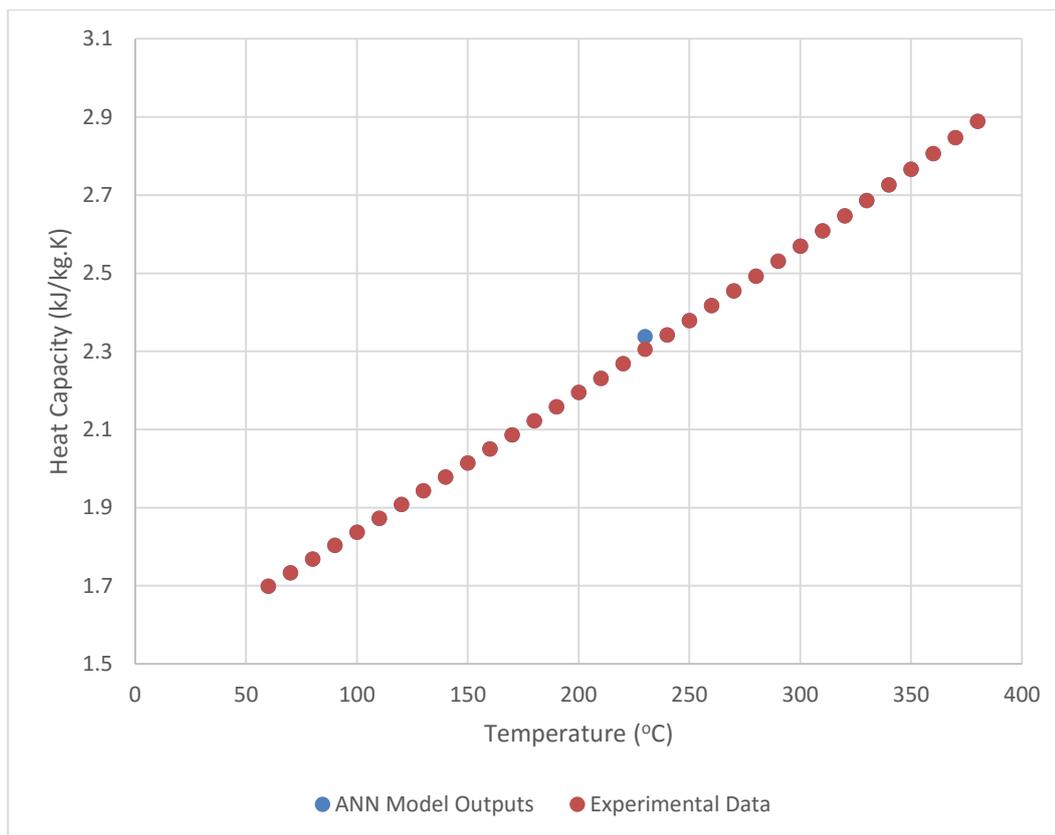


Figure 4.4 The comparison between the ANN model outputs with the experimental data for the prediction of Therminol 66 thermal heat capacity

Based on the result for each properties of the HTF, Therminol 66, the error is calculated using the following equation

$$relative\ percent\ error = \frac{Experimental\ data - ANN\ model\ outputs}{Experimental\ data} \times 100\%$$

The error calculated is to be found as much as 0.262%. It can be concluded that ANN is a good tool to determine the properties of HTFs.

Figure 4.5 displays the comparison of viscosity and density values predicted by the model with the experimental data for Therminol 72. It is clear that the model predicts the physical properties significantly.

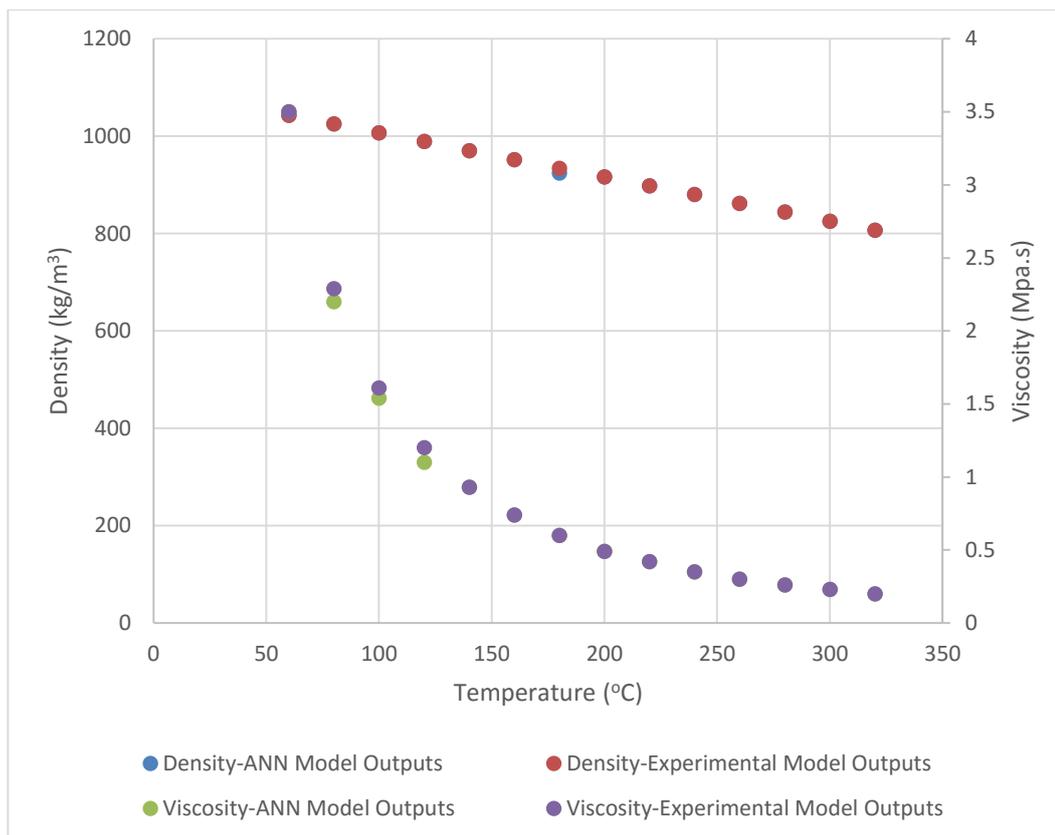


Figure 4.5 The comparison of the density and viscosity amounts predicted by ANN model with the experimental data for Therminol 72

Figure 4.6 depicts the comparison of heat capacity and thermal conductivity values predicted by the model with the experimental data for Therminol 72. It reveals that the model devised is able to predict the physical properties very well.

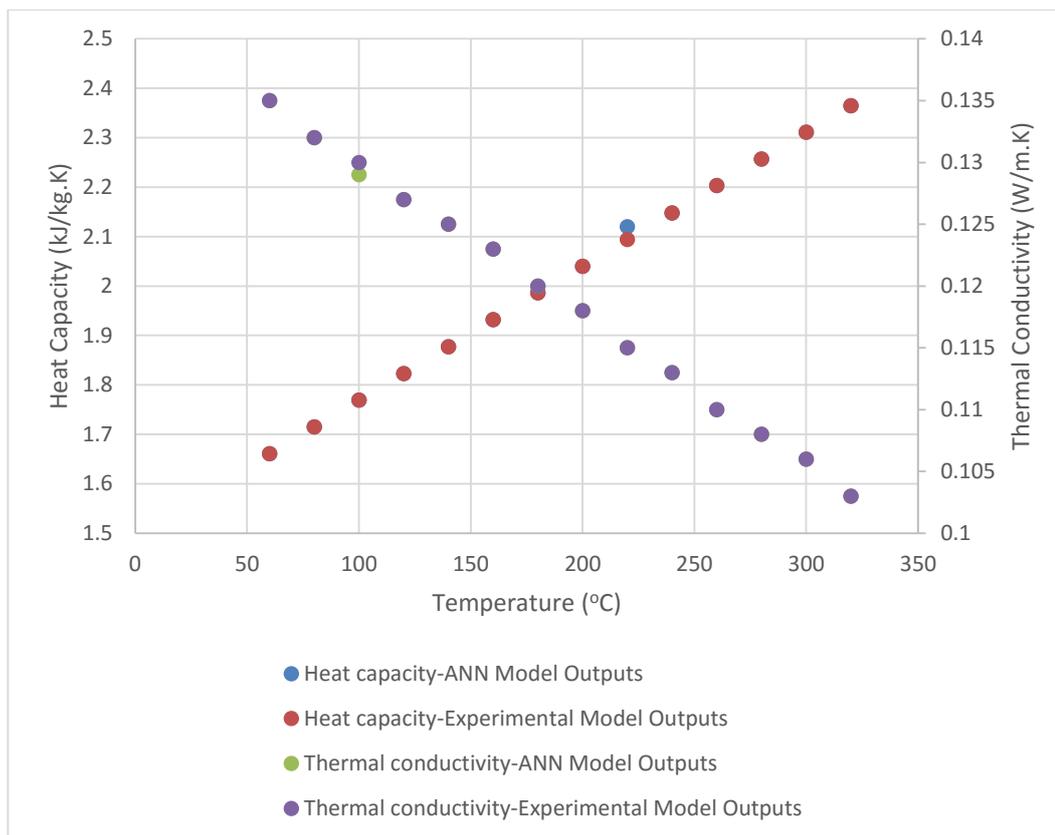


Figure 4.6 The comparison of the heat capacity and thermal conductivity amounts predicted by ANN model with the experimental data for Therminol 72

Figure 4.7 illustrates the comparison between viscosity and density amounts predicted by the developed model with the experimental data for Jarytherm, which is extensively used in the industrial production processes. It can be noticed that the model precisely predicts the physical properties.

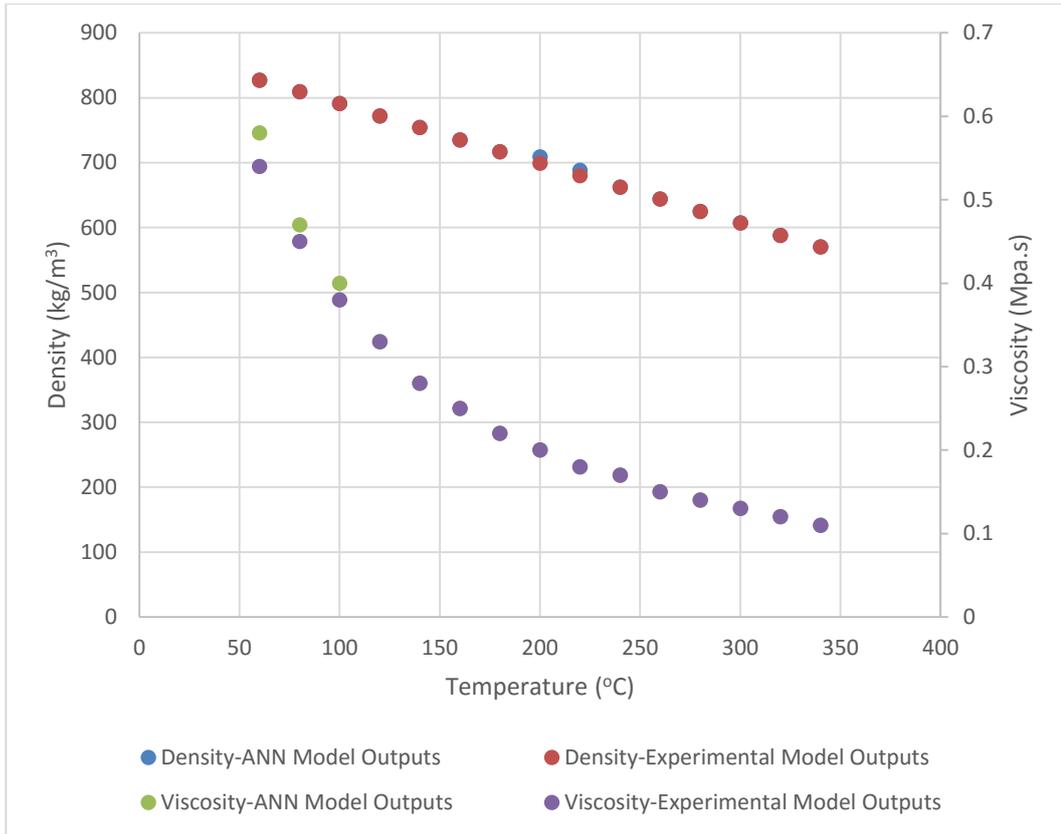


Figure 4.7 The comparison of the density and viscosity amounts predicted by ANN model with the experimental data for Jarytherm

Figure 4.8 shows the comparison between thermal conductivity and viscosity values predicted by the developed model with the experimental data for Jarytherm. It is clear that the devised model is efficient.

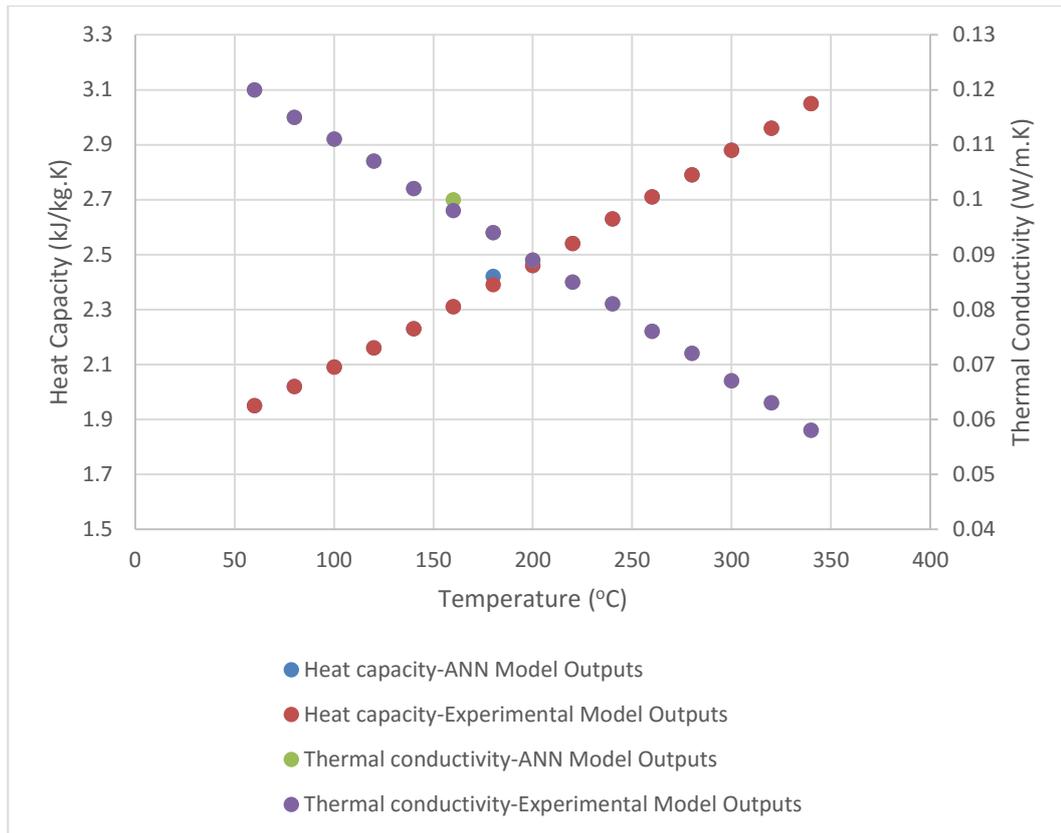


Figure 4.8 The comparison of the heat capacity and thermal conductivity values predicted by ANN model with the experimental data for Jarytherm

## 4.2 Effective Factors on the Degradation of HTFs

Over time, HTFs degrade and cause problems to the thermal fluid system. Generally, there are three major reasons for HTFs degradation, which are oxidation, thermal cracking and contaminants. This section will describe the factors affecting the HTFs degradation.

### 4.2.1 Thermal Cracking

Thermal cracking can take place whenever the heat transfer media absorb more heat than its capability at that particular time regardless of the chemistry of the heat transfer media. This is due to the formation of shorter hydrocarbons. Reduction of the overall fluid viscosity and increase of the volatility are the usual results of thermal cracking, which subsequently increase the risk of leakage and loss through evaporation. Besides, thermal cracking reduces the flashpoint, fire point, and AIT, and increases the vapor pressure. All these reduce the efficiency of HTFs.

It is discovered that in a typical heat transfer system, low viscosity fluids have better heat transfer behaviour in forced convection situation. It might be thought that thermal cracking is an advantage from thermal conductivity point of view. However, the operation of a closed system is unsafe when the operating temperature nears or exceeds the AIT. During thermal cracking, shortened molecules are not the only species formed. On the other hand, an open system is cannot be tolerated since the heated fluid is constantly in contact with the atmosphere. Drop in flashpoint and fire point could put in danger the entire operation.

Table 4.3 gives information on how thermal cracking leads to the decrease in flashpoint and viscosity of a typical HTF. Furthermore, it can be observed that the initial distillation point (GCD 10%) drops over time, which indicates an increase in the concentration of low boiling components.

The formation of carbonaceous residues is another major consequence of thermal cracking. Figure 4.9 shows the consequence of thermal cracking resulting the carbonaceous residues formation. Such undesirable carbon residues are not only destructive toward the piping but it also tends to stubbornly stick and harden onto the hot spot. Subsequently, it forms an insulation layer inside the pipe. This incident requires increasing energy consumption to maintain the desired operating fluid temperature.

Table 4.3 Analysis data showing thermal cracking of one type of HTF

Sample date	Flash point (°C)	Water content (ppm)	Viscosity at 40 °C (cSt)	GCD		
				10% boiling (°C)	90% boiling (°C)	% boiling below 335 °C
04/04/00	154	660	27	327	512	10.49
10/08/01	155	580	23.2	307	507	14.4
11/06/02	175	313	22.7	295	490	12.8
09/09/02	171	51	21.2	283	481	31.9
09/12/02	161	220	20.5	276	489	16.2
12/03/03	175	42	19.8	254	490	19
new fluid properties	209	-	35.6	380	498	0.8

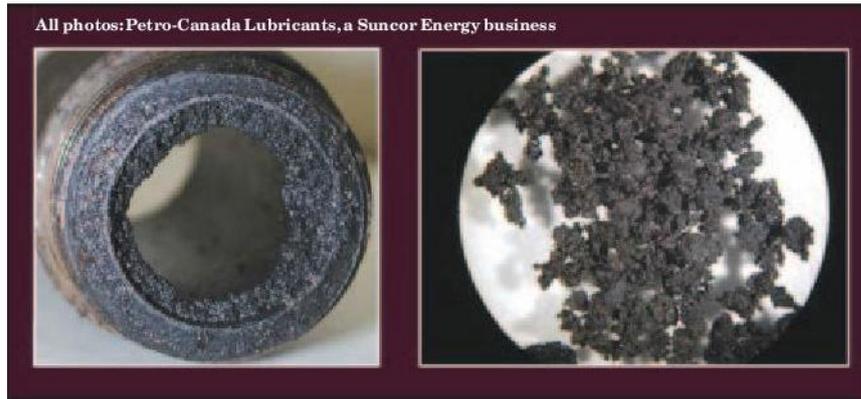


Figure 4.9 Carbonaceous residues

The system run at temperatures that are considered to be relatively mild is not excused from thermal cracking. For example, the system experiencing a defective pump, a fluid containing solids, some piping restriction has encountered carbon deposition that acts as insulation layer. These factors demand higher energy consumption in order to maintain the fluid operating temperature. Consequently, these factors lead to an increase in the skin film temperature. Thus, the fluid at the heated surface will receive more energy than its capacity.

Since the HTF viscosity is the most important factor to be analysed for the degradation phenomenon, the data of a typical HTF over the course of time have been utilized to squash out a simple relation relating the mentioned physical property with the time of operation. Therefore, Figure 4.10 represents the degradation of the HTF over time which extracted from Table 4.3. As it can be figured out, there is a reduction in the viscosity. This trend can be used as reference to have a look on the performance of HTFs for the next operation. The equation (1) relating the viscosity with the time of operation follows:

$$\mu = 2 \times 10^{-6}t - 0.0128t + 35.426 \quad (1)$$

Where  $\mu$  = viscosity (cSt)

t = period (days)

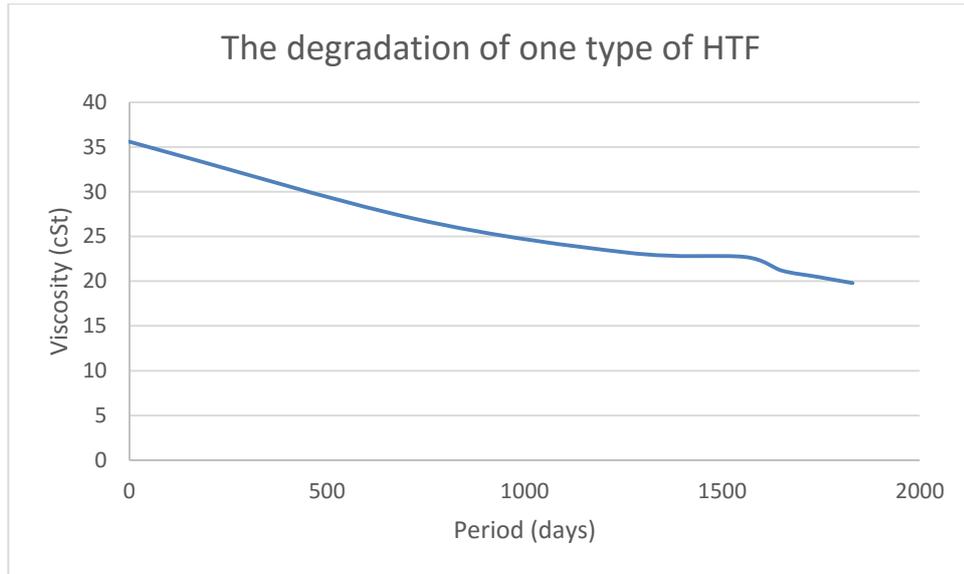


Figure 4.10 The degradation rate of one type of HTF

#### 4.2.2 Oxidation

The other important factor affecting the HTF degradation is the oxidation reaction of the fluid with air. The oxidation of organic compounds in the HTFs is complicated because it involves a series of chemical reactions that result in the high energy consumption, system instability, and reactive free radicals and peroxides production. Moreover, the fluid becomes more corrosive..

Additionally, the acids synthesized due to the oxidation are polymerized and able to modify the fluid properties, causing precipitation as varnish and sludge as illustrated in Figure 4.11. Due to large pipe diameters and valves with high tolerance, the varnish formation is not a serious concern. Nevertheless, further oxidation promotes the formation of heavier acids and sludge. Sludge is not very soluble in HTFs, so it has a tendency to adhere to metallic surfaces or settle in low flow area. Sludge can also move throughout the system and stuck in the control valve.



Figure 4.11 Varnish (left) and sludge (right) resulting from oxidation cause of degradation

### 4.2.3 Contamination

Tentatively, contamination is unlikely in view of the fact that the pressure is greater on the fluid side. However, there are many occurrences that lead to the HTFs stream contamination. The necessity required to repair a process leak rely on the type of contaminant, severity and the heat transfer media that it makes contact with. For example, a natural gas extraction facility bears an unintended leak of the process hydrocarbons into the HTFs system. The heated gaseous molecules mix up well with HTFs and within a short period of time, the HTFs viscosity heavily drops.

One more example of process contamination happens regularly at asphalt terminals. Any unintentional asphalt entering in HTFs will blend well with most of the fluids. The extremely viscous asphalt will speedily thicken HTFs. It will raise the viscosity of HTFs to hundred centistokes or even worse up to a level that it cannot be measured at 40°C. By this means, it ruins the fluid ability to transfer heat effectively. Besides, the heavy asphalt surely plugs the small lines and coats the system internals. In certain cases, even though the contaminant may be inert to HTFs but it may still form acidic or insoluble compounds when it reacts with traces of moisture.

Subsequently, it accelerates rust and causes fluid degradation and corrosion to the system.

On the other side, contamination could take place by air, the wrong fluid putting into the system, and the environment. For example, the system where the expansion tank is located outside and vented to the atmosphere tends to encounter this issue. This is because snow and rainwater falling directly to the expansion tank. If the system has any leakage or small hole, definitely oxidation will occur due to high water content.

Moreover, traces of water-based solutions or aggressive cleaning fluids could speed up fouling and corrosion. Water in HTFs is unforgiving because prolonged exposure to water causes pump cavitation and wear, hastens oxidation degradation and accelerates rust and corrosion to the system internals.

#### **4.3 Suggestion to Lengthen the Lifetime of Industrial HTFs**

Based on the findings through the literature review, physical properties values analysis through the developed model, and the industrial experience of a typical system, some suggestions are proposed which can be utilized in order to lengthen the lifetime of industrial HTFs. The suggestions are:

1. To choose the right HTF considering the process operating condition and the process needs. For example, the selection is to be done considering the range of HTF temperature in the operation and the fluctuation of the control parameters.
2. The venting is to be provided in the HTF system to not release the vapour into the atmosphere. It is suggested to send such HTFs to the collection drum.
3. Small pressurization of the HTF drum with an inert gas like nitrogen.
4. Fresh fluid needs to be added to the system as makeup to maintain the system efficiency. In this case, some HTF are to be removed from the system. The amount need to be removed is the same as the amount the user add to the system. This should be done after the analysis of the physical properties, especially viscosity, of the sample taken from the system.

5. It is advised the fresh fluid must never be added directly into the hot fluid stream. Instead, it should be added to the expansion tank.
6. Not to mix different HTFs in case of HTF lack of stock.
7. A sampling point is to be installed to do some analysis work on HTF. For example, to measure the flashpoint, auto ignition temperature and viscosity to know what is the condition of the HTF system. The results are to be compared with the fresh HTF properties.
8. During any operation, it is favoured to determine the rate of generation of low boilers during any operation.
9. It is recommended that the HTF system is to be heated up or cooled down gently with a specific rate for start-up and shutdown procedure. Based on the system operating temperature and the supplier suggestion, the rate of heat-up and cool-down of HTF is proposed.
10. For any operating modification which affects HTF efficiency, it is required to consult the supplier.
11. To dispose the collected HTF from the cleaning operation. It is not to be returned to the HTF loop.
12. It is advocated to continuously put the HTF filter in service.
13. Adding some more filters in the HTF circle is much recommended.
14. The pump strainers are needed to be cleaned regularly.
15. The HTF circulation pump should be well maintained. Specifically, pump seals and drive bearings on the electric motor should receive proper attention.
16. Daily inspections are recommended by using a consistent checklist of items to monitor. For example, fired heaters should be checked for flame impingement.

#### **4.4 Effect of Particles Concentration on Nanofluids Properties**

Concentration of particles would be regarded as one of the most significant features affecting thermal conductivity of nanofluids. Basically, it is expected that adding nanoparticles would improve heat transfer performance of nanofluids and also would increase thermal conductivity of them. It is also predictable that the more concentration, up to a certain reasonable limit, would cause the more increase in

thermal conductivity. Figure 4.12 shows thermal conductivity versus temperature for different concentrations.

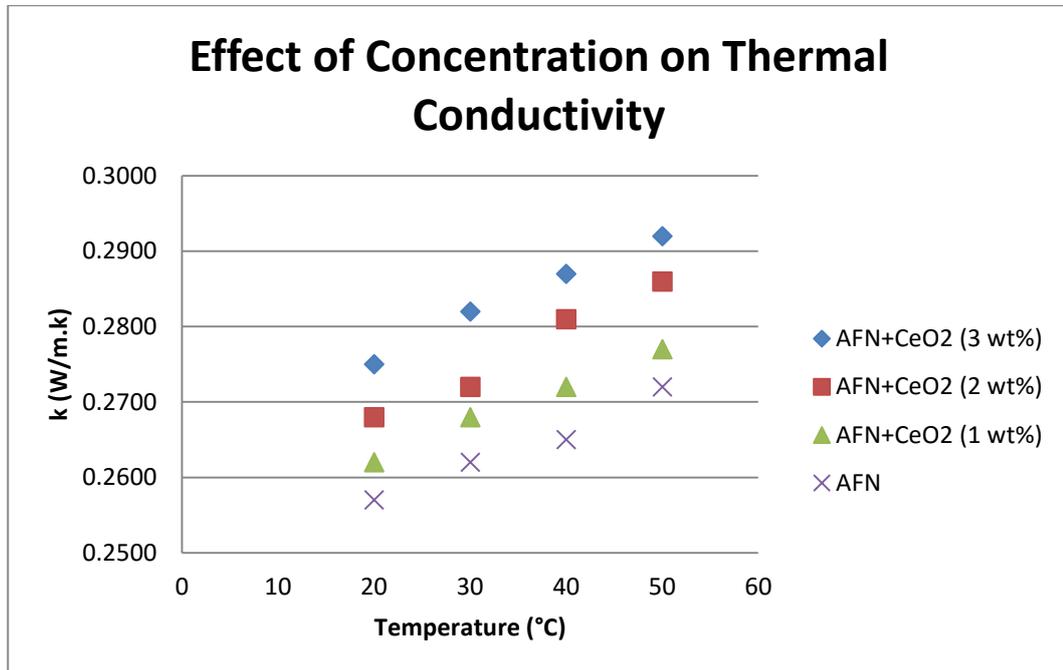


Figure 4.12 Effect of concentration on thermal conductivity at different temperatures

Three types of regression models are developed including linear, polynomial and exponential. This is to find the best regression model to relate the particle concentration with thermal conductivity. Table 4.5 is the summary of all the regression models. The polynomial has been chosen as it fits the most data of the experimental work.

Table 4.5 Evaluation on type of regression

Type of regression	R <sup>2</sup> value			
	20°C	30°C	40°C	50°C
<b>linear</b>	0.9945	0.966	0.9947	0.9888
<b>polynomial</b>	1	0.9849	0.9956	0.9898
<b>exponential</b>	0.9959	0.9693	0.994	0.9892

Resulting from the polynomial regression model, the equation (2) is developed to find the relationship between the particle concentration,  $C_p$  (wt%), and thermal conductivity,  $k$  (W/m.K).

$$k = 0.00048C_p^2 + 0.00635C_p + 0.2639 \quad (2)$$

The regression model has been compared to the experimented values. Thus, the percentage error is calculated as shown in Table 4.6. As it can be seen from the table, the regression model is acceptable since the highest percentage error is 3.5%.

Table 4.6 The percentage error for the regression model

Concentration (wt%)	Percentage error (%)			
	20°C	30°C	40°C	50°C
<b>3</b>	3.305204	0.843882	0.914205	2.672293
<b>2</b>	3.449518	2.008466	1.233901	3.035216
<b>1</b>	3.231764	1.015697	0.461681	2.308403
<b>0</b>	2.623851	0.729374	0.407313	3.059581

## **CHAPTER 5**

### **CONCLUSION AND RECOMMENDATION**

In this project, the influential parameters affecting on the HTFs degradation have been done. Moreover, the rate of degradation and the prediction of HTFs physical properties have been fulfilled using ANN model. Further, relying on the investigation accomplished and the analysis of the results fulfilled in this study, some effective suggestion have been provided to improve the HTFs system function and to enhance the HTFs lifetime utilized.

The model developed in this project can be generally used for the performance analysis of the HTFs systems and for the accurate prediction of the HTFs physical properties.

It can be concluded that ANN model structured by the Levenberg-Marquardt training algorithm and 27 number of neurons in hidden layer has the best performance for the prediction of the HTFs physical properties. Also, it has been figured out that the cause of HTFs degradation are thermal cracking, oxidation and contamination. Besides, a couple of suggestions have been made to lengthen the lifetime of HTFs along with the enhancement of the system performance. On the other hand, the regression analysis on the effect of particle concentration towards the thermal conductivity of nanofluids has been successfully carried out.

For recommendation, it is suggested that an experimental study be carried out considering the time to conclude the rate of degradation for a special HTF which is used in industry. To fulfil this, the system is to be operated in high temperature for a long time to adequately simulate the experimental setup with the actual system.

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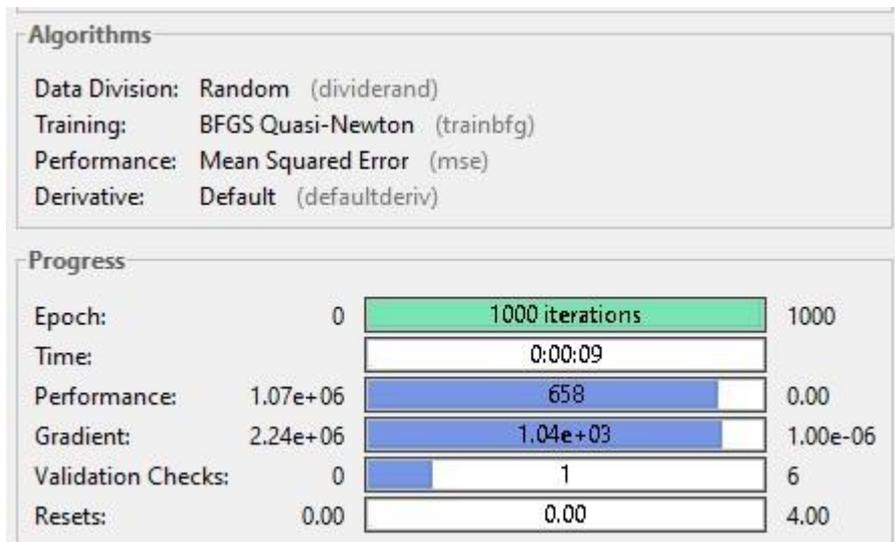
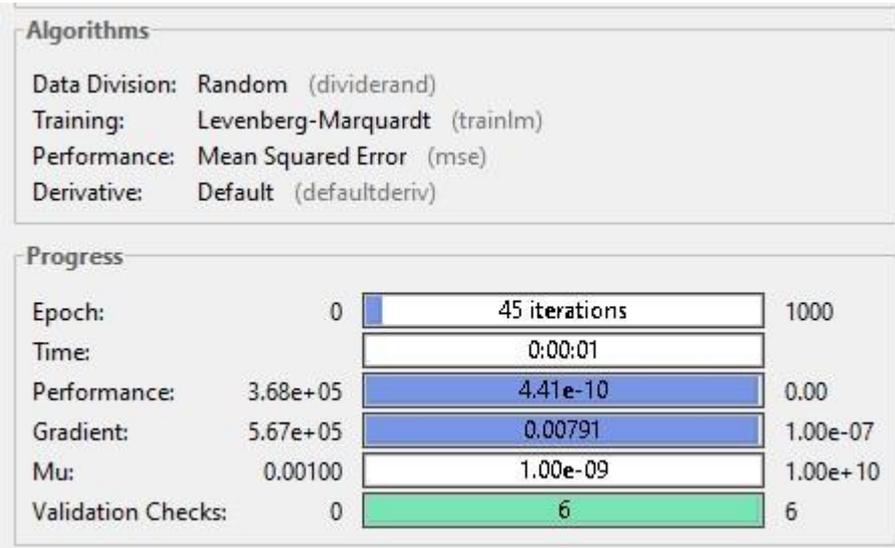
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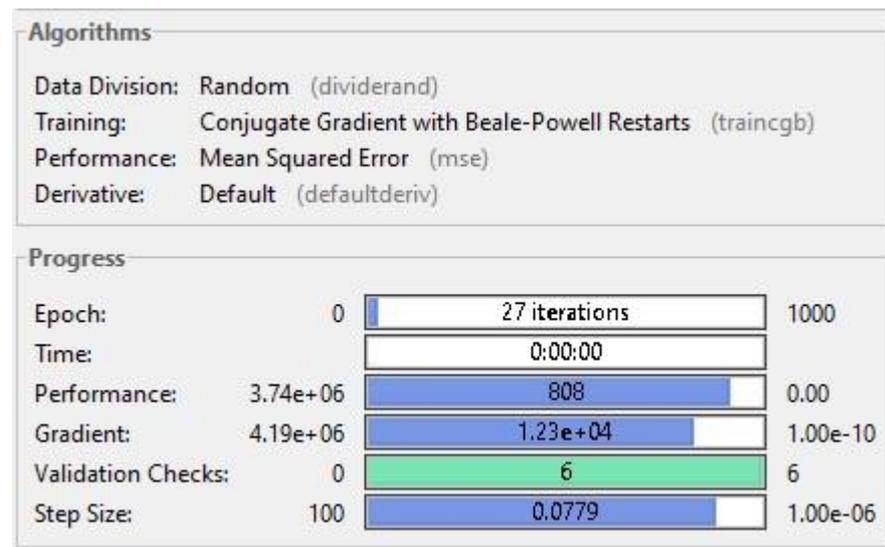
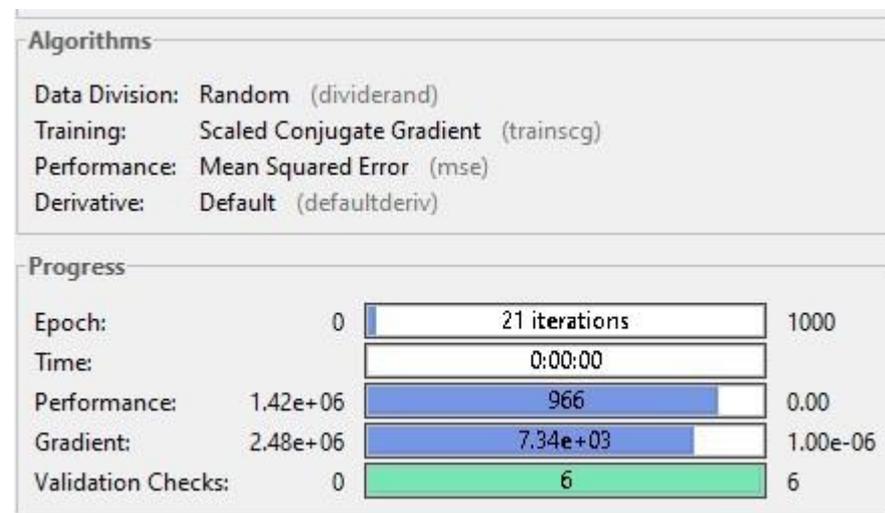
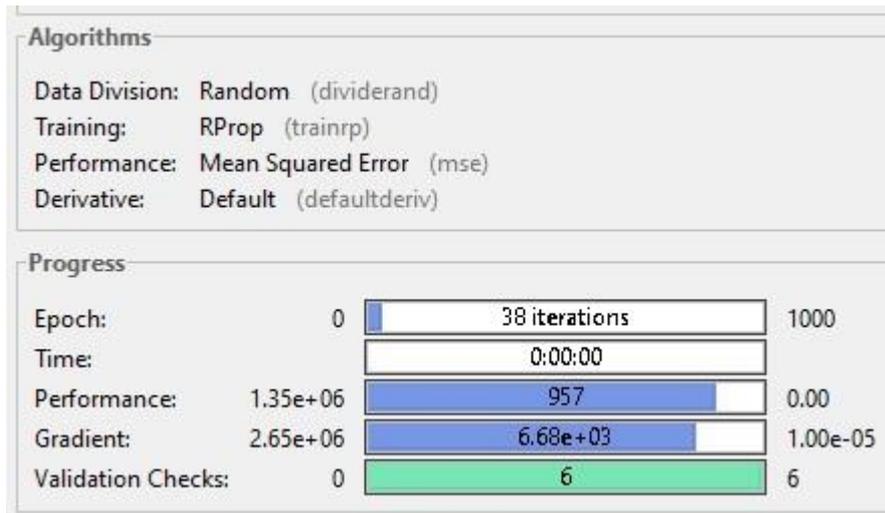
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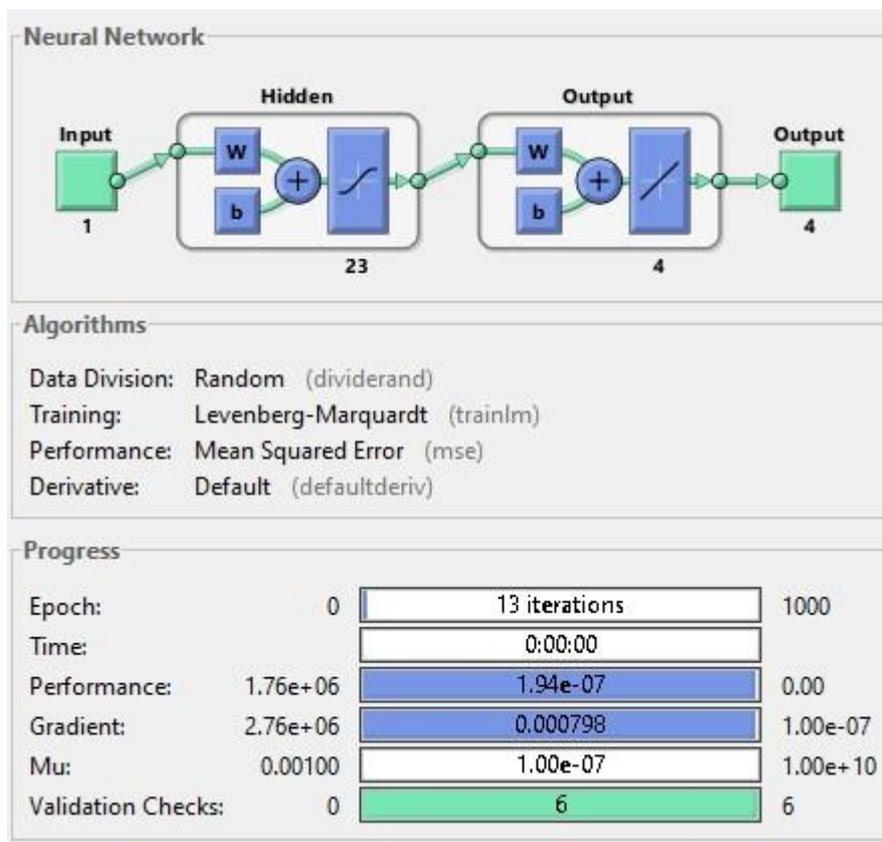
## APPENDICES

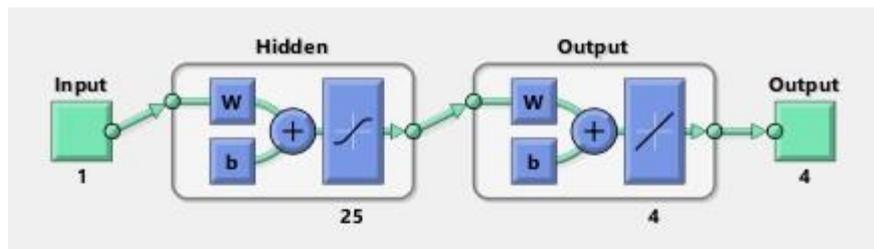
### Appendix A: Detailed results for each training algorithm





## Appendix B: Detailed results for different neuron numbers



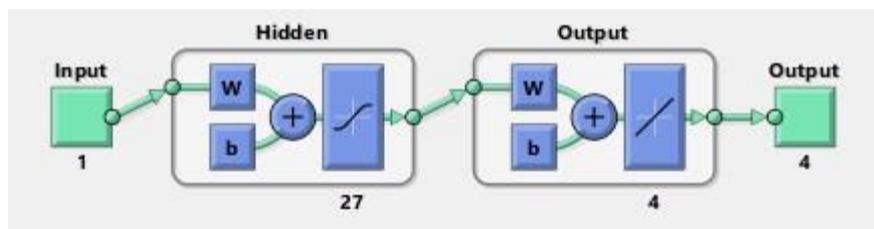


#### Algorithms

Data Division: Random (dividerand)  
 Training: Levenberg-Marquardt (trainlm)  
 Performance: Mean Squared Error (mse)  
 Derivative: Default (defaultderiv)

#### Progress

Epoch:	0	1000 iterations	1000
Time:		0:00:29	
Performance:	1.68e+06	2.15e-08	0.00
Gradient:	2.45e+06	3.88e-06	1.00e-07
Mu:	0.00100	1.00e-06	1.00e+10
Validation Checks:	0	0	6

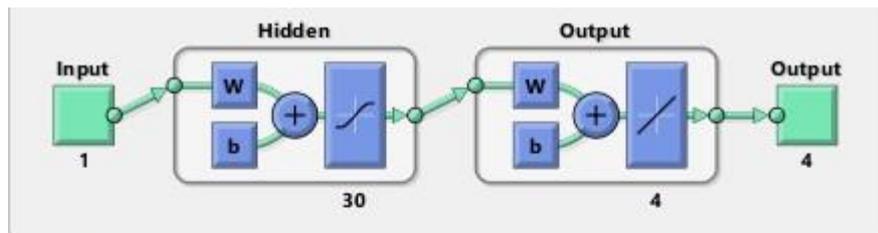


#### Algorithms

Data Division: Random (dividerand)  
 Training: Levenberg-Marquardt (trainlm)  
 Performance: Mean Squared Error (mse)  
 Derivative: Default (defaultderiv)

#### Progress

Epoch:	0	417 iterations	1000
Time:		0:00:12	
Performance:	2.37e+05	5.93e-09	0.00
Gradient:	4.36e+05	6.27e-06	1.00e-07
Mu:	0.00100	1.00e-07	1.00e+10
Validation Checks:	0	6	6

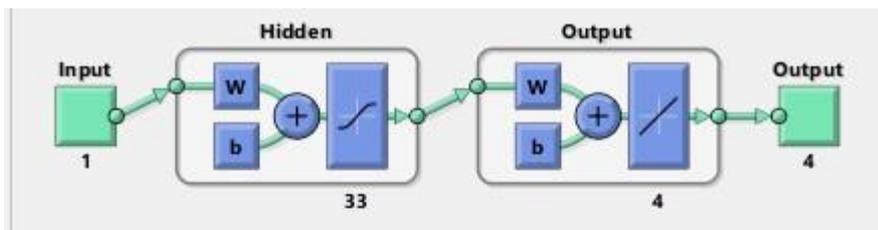


#### Algorithms

Data Division: Random (dividerand)  
 Training: Levenberg-Marquardt (trainlm)  
 Performance: Mean Squared Error (mse)  
 Derivative: Default (defaultderiv)

#### Progress

Epoch:	0	15 iterations	1000
Time:		0:00:00	
Performance:	9.08e+05	7.76e-08	0.00
Gradient:	2.05e+06	0.000257	1.00e-07
Mu:	0.00100	1.00e-05	1.00e+10
Validation Checks:	0	6	6



#### Algorithms

Data Division: Random (dividerand)  
 Training: Levenberg-Marquardt (trainlm)  
 Performance: Mean Squared Error (mse)  
 Derivative: Default (defaultderiv)

#### Progress

Epoch:	0	9 iterations	1000
Time:		0:00:00	
Performance:	7.16e+05	8.60e-06	0.00
Gradient:	1.68e+06	1.04	1.00e-07
Mu:	0.00100	1.00e-07	1.00e+10
Validation Checks:	0	6	6

Appendix C: Physical Properties of Therminol 66

Temperature (°C)	Viscosity (Mpa.s)	Density (kg/m <sup>3</sup> )	Thermal Conductivity (W/m.K)	Heat Capacity (kJ/kg.K)
50	17.64	988.6	0.116	1.665
60	11.53	981.9	0.116	1.699
70	8.06	975.2	0.115	1.733
80	5.93	968.5	0.115	1.768
90	4.55	961.8	0.114	1.803
100	3.6	955	0.114	1.837
110	2.92	948.2	0.113	1.873
120	2.42	941.4	0.112	1.908
130	2.05	934.5	0.111	1.943
140	1.75	927.6	0.111	1.978
150	1.52	920.6	0.11	2.014
160	1.34	913.6	0.109	2.05
170	1.18	906.6	0.108	2.086
180	1.06	899.5	0.107	2.122
190	0.95	892.3	0.107	2.158
200	0.86	885.1	0.106	2.195
210	0.78	877.8	0.105	2.231
220	0.72	870.4	0.104	2.268
230	0.66	863	0.103	2.305
240	0.61	855.5	0.102	2.342
250	0.57	847.9	0.1	2.379
260	0.53	840.3	0.099	2.417
270	0.49	832.5	0.098	2.455
280	0.46	824.6	0.097	2.492
290	0.44	816.6	0.096	2.531
300	0.41	808.5	0.095	2.569
310	0.39	800.3	0.093	2.608
320	0.37	792	0.092	2.647
330	0.35	783.5	0.091	2.686
340	0.34	774.8	0.089	2.726
350	0.32	765.9	0.0888	2.766
360	0.31	756.9	0.086	2.806
370	0.3	747.7	0.085	2.847
380	0.28	738.2	0.084	2.889

Physical properties of Therminol 72

Temperature (°C)	Viscosity (Mpa.s)	Density (kg/m <sup>3</sup> )	Thermal Conductivity (W/m.K)	Heat Capacity (kJ/kg.K)
60	3.5	1043	0.135	1.661
80	2.29	1025	0.132	1.715
100	1.61	1007	0.13	1.769
120	1.2	989	0.127	1.823
140	0.93	970	0.125	1.877
160	0.74	952	0.123	1.932
180	0.6	934	0.12	1.986
200	0.49	916	0.118	2.04
220	0.42	898	0.115	2.094
240	0.35	880	0.113	2.148
260	0.3	862	0.11	2.203
280	0.26	844	0.108	2.257
300	0.23	825	0.106	2.311
320	0.2	807	0.103	2.365

Properties of Jarytherm

Temperature (°C)	Viscosity (Mpa.s)	Density (kg/m <sup>3</sup> )	Thermal Conductivity (W/m.K)	Heat Capacity (kJ/kg.K)
60	0.54	827	0.12	1.95
80	0.45	809	0.115	2.02
100	0.38	791	0.111	2.09
120	0.33	772	0.107	2.16
140	0.28	754	0.102	2.23
160	0.25	735	0.098	2.31
180	0.22	717	0.094	2.39
200	0.2	699	0.089	2.46
220	0.18	680	0.085	2.54
240	0.17	662	0.081	2.63
260	0.15	644	0.076	2.71
280	0.14	625	0.072	2.79
300	0.13	607	0.067	2.88
320	0.12	588	0.063	2.96
340	0.11	570	0.058	3.05