Compilation and Validation of Heat Transfer Coefficients of Quenching Oils

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Diploma work No. 91/2012

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Summary

This reports scope is to map how the cooling ability for quenching oils is varying with parameters such as temperature, agitation, volume and condition of the oil. When hardening steel components in industry, oils are commonly used as quenchants. FEM-calculations predict the result after hardening and in these calculations it is of great importance how the cooling is described. Heat Transfer Coefficients are used as boundary conditions on components that shall be studied.

Four different oils from the industry have been investigated. Cooling curves have experimentally been compiled with equipment from Swerea IVF called *ivf SmartQuench*®. From the cooling curves Heat Transfer Coefficients can be calculated with the software *SQIntegra*, which is also from Swerea IVF. The cooling ability variation was then analyzed with regard to selected characteristics such as Heat Removal Capacity and Hardening Power. Two steels have been hardened with two of the oils investigated. This was to see if the experimentally measured and calculated results are according to each other.

The project has been following the methodology DMAIC, which is a part of the Six Sigma concept. It consists of five phases; Define, Measure, Analyze, Improve and Control. The method is often used on improvement projects in industry and other sectors.

Conclusions from the project were that the changeable parameters such as temperature, agitation, volume and condition of the oil are affecting the Heat Transfer Coefficient. Increased temperature increases the cooling ability of the oil and so do increasing agitation as well. The effect on increasing volume or changing condition of the oil showed a smaller or no general trend. The project showed that a new measure of cooling ability of oils can be used. This measurement is the Heat Removal Capacity which measures the derivate of the Heat Transfer Coefficient curve. A thorough Measurement System Analysis was performed which showed that the measurement system was operator dependent.

Keywords: Quenching, Quenchants, Quenching Oils,

Preface

This report presents a master thesis project performed at the department Material and Manufacturing Technology at Chalmers University of Technology. It is performed in collaboration with Swerea IVF AB. Swerea IVF is part of the Swedish Research Group Swerea AB. The master thesis was performed from January 2012 until June 2012.

Gothenburg June 2012

Cornelia and Augusta

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1 Introduction

Quenching is a heat treatment method that has been used for many thousands of years and is still of importance today. The process is performed to change the microstructure of metals and in turn their mechanical properties. The cooling rate must be fast enough so that a phase transformation occurs to a required microstructure. What is fast enough is dependent on which type of steel the component is composed of and how it is designed. A side effect of quenching is residual stresses that may cause distortions and cracks in the material. Therefore it is important to have knowledge and control over the quenching process.

To be able to predict the result in the microstructure of a material after quenching FEM-calculations is used. In these calculations it is of great importance how the cooling is described. Heat Transfer Coefficients are used as boundary conditions on components that shall be studied.

There exists a wide range of quenchants in the industry including water, vegetable and mineral oils, polymer solutions and brine. They all have different kinds of cooling ability and are used for different kinds of products. This thesis will be focusing on mineral oils and their cooling abilities.

1.1 Purpose

The purpose of the project is to analyze how the cooling ability of quenching oils is varying when the Temperature, Agitation, Condition and Volume of the oils is changed. The overall method of the project will be according to the DMAIC model of Six Sigma.

1.2 Limitations

The project will not evaluate the chemical compositions of the oils and how that effects the cooling. The software and how it performs calculations will not be investigated. The number of quenchants will be limited to four mineral oils. Agitation tests will only be performed on two oils and the hardening of steel samples will be performed in the same oils.

1.3 Thesis outline

The report will start with giving the theory concerning the project and then move on to the methods used. The result and analyze of the result will then be stated followed by a discussion, recommendations and conclusions.

2 Theory

Here the theory for the project will be stated.

2.1 Quenching

One way to improve metals mechanical properties can be to perform different heat treatments. It involves heating and cooling of a work piece and the aim is to increase strength, hardness and other mechanical properties. There are several different types of heat treatments like annealing, tempering, precipitation strengthening and quenching. In this report the focus will be on the last one, quenching.

When a work piece is rapidly cooled from its austenitizing temperature or solid solution treating temperature it is called quenching (G.E. Totten et al, 1991). By immersing the hot piece into a quenchant, it becomes cooled. This is to create a transformation in the microstructure. It will result in a certain hardness, strength and toughness. After a successful quenching the aim is minimize the risk of residual stresses or cracks.

One material that can be quenched is steel. The steel piece is heated to a temperature in to the austenite phase. After the microstructure has transformed into austenite, quenching can be performed. The aim when quenching steel is usually to obtain the microstructure martensite. A fast cooling suppresses the ferrite, pearlite and bainite phases and forms the martensite.

Depending on the characteristics of the metal work piece, different quenchants are used. Oil, water, water containing salt or aqueous polymer solutions are the most common quenchants (G.E. Totten et al, 1991). The focus in this report will be on mineral oil quenchants.

2.2 Cooling curves

To evaluate a quenchants performance, a so-called cooling curve can be used. Cooling curves can be obtained by heating a steel piece containing a thermocouple to an elevated temperature and then immersing it in to the quenchant of interest. The change in temperature versus time is recorded and plotted, see figure 1. From this curve the cooling rate is obtained by calculation. Both the cooling- and cooling rate curves are characteristic and unique for each quenchant (T.Holm et al, 2010).

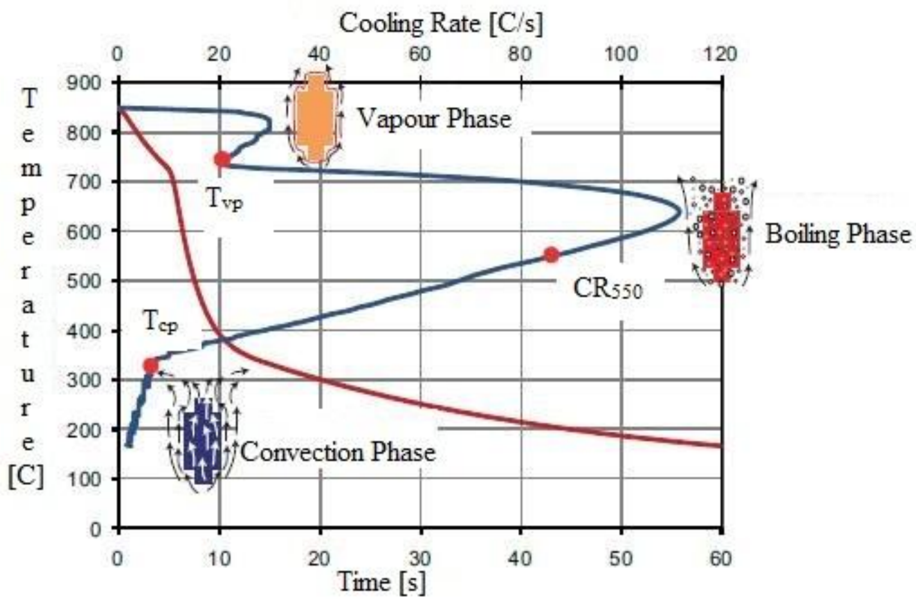

Figure 1 *Cooling curve and cooling rate curve including the three different phases and the characteristics values (T.Holm et al, 2010).*

Three different stages of heat removal are shown in figure 1. Stage A, also called the vapour phase is what happens right after the probe is immersed into the quenchant. The probe is so warm that the oil closest to it starts to vaporise, creating a vapour blanket that is isolating the probe from the rest of the oil. Heat transfer occurs mainly by radiation through the vapour blanket. The blanket works as a heat-isolating layer, which results in a slow cooling of the probe. How long this stage last depends on different properties of the quenchant.

After a while the supply of heat from the probe to the oil is not enough to exceed the amount of heat needed to vaporise the quenchant. Stage B is entered when the probe's temperature has decreased and the vapour blanket collapses, the temperature at which this happens is called T_{vp}. The oil close to the probe starts boiling and a flow in the quenchant is created. This increases the cooling rate and during this stage, also called the boiling phase, the highest cooling rate occurs.

When the temperature has decreased below the quenchant's boiling point the cooling is carried out through conduction and convection. Stage C, which is the last phase is called the convection phase. The temperature at which the boiling ends and stage C is entered is called T_{cp}. The cooling rate is low in this final stage, and depends mainly on the quenchants viscosity and flow (T. Holm et al, 2010).

2.2.1 Evaluation of Quenchants

It is important to be able to measure, evaluate and compare different quenchants. This is to make sure that the right quenchant giving the wanted phase transformation is being used. Different factors are affecting the quenchants characteristics. According to the ASM Handbook (G. E. Totten et al, 1991) the evaluation of quenchants are divided into two categories:

- Heat removal ability tests
- Hardening power tests, the metallurgical response

This means that one way to classify the characteristics of a quenchant is to relate it with the quenchants ability to remove away heat from the work piece. The other way is to relate it to the results given when hardening a metal work piece in the quenchant.

2.2.2 Heat Transfer Coefficient

Heat transfer coefficients are used for calculating the convection heat transfer when cooling a solid in a fluid. It is a measure on a materials ability to lead heat (T. Holm, 2010). Convection heat transfer is energy transfer due to movement of either random molecular motion or macroscopic motion of the fluid. This motion together with temperature gradients results in heat transfer.

The Heat Transfer Coefficient is depending on the heat flow, the surface area of the solid and the difference in temperature between the solid and the fluid. Equation 1 describes how the Heat Transfer Coefficient is calculated. From this equation the Heat Transfer Coefficients unit can be decided that is [W/m^{2}K] watts per square meter- Kelvin (F.P. Incropera et al., 2007).

$$\text{Heat Transfer Coefficient} = \frac{q}{A * \Delta T} \quad \text{(Equation 1)}$$

Where q is the heat flow input per second, A is the heat transfer surface area and ΔT is the difference in temperature between the solid and fluid. The Heat Transfer Coefficients are calculated from the cooling curve.

2.2.3 Hardening Power

The ability of a quenchant to harden steel is related to a number called the Hardening Power. This value takes some characteristic values from the cooling curve and evaluates the cooling ability of the quenchant. The Hardening Power for unalloyed steel when quenched in oil is according to the following equation (T. Holm et al, 2010):

$$\text{Hardening Power} = 91{,}5 + 1{,}34 * T_{vp} + 10{,}88 * CR_{550} - 3{,}85 * T_{cp} \qquad \text{(Equation 2)}$$

Where T_{vp} is the transition temperature between the vapour and boiling phase, CR_{550} is the cooling rate at 550°C and T_{cp} is the transition temperature between the boiling and convection phase. This means the Hardening Power relate the cooling ability of a quenchant with values from the cooling rate curve, see figure 1 where T_{vp}, CR_{550} and T_{cp} can be read. Equation 2 is experimentally based on measurements according to the ISO standard 9950 for oils. Oils with high Hardening Power give a faster quenching and increase the hardness of the work piece when hardening it (Swerea IVF AB, 2002).

2.2.4 Factors Affecting Quenching Results

How well a quenchant is able to harden a specific work piece depends on different factors. For example the cooling characteristics the quenchant have, what type of quenchant it is and for how long it has been used. The viscosity, wetting characteristics, contamination and concentration of additives the quenchant have. All these factors are related to the quenching media but some external factors can also affect the quenching result:

- Temperature, agitation and volume of the quenchant
- The effective Heat Transfer Coefficient between the metal piece and the quenchant
- Design of quenching tank
- Thickness, geometry, mass and surface area of the metal piece being quenched
- Oxidation on the surface of the metal piece

This makes it important to evaluate the design and properties of the product when choosing the quenchant and quenching method to get the wanted result and to have a sustainable system.

2.3 Quenching Oils

Oils are used in quenching to control the heat transfer from the metal piece that is going to be hardened. This reduces the thermal gradients that can lead to distortion and cracking in the metal piece. Quenching oils can be based on either mineral oils or vegetable oils. The most common ones are based on mineral oils (Chandler, 1995) and consist of petroleum and additives to improve its properties such as wetting ability and cooling (G. E. Totten et al, 1991). They are equivalent to other petroleum oils such as engine oils and industrial lubricants.

Vegetable oils are used as well since mineral oil has become more regulated in how they should be used. Improper disposal of mineral oils may cause environmental damage, for example if they leak out into the ground water. Vegetable oils are based on oil that is naturally occurring for example canola oil and soybean oil.

The three major classifications of quenching oils are Conventional also known as Cold oils, Accelerated oils and Marquenching oils also known as Hot oils (G.E Totten et al, 1993). There exists other classifications but these are most common. Conventional oils are mineral oils that contain additives to prevent oxidation and degradation. Their usage temperature goes up to 65 ˚C. Accelerated oils have usage temperatures up to 120 ˚C and are mineral oils that contain more additives to increase the cooling rate. Marquenching oils are refined mineral oils with some additives to enhance the oxidizing and thermal stability. They are used between 95 to 230 ˚C.

There is some advantage of using oil instead of water, brine etcetera as quenchant. Oil has a higher boiling point and the boiling phase is entered earlier. The cooling is increased in the beginning and the risk of entering the pearlite or bainite noses are decreased. The cooling rate during the convection phase depends on the viscosity of the quenchant. A higher viscosity results in a lower cooling rate.

Since oil has a relatively high viscosity compared to other quenchants it has a slow cooling rate in the convection phase. The boiling phase is entered early, which allows the convection phase to start just around the martensitic transformation temperature. The convection phase is as mentioned a slow cooling stage and this allows the stresses to be released and the risk for cracking decreases in the material. When water is used as quenchant the boiling phase is still occurring when the martensite start temperature is reached. The cooling in this stage is still so high that the thermal stresses is locked in the material causing distortion and cracks. In order to minimize distortions and cracks in the work piece oil is preferred as quenchant.

2.4 Steel

The steels grades that will be hardened are 100Cr6 and 16MnCr5. Below the two steels characteristics are shortly described.

2.4.1 100Cr6

This steel is used as rolling bearing steel and is composed of 1% Carbon, 0.25% Silicon, 0.35% Manganese and 1% Chromium (SKF Steel, 1984).

2.4.2 16MnCr5

16MNCr5 is case-hardening steel commonly used in transmission components. It is composed of 0.15% Carbon, 0.25% Chromium, 0.9 % Manganese, 0.8% Chromium and 1% Nickel (SKF Steel, 1984).

2.5 Equipment used when Measuring Cooling Curves

The cooling curves were compiled by performing practical measurements. The equipment used will now be described.

2.5.1 *Ivf SmartQuench*®

Some of the equipment used for producing cooling curves of the quenchants is equipment from Swerea IVF called *ivf SmartQuench*®. The equipment is designed according to ISO 9950 that specifies how the cooling characteristics of quenching oils should be measured. Using a nickel-alloy probe and non-agitated oil could rank the different oils characteristics in a standardized way (International Organization for Standardization, 2011). A system overview of *ivf SmartQuench*® is displayed in figure 2.

Figure 2 *Ivf SmartQuench® (Swerea IVF AB, 2012).*

Ivf SmartQuench® components:

- Hand-unit connected to the test probe
- *Ivf SmartQuench®* software *SQIntegra*
- 1 litre beaker with holder for the test probe
- Standard test probe according to ISO 9950
- Furnace for heating the test probe

The hand-unit is a microcomputer that records and stores measured data. This is also where all the settings are chosen. This is settings like the Start Temperature which is where the recording starts and it is generally set to 850 °C. When automatic settings are used the temperature first has to reach three degrees above the Start Temperature and then go below it for the recording to start. The sampling rate is the number of recordings per second. The hand-unit can communicate and transfer data to the *ivf SmartQuench®* software that is installed on a computer.

According to the ISO 9950 standard, the beaker should hold two litres of oil. Measurements at Swerea IVF and other laboratories show that having a one-litre beaker instead of two does not change the results in any significant way (Swerea IVF AB et al. , 2007). So for practical reasons, a one-litre beaker is used when performing measurements with *ivf SmartQuench®*.

The test probe used is according to the international standard ISO 9950 and the American standards ASTM D 6200-01 and ASTM D 6482-06 (Swerea IVF AB et al. 2007). The probe consists of two parts both made of Inconel 600. The lower end is a solid part, called the test probe body. Inside there is a thermocouple placed in the centre. The upper part of the probe is a hollow tube supporting and protecting the thermocouple.

The furnace is used for heating the probe. When the furnace is heated from cold, the temperature will first exceed the operating temperature and then stabilize at 870 °C after a while. At this chosen temperature the test probe will be heated up relatively quickly.

2.5.2 Additional Equipment

Additional equipment, not included in *ivf SmartQuench®* standard system that is going to be used:

- 10 litre quench tank with holder for the test probe
- Stirring device consisting of a propeller driven by an electrical motor. Can be used with the 10 litres quench tank
- Equipment for measuring the flow rate in the oil

2.6 Vickers Hardness Test

The Vickers hardness test measures the hardness of a material. It consists of a diamond shaped indenter that is pressed into the material with a set kg amount for about 10 to 15 seconds. It can be used on most metals and have a wide range of scales. The hardness has the unit Vickers Pyramid Number (HV) or Diamond Pyramid Number (DPH). They are defined as the load divided by the area of the indentation (Smallman et al, 2007), see Equation 3.

$$HV = Constant * (Indent\ Force/\ Indent\ Area) \qquad \text{(Equation 3)}$$

When performing Vickers hardness test it is essential that the sample surface is clean and smooth. It is important to make sure that the indents are not to close causing them to affect each other. The distance between two indents should be three*diagonal of one indent or more (T. Holm, 2010). The distance between the edge of the sample and the centre of an indent should be at least 2.5*diagonal.

2.7 Software

The software used during the project is SQIntegra and Minitab 16. Below will a description of each program be stated.

2.7.1 *SQIntegra*

SQIntegra is used to calculate Heat transfer Coefficients and is part of the ivf Smartquench® equipment. It is a program that uses assembled cooling curves to calculate Heat Transfer Coefficients and predict properties of a quenched piece (Swerea IVF AB, 2007). To calculate Heat Transfer Coefficients the software is solving inverse heat conduction equations. It is an equation that is solved based on the observed effects of it and not as in the opposite case a problem where the effects are determined. The problem is to get the Heat Transfer Coefficients of temperature curve from the assembled cooling curve temperature of time. Temperature of time is the effect of Heat Transfer Coefficients of temperature and this is therefore an inverse heat conduction problem.

To solve the problem the software needs the following parameters (Swerea IVF AB, 2007):

- A cooling curve, measured by a probe
- Location of the thermocouple inside the probe
- Thermal conductivity and specific heat of the probe
- Density of the probe
- Temperature of the quenchant that’s been used
- An initial guess function

The Heat Transfer Coefficients that are calculated in the software are the characteristics of the oil. The input data is the data recorded by the probes thermocouple, which is located inside the probe. They need to be recalculated so that they are for the surface of the probe. That is done by Fourier partial differential equations (Swerea IVF AB, 2007). It calculates the temperature distribution allowing the program to get the temperature at the surface of the probe. From all of this the Heat Transfer Coefficient of the oil is calculated.

The initial guess function of time steps is set by the operator and is changed for each individual curve. The resulting curve is depending on the initial guess and changes if the guess changes. This means that the curve needs to be carefully worked on to get the optimal solution of the Heat Transfer Coefficients curve. It is important to have more time steps at the places on the curve where it is a dramatic change and fewer where the curve is not changing in the same extent. An example of the dependence of the time steps can be seen in figure 3. The Heat Transfer Coefficient curves are calculated from the same cooling curve measured on one litre of QuenchWay 125 B at 130 °C. The two different curves have different time steps.

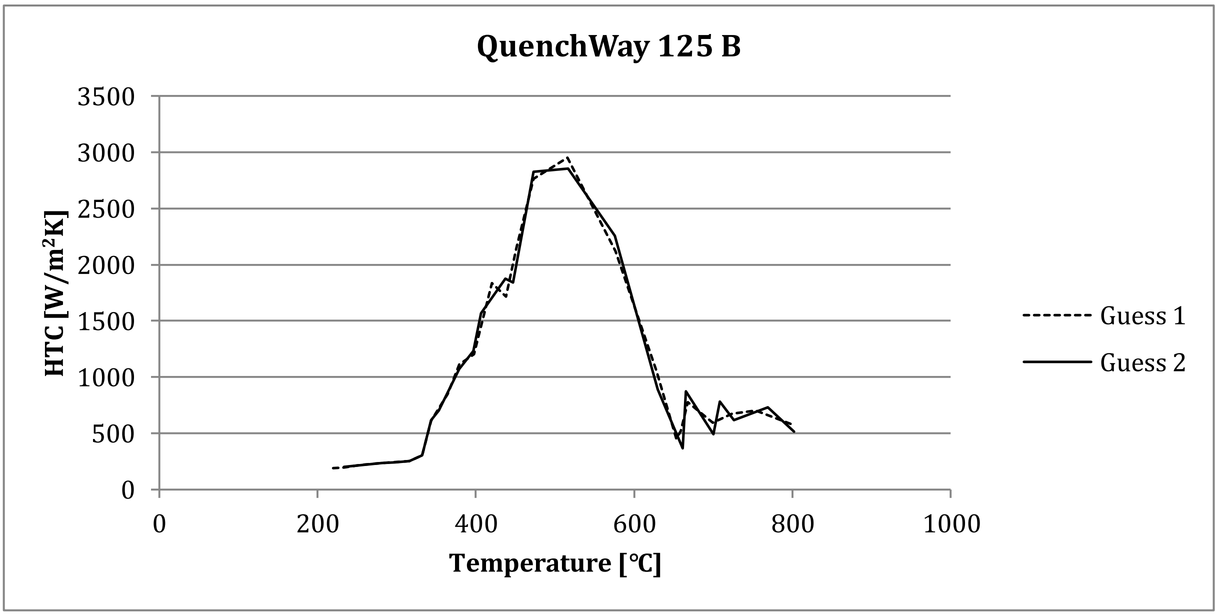

Figure 3 *Heat Transfer Coefficients curve for QuenchWay 125 B at 130°C showing the dependence on the selected time steps.*

The Software also calculates the cooling rate curves characteristic values T_{vp}, T_{cp} and CR_{550}. From them the Hardening Power in Equation 1 is calculated. The values are calculated by third degree integrals to find the temperatures were the phases starts and ends.

2.7.2 Minitab

Minitab 16 is software developed by Pennsylvania State University that handles statistical problems. The program is used in Lean Six Sigma and other statistically based methods. It is used to analyse and organize data with for example regression analysis, correlation and variance analysis (Minitab Inc., 2012). Other tools that could be used with the software are General Linear Model, Main Effect plot, Interaction Plot, Fitted Line plot, Correlation and Gage R&R.

The General Linear Model is a multiple regression analysis that defines the relationship between the inputs and the output. It uses regression analysis with scatter plots and correlations equations to predict future performances from the current data. The variables that impact the output in a significant way are identified. It calculates a model and assumes that the data can be fitted into a linear relationship and that the data that is outside of it is residuals or uncontrollable variation. The response from Minitab when doing a general linear model is four graphs. The first graph is a Normal Probability Plot and shows if the data is normally distributed. To be normally distributed the data should be on the line. The Versus Fits Plot shows the predicted value on the x axis and the residual for from the data on the y axis. The two other two graphs are the histogram of the residuals and the observation order of the data against the residuals of the data.

A Main Effects Plot plots the means of the selected data against one another and shows how they affect each other. The Interactions Plot plots the means of one level of selected data whereas the other level is held constant. This shows how the data are interacting with each other.

The Fitted Line Plot is a tool that performs a linear regression on the selected data and plots them against one another. A regression line is plotted trough the data that shows the relationship between the variables. It shows the 95% control interval and the 95 % prediction interval. The control interval lies inside the prediction interval and shows the interval which 95% of the data is inside. The prediction interval is the interval which new measurement data will with a 95% certainty fall inside.

The Correlation calculates the Pearson product moment between the selected variables. It measures the linear relationship. The number is stated between + and - 1. If the number is positive it means that when one variable increases the other does too. When the number is negative the variables go the opposite way. The Gage R&R can be read about in Section 2.9.

2.8 Six Sigma

During the project the methodology DMAIC of Six Sigma has been followed. Six Sigma is often used on improvement projects in industry and other sectors. DMAIC consists of five phases; Define, Measure, Analyze, Improve and Control. Here are the five phases described more in depth.

2.8.1 Define Phase

The purpose of the define phase is to get everyone in the project on the same page when it comes to the problem to be solved, scope, goals and performance targets. The focus is on the voice of the customer and project planning. To do this a set of different tools such as SIPOC, AIM and a Gantt-schedule are used, see Appendix A for more information about the tools.

The Define Phase starts with finding a project that is suitable for the Six Sigma methodology. A suitable project is a project that has a problem within a process with a potential to improve. The potential could for example be to improve cost savings, process performance, technical complexity and organizational complexity (George et al. 2005). When the project is found a team should be put together. The team members should possess knowledge about the process and/or knowledge about the Six Sigma method. A Gantt-schedule is made for setting a time line for the project. Then the expected outcome from the project is identified. Hence analysing the voice of the customer, developing the big Y. The big Y is the factor that should be improved in the project.

2.8.2 Measure Phase

Measure is the second step in DMAIC. In the Measure Phase the current process needs to be understood, how it works and how well. Some tools that are used during this phase are Fishbone Diagram and Process Map see Appendix A. By gathering data the current situation can be described. This phase includes some numerical studies and data analysis. The collection of data means gathering of small y:s and x:es. Small y:s are the variables that the improvements of the big Y can be measured on and x is the variable that is put in to the process (George et al. 2005). Focus is to find adequate data that helps describe the problem. The team must make sure the collection process is valid and that the correct data is gathered.

The output from the process usually has some variation. It can be depending on variation in the process but sometimes also the measurement system can influence the output. The amount of variation induced from the measurement system can’t be too big. A validation of the measurement system needs to be performed to make sure that it is stable. By performing a so-called Measurement System Analysis also called a Gage R&R this can be done. It is also helpful to early identify the baseline, in the report referred to as hypothesis, of the current process. How is the process working today and what do we know about it? It makes it easier to evaluate the result after the project is performed to recognize potential improvements.

2.8.3 Analyze Phase

In this phase a statistical analysis of the problem is performed. The data collected earlier in the Measure Phase are now being analysed. One goal is to find additional factors that are affecting Y to the ones known from before, stated in the hypothesis. It is important to find how Y is varying with the x's.

With the help of statistical tools the Cause-and-Effect relationship could be identified. When trying to find relationships between variables a theory called the Null Hypothesis is used. It can either "fail to reject" or "fail" a theory. The theory stated is usually that there is no relationship between an x and a small y. The result displayed in Minitab is shown both graphically and numerically. A number called the p-value for probability shows if the theory fails or is failed to reject. The p-value depends on alpha that is usually set to 5%. Alpha is called the level of significant and is the maximum acceptable risk of being wrong when rejecting the Null Hypothesis (Minitab, Inc. 2009). If the p-value is under the alpha's 5% the theory fails and it exists a relationship between the x and small y. If the p-value is above 5% the theory is failed to reject. It means that the theory that there is no relationship can't be rejected and that it might be true. In the end of the Analyze Phase the factors that have the most significant effect on Y should be identified. All this should give the project team directions for improvements in the next phase.

2.8.4 Improve Phase

Here the aim is to come up with potential solutions to improve the performance of the process based on the results from the Analyze Phase. After generating potential improvement ideas they should be evaluated. Then the best solution when it comes to benefits, costs or other variables of interest should be selected and optimized. A tool to use when finding the best solutions is Design of Experiments. It identifies the factors having the biggest impact and reduces the time and amount of experiments that needs to be performed. After that, one improvement idea should be selected, optimized after that implemented.

2.8.5 Control Phase

After the improvements in the earlier phase have been implemented the outcome has to be controlled and monitored. This is to guarantee the improvement worked and to see how it affected the outcome.
In this last phase the aim is to complete the project work and hand over the improved process with all needed information to the process owner. A comparison between before and after the implementation should be performed. Recommendations for further actions and other possibilities should be given to the process owner to make the handover as smooth as possible.

2.9 Measurement System Analysis

Measurement System Analysis is a method used to evaluate the variation in a measurement system. When measuring a process the output may have variations. The goal of the Measurement System Analysis is to find out how much of the total variation is caused by the measurement system. The variation of the measurement system has to be so low that variations in the process can be seen.

The variation in a measurement can be part-to-part or measurement system variation. To find out how much of the total variation either of them contributes to, a Gage R&R study can be made. Gage R&R stands for Gage's system Repeatability and Reproducibility. The measurement system variation can be divided into Repeatability and Reproducibility. Repeatability is the variation due to the measurement device. It can be measure by letting one operator measure the same part several times with the same measurement device and then look at the variance between the measurements. Reproducibility is the variation due to the measurement procedure. It can be measured by letting several operators measure the same part with the same measurement device and then look at the variance between the measurements.

The measurement system errors can be classified by accuracy and precision. Accuracy consists of bias, linearity and stability. Bias is the difference between the measured value and a reference value. Linearity is the difference in the bias value over how many parts that have been measured and stability is how the bias value is changed when measured on different times. Precision is Reproducibility and Repeatability. The Gage R&R study tests all of these different aspects. The measurements are calculated and plotted so the measurement system variation can be seen compared to the part-to-part variation. The variation contributed by the measurement system should be less than 30% (George et al., 2005). If it is the opposite way the measurement system needs to be corrected.

To do the Gage R&R study the software Minitab could be used. The measurements are stated in a table with measurement value, operator and part. It is important that all parameters and measurements are randomized. The Gage R&R study is made on these values with a method called ANOVA, meaning analysis of variance. The results can be seen in a collection of diagrams seen in figure 4.

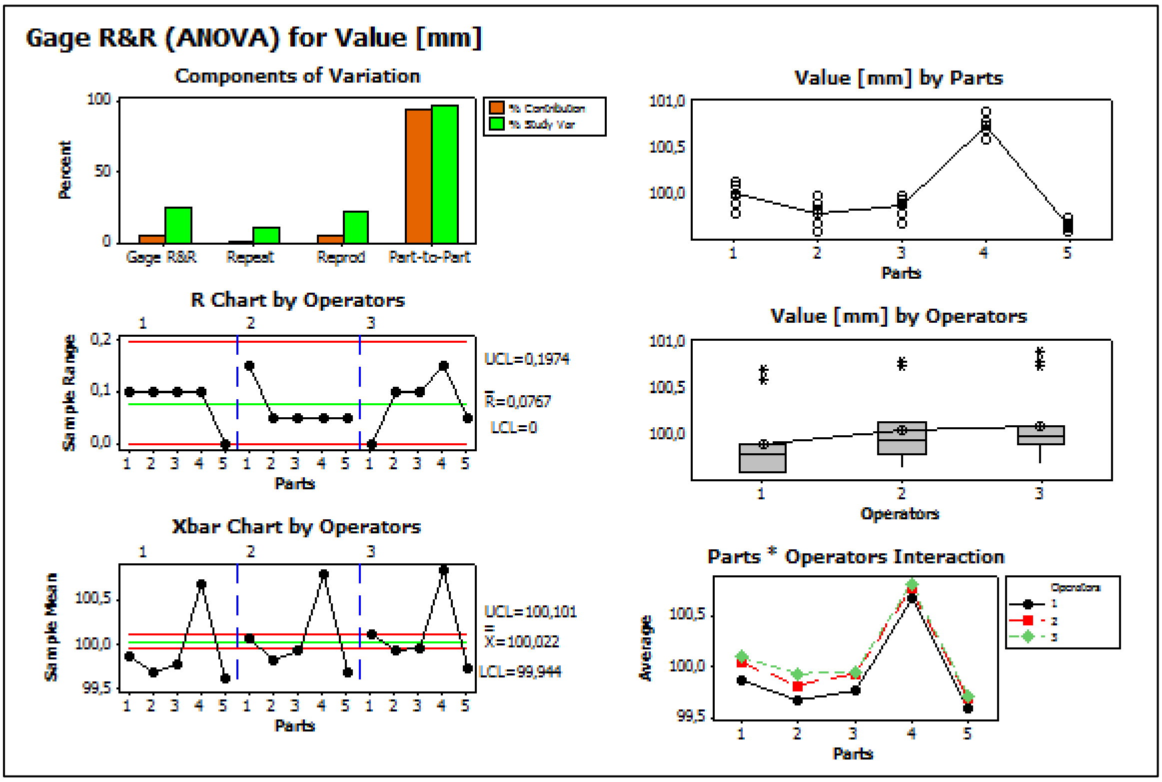

Figure 4 *An example of a Measurement System Analysis.*

In figure 4 three operators are measuring one value on five different parts two times each. The first graph shows the Components of Variation. It shows the amount of variation each of them contributes with.

The R Chart can be seen in the middle to the left in figure 4. It is a control chart showing the operators consistency. Each plotted point is the difference between the largest and lowest measured value for that part by each operator. The centre line is an average based on all the operators and parts averages. The upper and lower limits are based on variations in subgroups. When all points are inside the upper and lower limits a good measurement system is presented. If not, the operators have problem keeping consistency in their measurements.

The Xbar Chart in figure 4 shows the part-to-part difference for each operator. The plotted points show the average measurement for each part. The centre line is the average measurement of all operators for all parts and the upper and lower limits are based on number of measurements and the Repeatability estimate. A good measurement system has a lot of parts located above or below the limits showing a larger part-to-part variation than measurement system variation.

The Value by Parts graph shown in the upper right corner in figure 4 is stating all the measurements performed on each part, represented by empty circles. The bold circles are the measured averages and the lines are drawn between the parts averages. The empty circles should preferably be close together and the averages should vary so that the parts difference can be seen.

The difference between the operators can be seen in the Value by Operators graph in figure 4. It shows all the measurements for each operator and the average measurement for the operators. The line shows how the averages differ between the operators. A line that is parallel to the x-axis indicates that the system is good since all the operators are on average getting the same value.

The last graph in figure 4 show the Parts*Operator Interaction. It displays the operators' average measurements for each part. The lines are then drawn between these points to show the variation. The perfect case would be that the measured averages on each part for the operators would be equal and there would be a variation between the parts that is clear to see.

2.10 Hypothesis

When hardening steel it is important to not only choose the right quenchant but also to use it properly. The cooling has to be fast so the steel gets hard enough, but not so fast that it causes unwanted distortion or cracks. One property that is characterizing a quenchant is its flexibility. The flexibility is a measure on how the oils cooling ability is varying when different parameters are changed. It is important to understand how different quenching parameters are affecting the outcome from the quenching procedure to get control over the mechanical properties, distortions and risk of cracking. Some parameters that can be changed are temperature, agitation, condition and volume of the oil. The hypothesis is based on current literature and evaluates how the stated parameters affect the cooling of the quenchant.

2.10.1 Temperature

Different oil types should be used at different temperatures. They have recommended using temperatures that should give a specified cooling effect. According to Bates, Totten and Brennnan (1991) the temperature of the oil does not affect the cooling in a large extent if only a moderate increase in the temperature is performed. Although a raise in temperature decreases the viscosity of the oil that gives a faster cooling. If the increase is inside the recommended oil temperature intervals the viscosity change will not affect the cooling to any large rate (Herring, 2011). A rise in temperature will also increase the temperature at which the maximum cooling rate occurs. The hypothesis for the effect of temperature is that for increased temperature within the selected temperatures the cooling ability of the oils will increase.

If the temperature of the quenchant is continuing to increase the time the vapour phase exist starts to increase. This means a longer vapour phase, which is a slow cooling stage. A large increase in temperature also results in a decrease in cooling rate in the boiling and convection phase (G.E Totten et al, 1991).

2.10.2 Agitation

Agitation refers to the movement of the quenchant relative to the work piece that is still. Agitation increases the cooling of all phases when used (G.E Totten et al, 1991). The agitation forces the hot oil at the surface of the probe to constantly be replaced by oil not yet heated by the probe. The vapor blanket is removed at an earlier stage when agitation is used and the boiling phase is entered faster (G.E Totten et al, 1991). This means that a phase with faster cooling, the boiling phase, is replacing a slower phase, the vapor phase resulting in an increased cooling rate. The hypothesis for the effect of agitation is that for increased agitation the cooling ability of the oils will increase.

2.10.3 Condition

The condition of the oil is divided into new and used oil. Used oil is oil that has been used in production. Its properties are dependent on how long and in which environment the oil has been used. With time, the characteristics of the quenchant are changing due to thermal and mechanical degradation. The oil gets contaminated from usage in production by soot from the furnace atmosphere, water from the cooling system and oxidation when getting in contact with air. Fine soot particles can in small amounts increase the cooling by acting as nucleation points for the oil to start to boil from. The opposite effect occurs when the particle amount is increased above a critical value so that the cooling instead is decreased (D. S. MacKenzie et al, 2002). The particles help to increase the oxidation and change the heat transfer of the oil that decreases the cooling rate.

Quenching oils often contain additives to increase the cooling rate or to prevent oxidation. When using the oil in production for hardening metal workpieces there is always some drag-out of oil with the pieces. This drag-out could be so-called selective drag-out where the additives are the ones following the piece out of the quenching system (G.E Totten et al, 1993). This causes degrading because the oil loses its additives leading to increased viscosity of the oil which decreases the cooling ability (G.E Totten, et al, 1993). The hypothesis for the change in condition is that the cooling ability will also change.

2.10.4 Volume

The volume of an oil bath when quenching is dependent on the weight and temperature of the steel that shall be quenched (G.E Totten et al, 1991). For one type of oil and product one litre may be enough to get a required quenching and then an increase in volume won’t change the results. For another oil and product one litre may be too small and then an increase in volume will give a dramatic improvement to the quenching. The hypothesis for changing the volume from one to ten litres the cooling ability of the oil will increase.

3 Method

The general method of the project was DMAIC of Six Sigma, more about DMAIC can be read in Section 2.8. After the Define Phase and the problem were characterized the oils were collected from different manufacturing companies in Sweden. Before the measurements were performed a Measurement System Analysis on the *ivf SmartQuench*® equipment was performed to ensure the measurement system. After that the oils cooling curves were measured and compiled when the different setting of the selected parameters were varying. Parallel to the measurements the resulting cooling curves were calculated into cooling rate curves and Heat Transfer Coefficients curves. When all data was collected it were compiled and analyzed in Mintab 16. Steel was also hardened in the oils and the result was analyzed by performing Vickers Hardness Tests. A more detailed description of the methods is stated below.

3.1 Collection of Oils

The investigated quenching oils were gathered from different companies, see table 1. Type of oil, specified using temperature according to the manufacturer of the oil and the companies using temperature is displayed in the table. The names of the companies have been excluded in the report. The types of oils mentioned in the table could be read more about in Section 2.3 where the Step-quenching oil is similar to the Marquenching oil described and the Fast quenching oil is an Accelerated oil.

From all companies both new and used oil of their oil type was collected. In production the volume of oil continuously decreases due to drag-out. Drag out is the excess of oil that adheres to the surface of a metal workpiece when it is taken out from the furnace. The companies add new oil to the furnaces when needed. This means that the entire quench tank is never changed at the same time. This makes it impossible to tell for how long the oil has been used. The volume of oil used in production varies between 4 000 and 20 000 litres per furnace and company. All companies have agitation in the oil when the hardening is performed.

Table 1 *General information about the investigated oils (T. Holm, 2010). (Klen Quench 140 data from Southern Lubricants Inc., 2008. Quenching Oils).*

Oil	Type of Oil	Company	Specified Temperature	Company Temperature
QuenchWay 125 B	Step-quenching oil	A	120 – 200°C	70, 90, 130°C
		B		60, 90, 120°C
Klen Quench 140	Cold quenching	C	< 100°C	75, 95°C
Isorapid 277 HM	Fast quenching	B	50 – 130°C	60, 90, 120°C
		D		60, 80, 100°C
Durixol W72	Fast quenching	E	50 – 130°C	60, 80, 100°C
Belini FNT	Fast quenching	F	< 100°C	

3.2 Measurement System Analysis

Three Measurement System Analyses were performed to ensure that the measurement system was acceptable. It has to be good enough to measure the difference between different oils. It also has to be stable enough so when different operators are performing the same measurements, the results will be similar.

The cooling curves used in the first Measurement System Analysis were compiled from using QuenchWay 125 B and Bellini FNT at 70°C. Two oils were used so the part-to-part difference could be measured. This value depends on how similar the two chosen oils are, and can vary a lot. If the oils are similar, the result from the Measurement System Analysis will be less good, than if two very different oils are used. With *SQIntegra* the Heat Transfer Coefficients curves were compiled and from them the Heat Removal Capacity was calculated, read more about this characteristic under Section3.5. These values were used as the measurement data when the Measurement System Analysis was executed. The two operators performed the same measurements four times each in a random order. A total of 16 cooling curves were compiled.

Also, the measurement system has to be precise enough to detect any changes in Heat Transfer Coefficients when parameters, like the temperature of the oil are changed. This is a much smaller part-to-part difference compared to the one used in the previous Measurement System Analysis. To make sure the measurement system was valid enough for this a Measurement System Analysis was executed on QuenchWay 125 B. Nine different cooling curves were compiled by using the *ivf SmartQuench*® equipment. The measurements were performed at three different temperatures; 70°C, 90°C and 130°C by two operators. The results from that one showed that the measurements system was not valid enough so a third Measurement System Analysis was performed. This time only one operator was performing the measurements. This is not according to the standard when performing a Measurement System Analysis. Conclusions were drawn that the influence of different operators wasn't needed to be included in the evaluation. This is since only one operator is going to perform all the measurements for the same oil.

3.3 Cooling Curve Compilation

Here the general method when compiling cooling curves with the *ivf SmartQuench*® equipment will be stated together with some specific exceptions used in this project.

3.3.1 Test Procedure when Measuring Cooling Curves

The cooling curves were compiled with the equipment *ivf SmartQuench*®, more information in Section 2.5. It starts with heating up the furnace to the wanted temperature and when the temperature has stabilized the test probe can be placed in the ceramic tube for heating. The temperature at where the recording starts was set to 850°C.

At the same time the beaker containing the oil of interest was heated on a hotplate to the wanted temperature. The temperature of the furnace and oil are alternating and needs to be synchronized. When they do and the wanted temperatures are reached, the quenching can be performed. The test probe, when heated to 855°C is quickly removed from the furnaces and immersed into the beaker with oil. The reason for heating the probe higher than 853°C is to get the recording to start when the probe is immersed in the oil and not before. When a 10 litre quench tank is used a stirring device can be added to create flow in the oil. The thermocouple records the temperature versus time. After 60 seconds, the recording stops.

It is important that the test probe body becomes located in the centre of the oil sample. If this is performed carelessly a flame around the probe at the surface can be formed. This is undesirable since it creates an uneven heat transfer and a risk of fire. The probe and oil cools down after a while. Before the probe can be used again it needs to be grinded. This is performed to remove all the oxide formations created during heating and quenching.

3.3.2 Specifics for the Project

From the performed Measurement System Analysis it was found that the measurement system was stable for measurements performed by the same operator. Because of this the same operator did all the measurements on one oil. The measurements were not performed in a random order because of the time it took to change the temperature of the oil after a measurement was performed.

A 1 litre beaker and a 10 litre quench tank were used. When the 10 litre quench tank was used, a stirring device was added to measure the oils at different agitations and to see how the volume of the oil affected the results. All oils have different using temperatures. The oils were measured for two or three of these temperatures. The agitation was set to be at 0.0, 0.2, 0.35 and 0.5 m/s. For each change of parameters two measurements were performed to ensure valid results.

3.4 Calculating of Heat Transfer Coefficient

To calculate the Heat Transfer Coefficients the software *SQIntegra* was used. The compiled cooling curves are loaded into the computer from the hand unit. The probe material and dimensions are set from either a database containing the ISO 9950 standard probe or manually. The quenchants temperature needs to be set and the initial guess can be stated. The initial guess can be selected either from a database or stated manually. When all parameters are set the inverse calculating can start. It needs to be performed about two to three times to get a stable value. The resulting curve needs to be optimized; by adding or removing time steps in the initial guess this is done. The Heat Transfer Coefficients curve should have it characteristic features and be smooth.

3.5 Definition of characteristic values

The characteristic values are the ones that the variation in Heat Transfer Coefficients will be measured on. From the Six Sigma method they are called the small y:s. One of the characteristic values used in the project is calculated from the cooling curve and one is from the Heat Transfer Coefficient curve.

3.5.1 Heat Removal Capacity

The integral of the Heat Transfer Coefficients curve is called the Heat Removal Capacity of the oil and has the unit $\frac{\mathrm{W}}{\mathrm{m}^2}$. This value has not been used earlier according to the literature study for determine the variance of the Heat Transfer Coefficients.

3.5.2 Hardening Power

In Section 2.2.3 the Hardening Power is mentioned to be a measurement of the oil's ability to harden steel. In the Hardening Power equation T_{vp}, T_{cp} and CR_{550} are the characteristic values from the cooling curve. These values and the Hardening Power value are chosen as small y:s. The software *SQIntegra* calculates these values from the cooling curves. T_{vp} and T_{cp} are chosen by a third degree integral on the cooling rate curve. From the same curve CR_{550} is taken at 550°C.

3.6 Statistical Analysis

The analysis of the data were performed in the software Mintab, see Section 2.7. It started with gathering all measurement data in worksheets and arranging them according to type of oil, company, temperature, agitation, condition and volume. The tools used to analyse were General Linear Model, Main Effect Plots, Interaction plots and Fitted Line Plot. The tools can be read more abut in Section 2.7.

The different sets of arranged data were analysed with the selected tools. This was to see how the in-parameters temperature, agitation, oil, condition and volume are affecting the selected response variables Heat Removal Capacity and Hardening Power.

In the General Linear Model a response variable was selected, either Total Heat Removal Capacity or the Hardening Power. For the selected data that was going to be analysed the parameters that were varying were selected to be in the model, for example temperature, agitation, condition and volume. For each analysis a Residual plot, Main Effect Plot and Interaction Plot was shown.

When doing a General Linear Model all the data and their parameters are evaluated. If some of the parameters were found to be not significant, having a p-value over 0.05, they were removed from the model. This was performed until all the selected parameters are significant to get an optimal model. The General Linear Model adapts the selected data according to the parameters as good as it can, if some of the parameters are not significant the model tries to involve them as well which results in an bad optimization. An example of this can be seen in the Analysis Section 4.

3.7 Hardening of steel

Two different steels were hardened, 16MnCr5 and 100Cr6, see Section 2.4. 16MnCr5 was quenched in new and used QuenchWay 125 B from Company A and 100Cr6 was quenched in new and used Klen Quench 140 from Company C. Three different sized test bars were quenched all with a length of 100 mm and different diameters of 20, 30 and 40 mm. Two bars of each size were tested, giving a total of six bars per steel.

Depending on size and steel of the test bar the time and temperature in the furnace was decided. For 16MnCr5 the furnace had a temperature to 880 ˚C and for 100Cr6 it had a furnace temperature of 860 ˚C (SKF Steel, 1984). The probes with 20 and 30 mm diameters were heated for two hours and the bars with 40 mm diameter for two and a half hours. Inside the furnace the bars were put in a closed container that had an inlet for gas. The gas was used to minimize oxidation of the test bars. The gas used was Mison H2.

After the heating the bars were immersed in to the 10 litres quenching tank for 20 minutes. During all hardening tests an agitation of 0.2 m/s were used. QuenchWay 125 B had a temperature of 130 ˚C and Klen Quench had a temperature of 75˚C.

3.8 Hardness testing

On the hardened test bars hardness measurements were performed. Here the method for preparing the test bars for the hardness measurements are described and the method for performing the Vickers Hardness Tests.

3.8.1 Preparations

To analyse the hardness of the hardened steel some preparations needed to be performed. From the steel probes a steel piece was cut out from the middle of the probe. It is important to be careful not to burn the steel piece when cutting it. This will affect the steel properties because of the additional heating it will cause.

The cut out pieces were moulded into Bakelite to get them into the same sizes, make the handling easier and for protecting the edges. The moulding is performed in machines where the steel piece is put together with Bakelite powder. For about twenty minutes the steel piece and Bakelite was heated under pressure to form a solid mould around the piece.

The steel piece surface was scratched from the cutting and from the moulding some Bakelite was in some places covering it. So the surface needed to be grinded to get accurate hardness values. The grinding was performed both manually and automatically. From this the roughness of the grinding paper was decreasing for each step until the surface of the steel was smooth and almost mirror like. In the automatic machine some additional steps with polishing was performed to get a completely smooth and mirror like surface.

3.8.2 Measurements

The Vickers Hardness Tests were performed according to standard, described in Section 2.6. Two profiles were measured on each sample starting at the edge and going to the centre. The load applied was 1 kg. The hardness close to the surface was the most important so the indents were laid closer to each other in this area. The number of indents performed per profile was between 11 and 13. From the two profiles an average was calculated.

4 Results

Here the results from the performed Measurement System Analysis and measurements performed with *ivf SmartQuench*® equipment are presented. Also the results from the Vickers Hardness Tests performed on the hardened steel will be presented.

4.1 Measurement System Analysis

Three Measurement System Analyses were performed in the project. The first one was performed on measurements on Heat Removal Capacity from two different oils at the same temperature. Two operators performed three measurements each on the two oils in a random order. The hypothesis was that there was no difference on the results when two operators were doing the measurements. The resulting Gage R&R can be seen in figure 5.

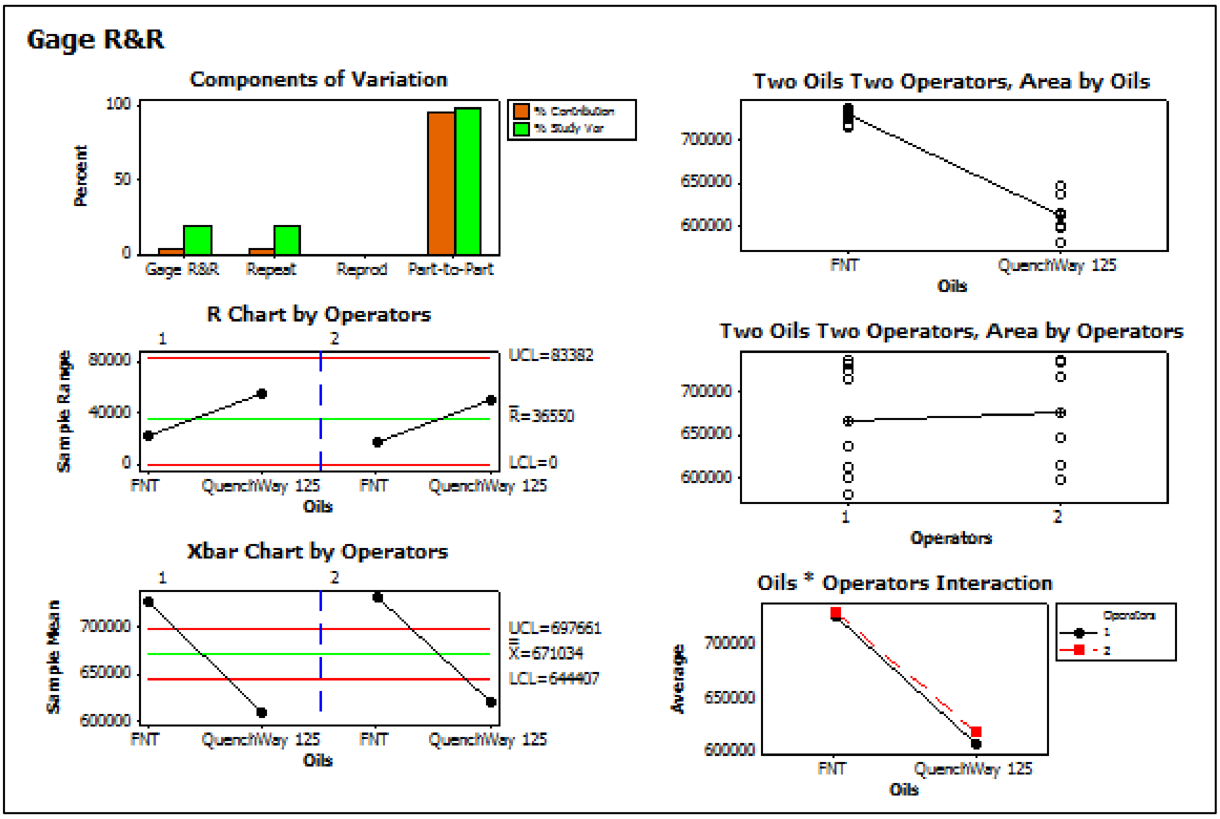

Figure 5 *First Gage R&R showing the results from measurements on Heat Removal Capacity performed by two operators on two oils.*

The graphs show that the measurement system is valid because the part-to-part variation is substantially larger than the Repeatability and Reproducibility variation is. The hypothesis is confirmed. This means that values from different operators and oils can be compared against each other. But in the project the same oil will be compared against itself when only different parameters are changed. This means that a second Measurement System Analysis needs to be performed to se if the measurement system is valid then as well.

The second Measurement System Analysis were performed on measurements from two different operators measuring the Heat Removal Capacity one oil at two different temperatures, see figure 6. The hypothesis was that there is no difference on the results when two operators were performing the measurements. Each operator made three measurements on each temperature. First of all a difference could be seen in the cooling curves performed by the two operators.

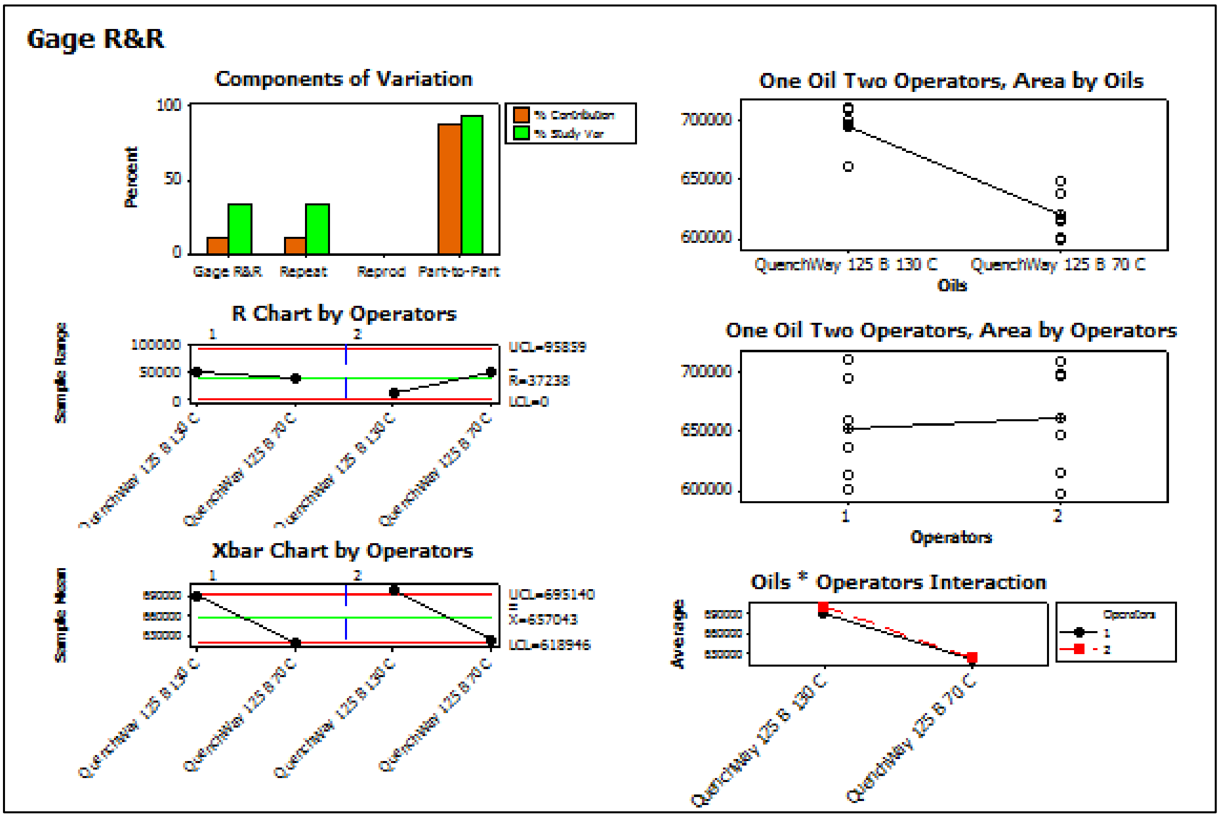

Figure 6 *Second Gage R&R showing the results from measurements on Heat Removal Capacity performed by two operators on one oil.*

When different operators performed the same measurement the resulting cooling curves did not look the same, the hypothesis was rejected. But all the cooling curves from one operator performing the same measurements were consistent and similar. The Gage R&R showed that the part-to-part variation was less than ten times the Gage R&R, repeatability and reproducibility variation. This means that the measurement system is not valid for comparing measurements on the same oil compiled by different operators with different settings. The part-to-part variation is then too small to be noticed. Because of this a third Measurement System Analysis was performed.

The last Measurement System Analysis was performed on measurements on Heat Removal Capacity from one operator measuring one oil at the three different temperatures 70, 90 and 130°C. The hypothesis was that there was no difference on the results when one operator was performing the measurements. On each temperature three measurements were performed. The resulting Gage R&R can be seen in figure 7.

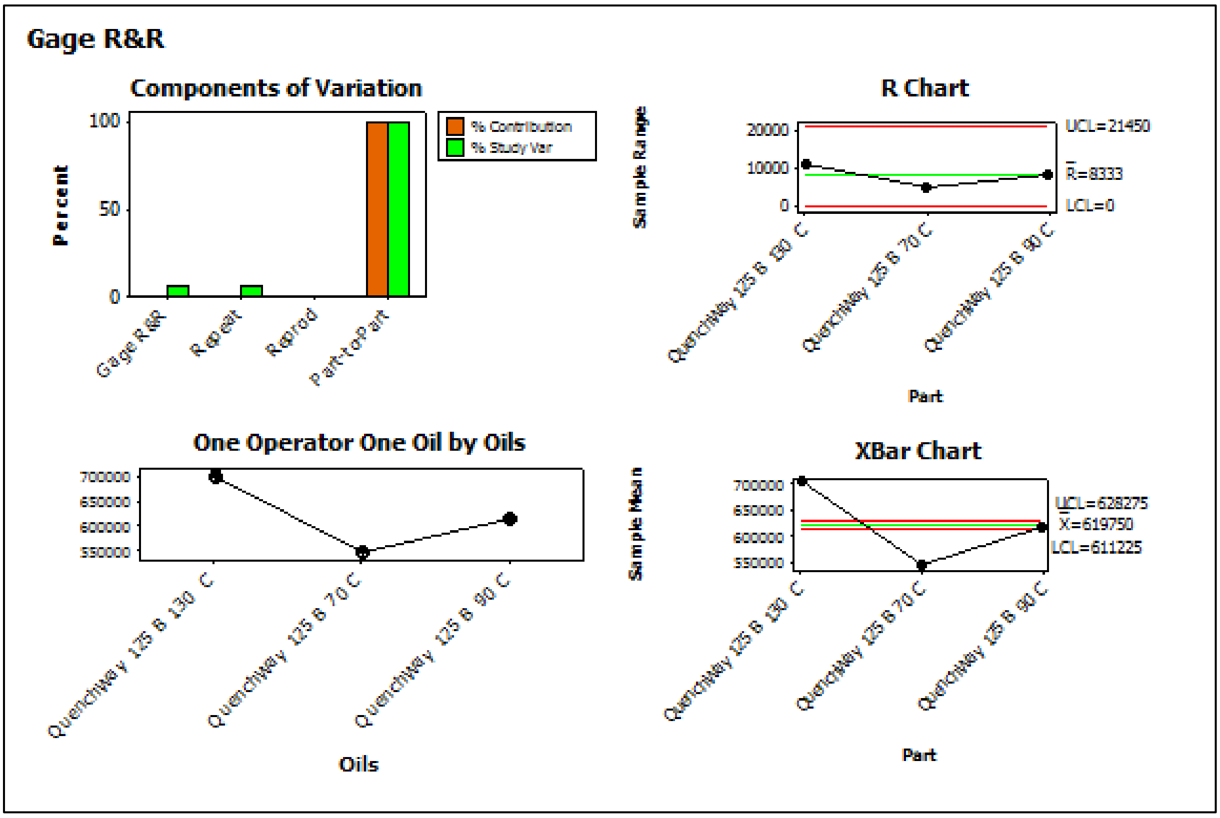

Figure 7 *Third Gage R&R showing the results from measurements on Heat Removal Capacity performed by one operators on one oil.*

The part-to-part variation is much larger than the other variations indicating that the measurement system is valid when one operator is performing the measurements. The hypothesis is confirmed.

4.2 Compilation of Cooling Curves

Presented in this section are the results from the measured cooling curves performed with *ivf SmartQuench®* equipment and the software's calculation of Heat Transfer Coefficients. The results are divided between the parameters temperature, agitation, condition, volume, and oil type. A full collection of all graphs and Heat Transfer Coefficient curves from the measurements performed during this thesis can be seen in Appendix B. First presented is a short summary of the results.

4.2.1 Summary

Both the cooling curve and the cooling rate curve for all oils are shifted when the different parameters are changed. The general trend for an increase in temperature is that the cooling rate curve is shifted to the right in the graph giving a general increase in cooling rate and cooling rate maximum, CR_{max}. T_{vp} is moved to the left, making that the vapour phase to collapse earlier. The point T_{cp} is also moved to the left, making the convection phase to start earlier. The result is that the boiling phase is occurring earlier at higher temperatures. Looking at the corresponding Heat Transfer Coefficient curves, the increase in temperature results in the curves being shifted upwards. This means that integral of the curve, the Heat Removal Capacity, is increasing. Although for Durixol W72 the trend for CR_{max} is the opposite, shifted to the left.

When agitation is added the curves are changing in a similar way as when the temperature is increased. The cooling rate curve is shifted to the right. This gives an increase in cooling rate and CR_{max}. T_{vp} is moved to the left making the boiling phase to start earlier. The difference between new and used oil gives no general trend that can be seen as representative for all oils. When changing the volume from one to ten litres the general trend is that the cooling is increased.

There is a big variety between the different oils measured. This results in unique cooling and cooling rate curves for all of them. Below are figures and descriptions of how the curves are affected by the different parameters.

4.2.2 Temperature

The temperature of the oils has been changed when performing measurements on the cooling curves. The temperatures are according to the temperatures the companies' are using. In figure 8 the change in cooling for 1 litre new QuenchWay 125 B from Company A over a range of temperatures can be seen. The cooling rate is increasing for increasing temperature and the phases of the cooling curve is changed in their sizes. The characteristic values of the hardening power equation are moved.

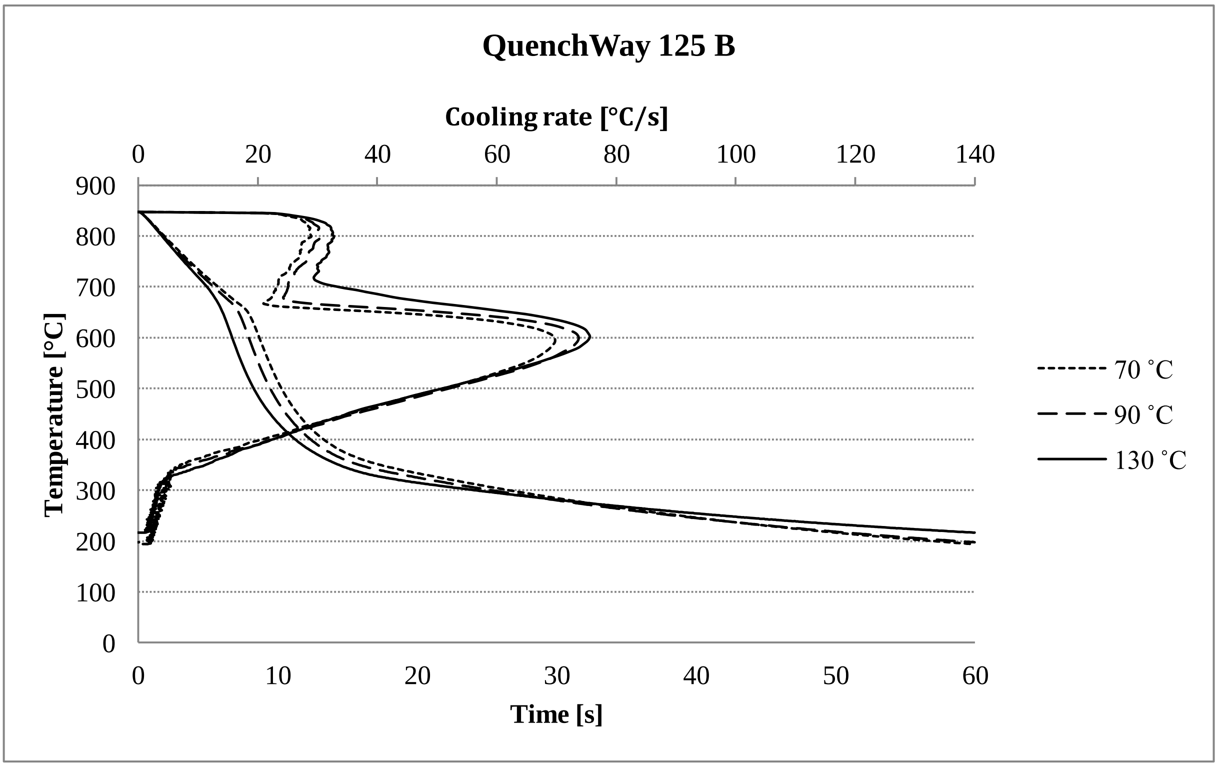

Figure 8 *Cooling and cooling rate curve depending on temperature for 1 litre new QuenchWay 125 B from Company A without agitation.*

How the Heat Transfer Coefficients for QuenchWay 125 B are depending on temperature can be seen in figure 9. The Heat Transfer Coefficient curves are increasing in height with increasing temperature giving an increased integral of the curves.

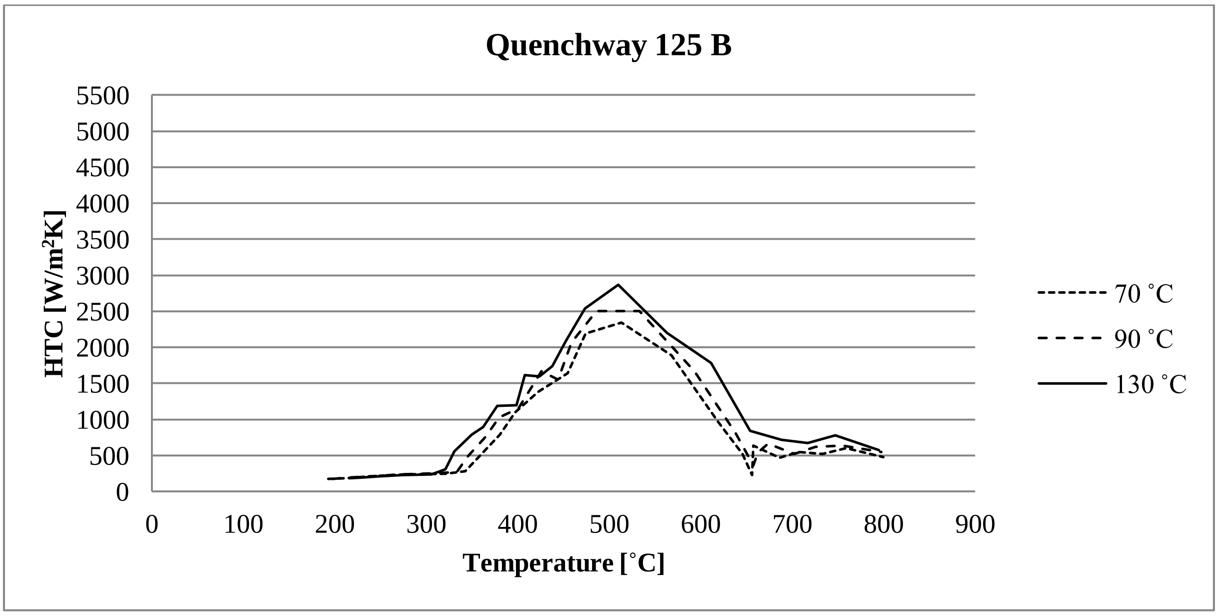

Figure 9 *Heat Transfer Coefficient curve depending on temperature for 1 litre new QuenchWay 125 B from Company A without agitation.*

The temperature dependence for 1 litre Durixol W72 can be seen in figure 10. The differences between the different temperatures are small but the trend is the opposite compared to QuenchWay 125 B's. CR_{max} is decreasing for increasing temperature. The Heat Transfer Coefficient curve in figure 11 shows how the coefficients are changing with the temperature. The trend is small but the curves are increasing with increasing temperature.

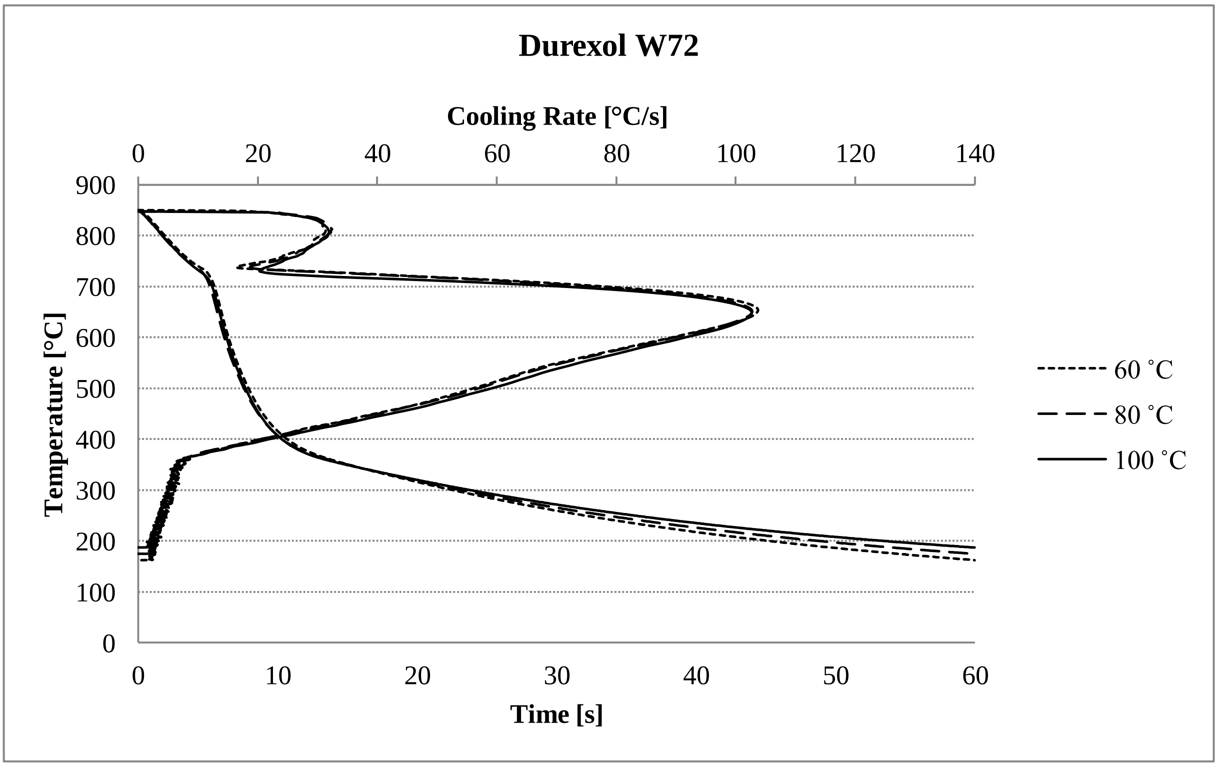

Figure 10 *Cooling and cooling rate curve depending on temperature for 1 litre new Durixol W72 from Company E without agitation.*

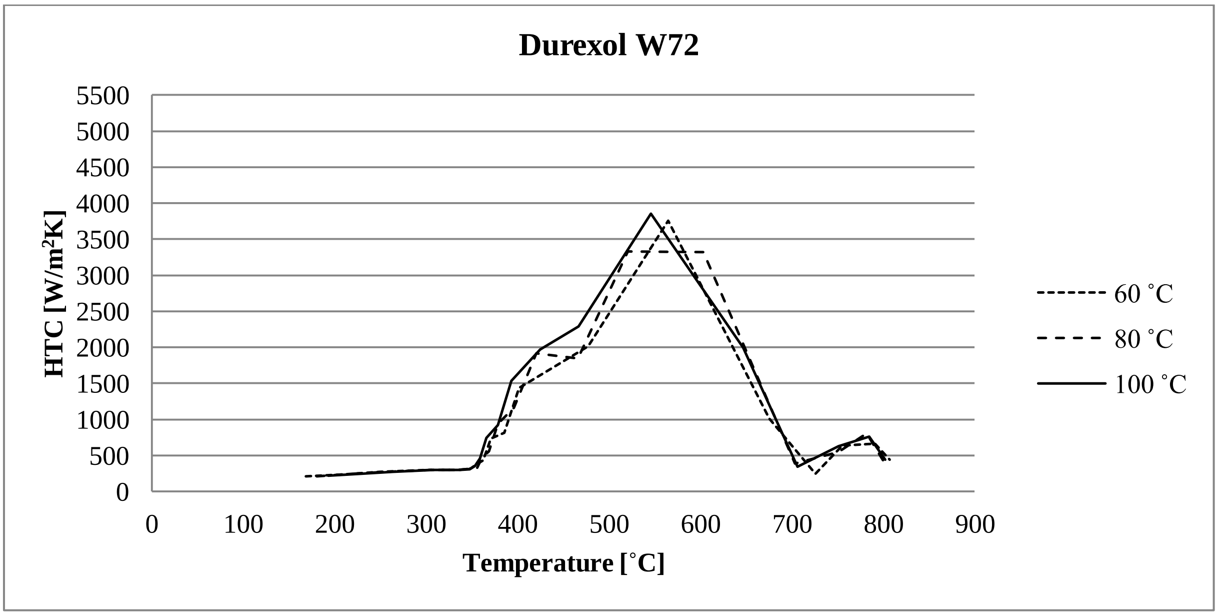

Figure 11 *Heat Transfer Coefficient curve depending on temperature for 1 litre new Durixol W72 from Company E without agitation.*

4.2.3 Agitation

Measurements with agitation were performed on QuenchWay 125 B and Klen Quench 140 in a 10 litres quench tank. The result from the first one can be seen in figure 12 and 13. Figure 12 shows how the cooling and cooling rate curves are depending on agitation. Figure 13 is showing the resulting Heat Transfer Coefficient curve. The graphs show a trend when agitation is added that is representative for Klen Quench 140 as well. The cooling rates are increasing with increasing temperature.

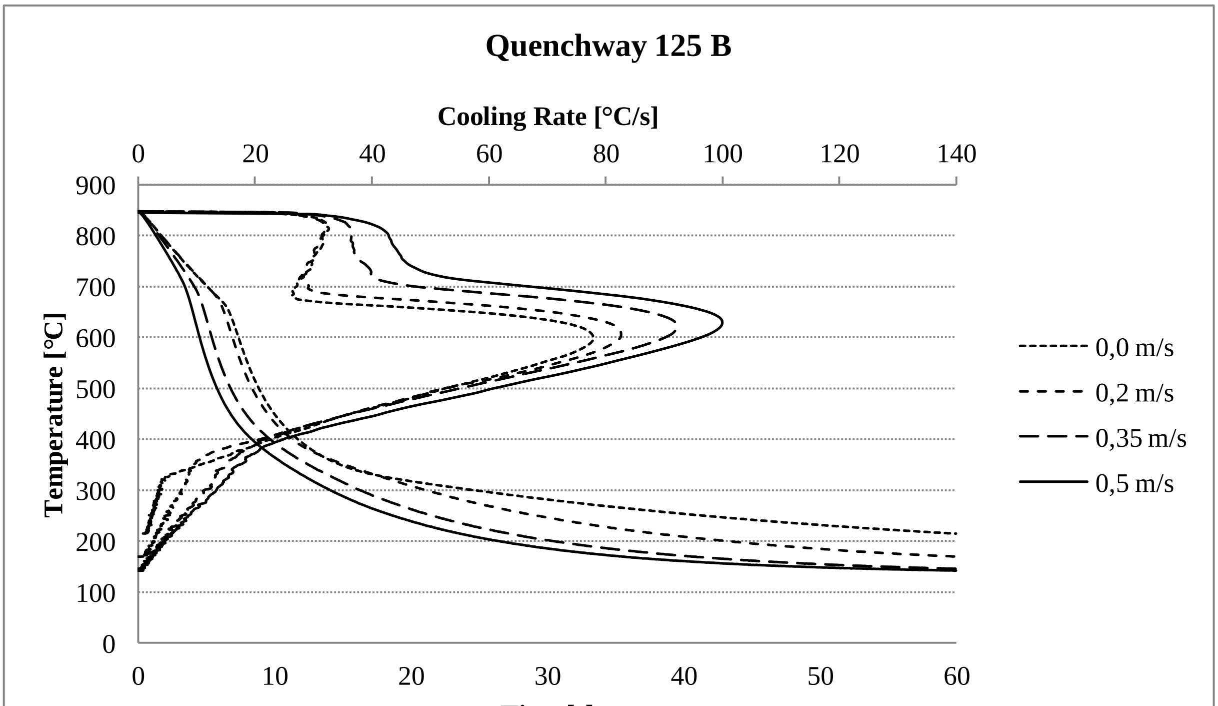

Figure 12 *Cooling and cooling rate curve depending on agitation for 10 litres new QuenchWay 125 B from Company A at 130°C.*

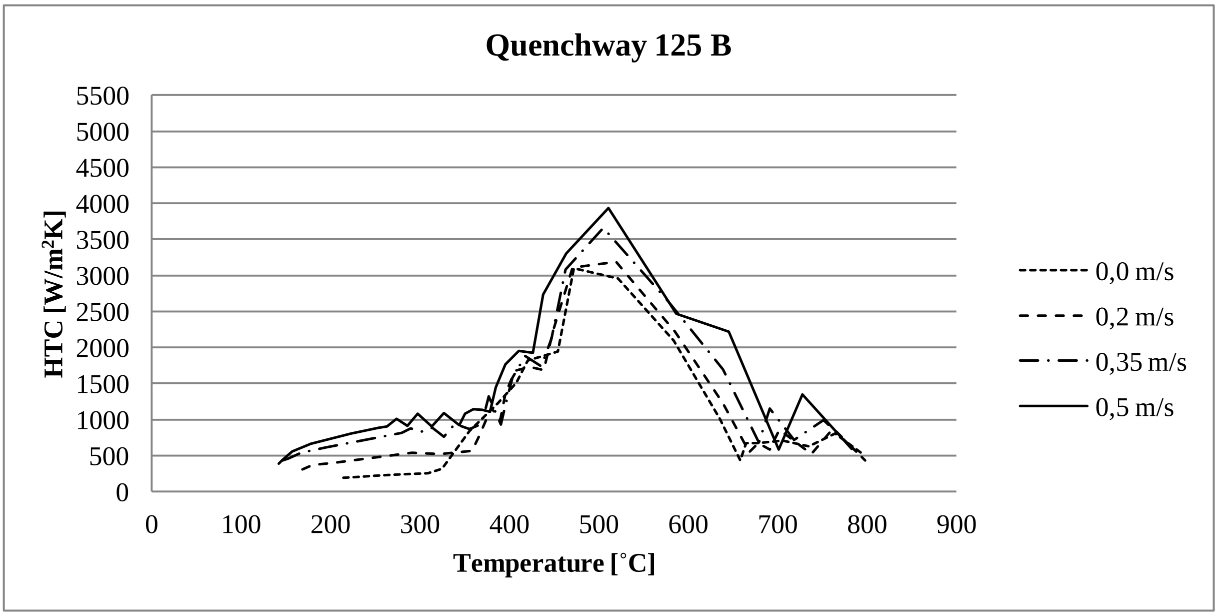

Figure 13 *Heat Transfer Coefficient curve depending on temperature for 10 litres new QuenchWay 125 B from Company A at 130°C.*

4.2.4 Condition

How the cooling for 1 litre QuenchWay 125 B is depending on Condition is shown in figures 14-17. In figure 14 and 15 the oil is from Company A and in figure 16 and 17 the oil is from Company B. The two oils from the different companies show dissimilar trends. In figure 14 with oils from Company A the new oil has a lower cooling rate than the used oil, which also can be seen in figure 15. The opposite can be seen in figure 16 and 17 where new oil has a higher cooling rate than the used oil.

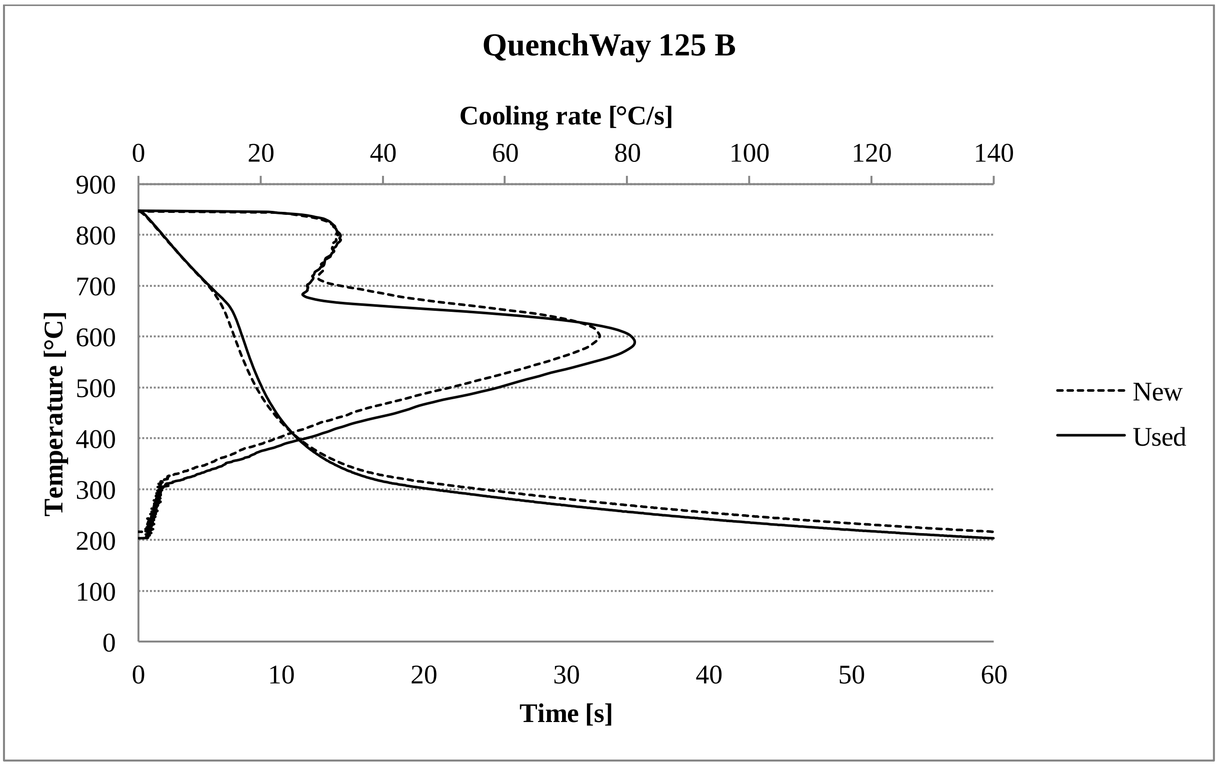

Figure 14 Cooling and cooling rate curve depending on condition for 1 litre QuenchWay 125 B from Company A at 130°C without agitation.

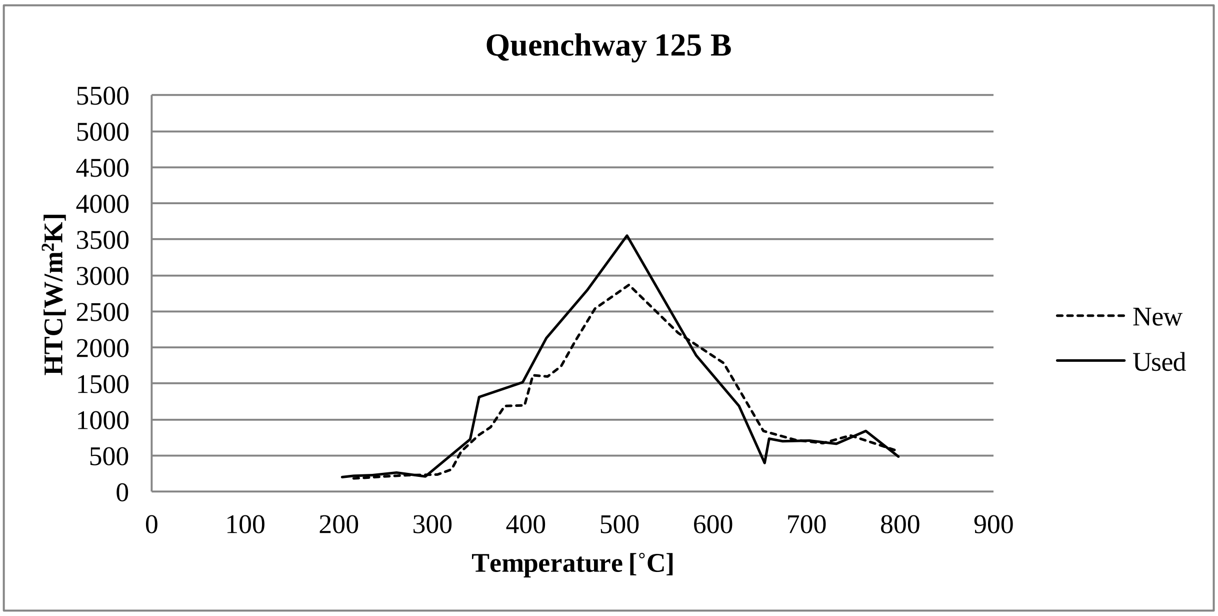

Figure 15 *Heat Transfer Coefficient curve depending on condition for 1 litre QuenchWay 125 B from Company A at 130°C without agitation.*

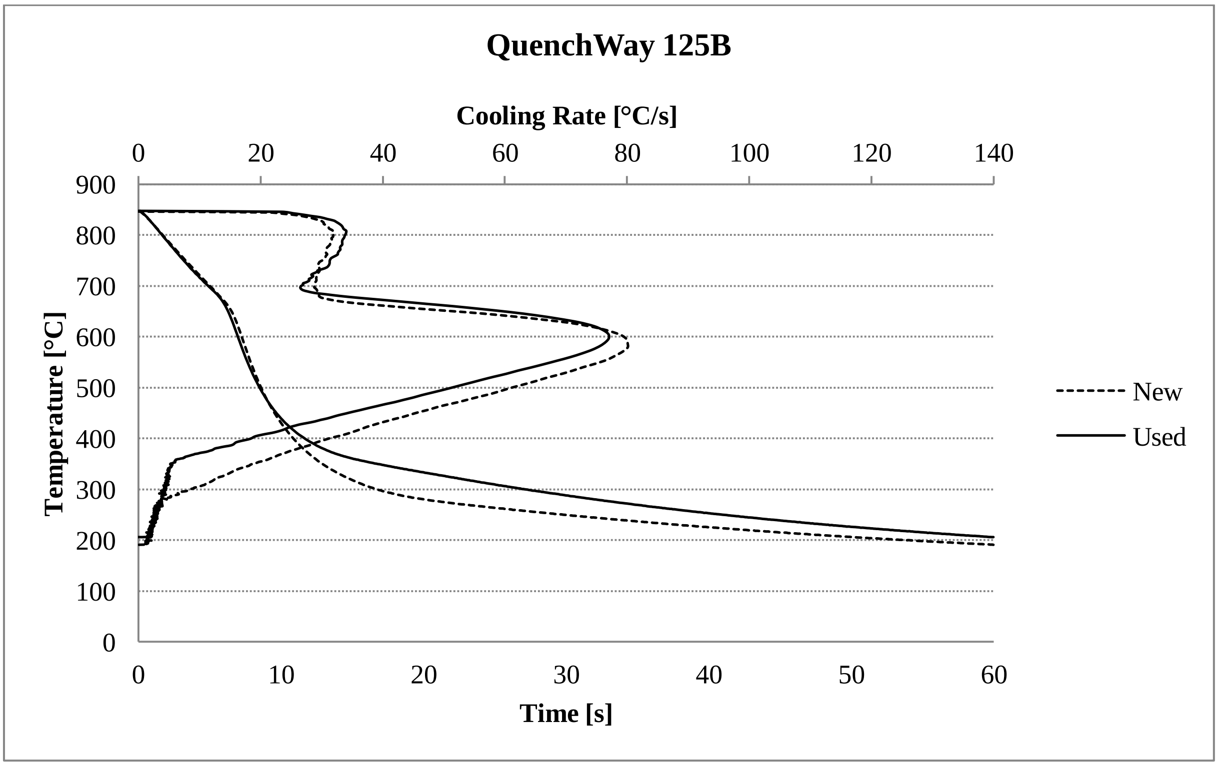

Figure 16 *Cooling and cooling rate curve depending on condition for 1 litre QuenchWay 125 B from Company B at 120°C without agitation.*

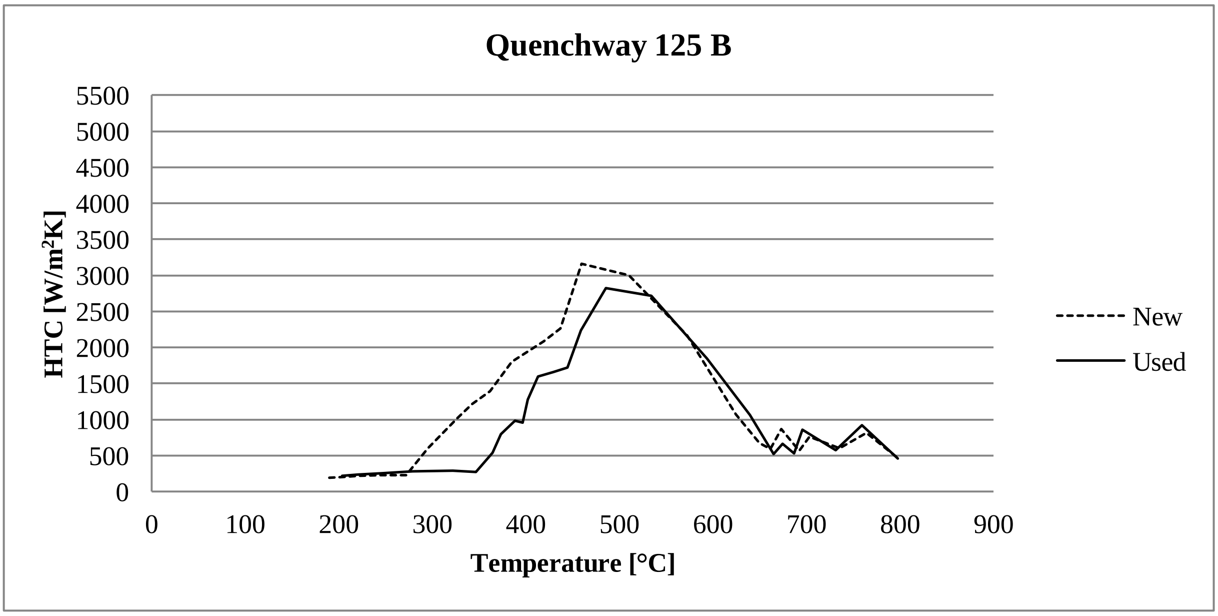

Figure 17 *Heat Transfer Coefficient curve depending on condition for 1 litre QuenchWay 125 B from Company B at 120°C without agitation.*

Figure 18 shows the difference in cooling and cooling rate curves between 10 litres of new and used Klen Quench 140 from Company C. The trend shows clearly that new oil has a higher cooling ability than used oil.

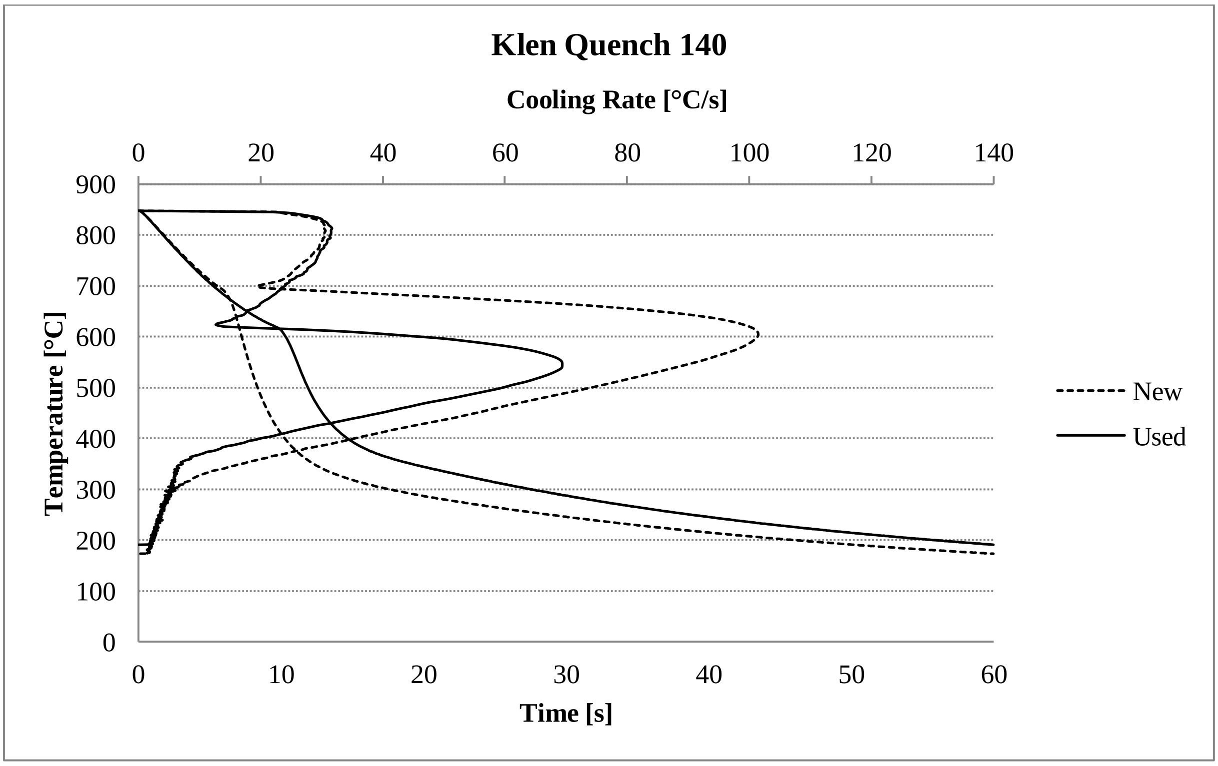

Figure 18 *Cooling and cooling rate curve depending on condition for 10 litres Klen Quench 140 from Company C at 95°C without agitation.*

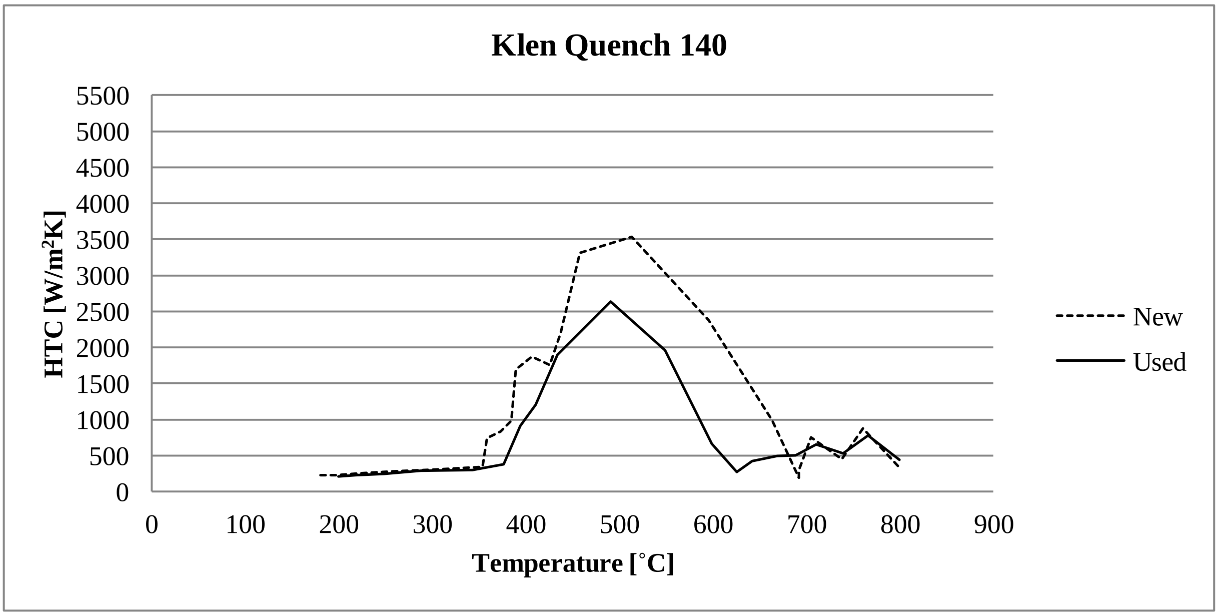

Figure 19 *Heat Transfer Coefficient curve depending on condition for 10 litres Klen Quench 140 from Company C at 95°C without agitation.*

4.2.5 Volume

How the cooling of QuenchWay 125 B is depending on the volume of the oil is shown in figure 20 and 21. The volume is changing from 1 to 10 litres and the cooling rate is increasing with the increasing volume.

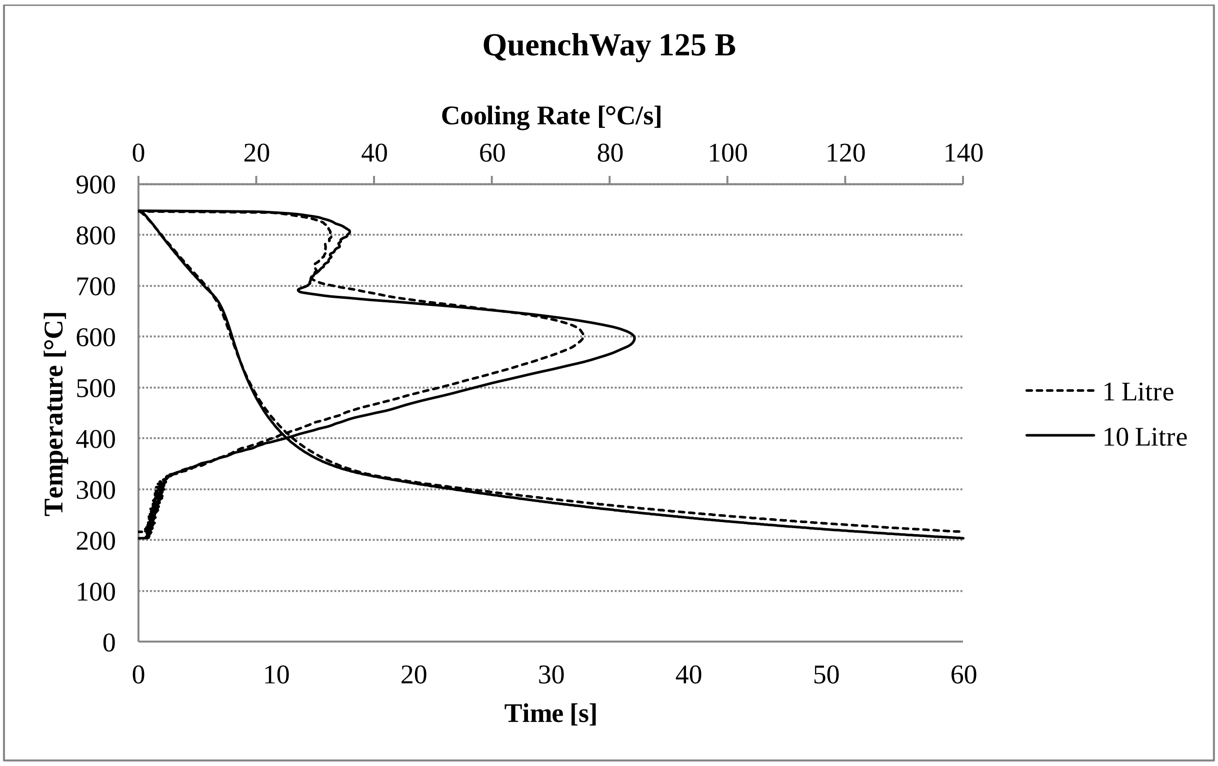

Figure 20 *Cooling and cooling rate curve depending on volume for new QuenchWay 125 B from Company A at 130°C without agitation.*

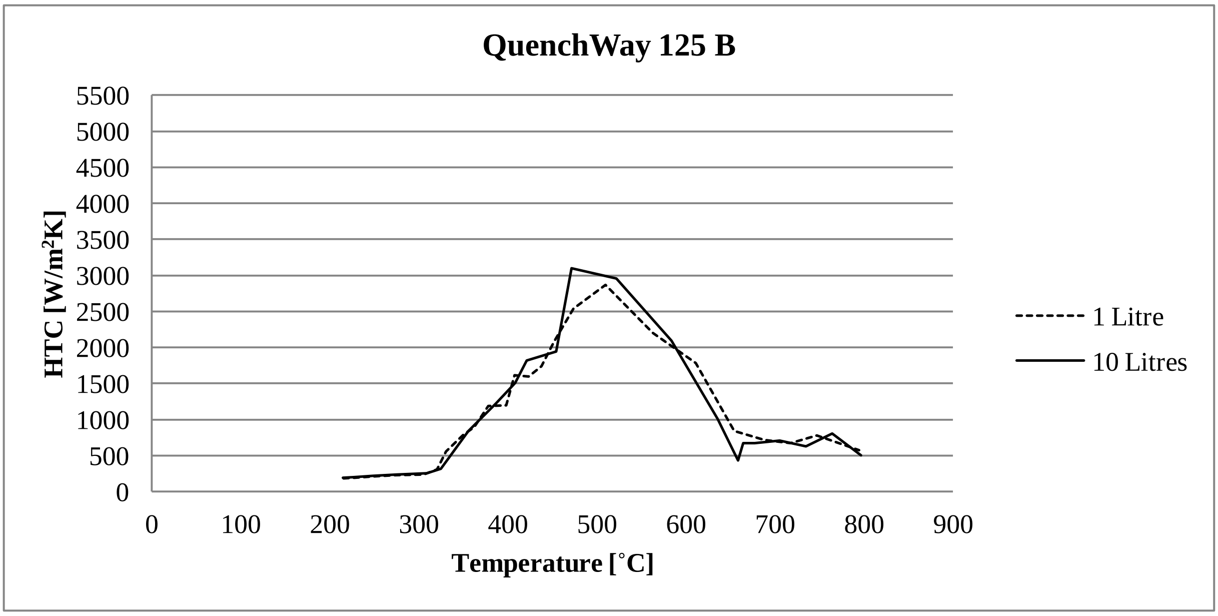

Figure 21 *Heat Transfer Coefficient depending on volume for new QuenchWay 125 B at 130°C without agitation.*

4.2.6 Oil

Different kinds of oils have been investigated during this thesis and they all have different properties and cooling abilities. In figure 22 the cooling and cooling rate curve for the different oils can be seen. Each measure was performed on 1 litre of new oil.

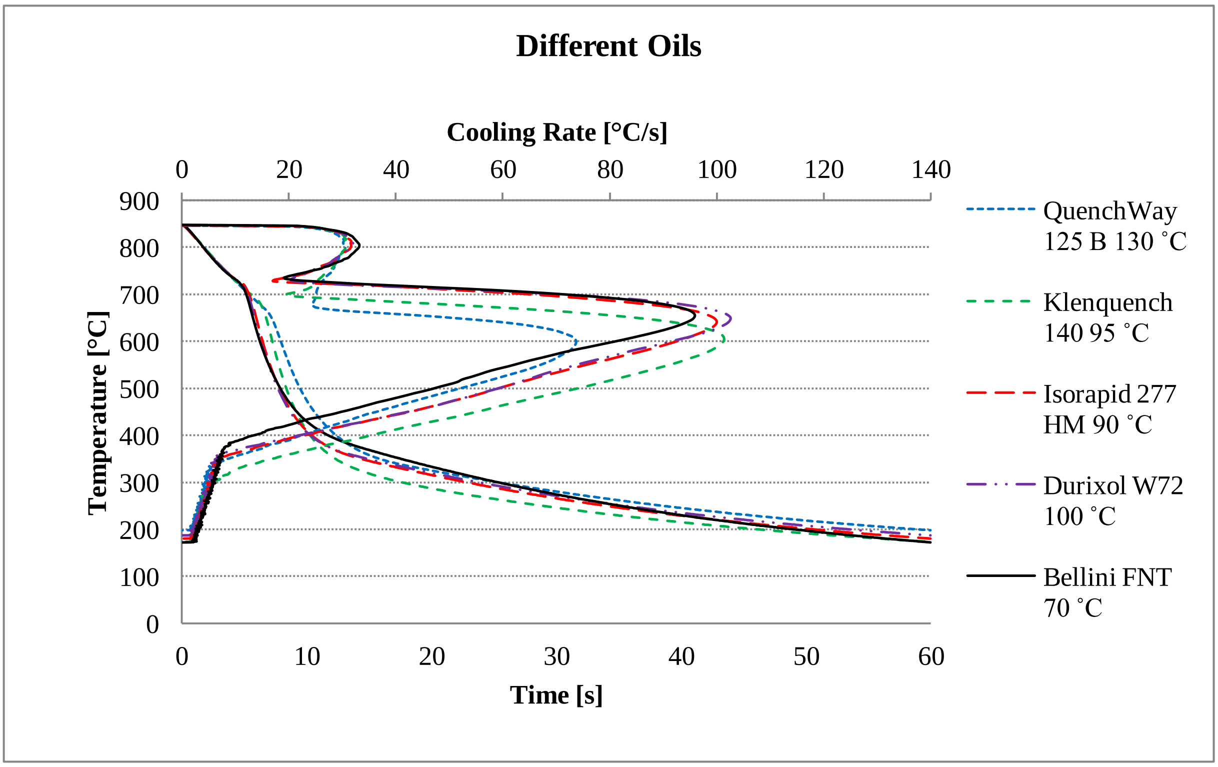

Figure 22 *Cooling and cooling rate curve from different oils without agitation.*

4.3 Hardening of steel

Some of the results from the Vickers Hardness Test performed on the hardened steel are here presented. On each steel piece two hardness profiles were measured and the values in the figures are the mean values of the two profiles. In figure 23 the hardness profiles for steel 100Cr6 with diameter

40 mm quenched in Klen Quench 140 can be seen. Closer to the surface the steel piece is harder compared to in the core. The steel piece that has been quenched with new oil has a higher hardness then the one quenched in used oil.

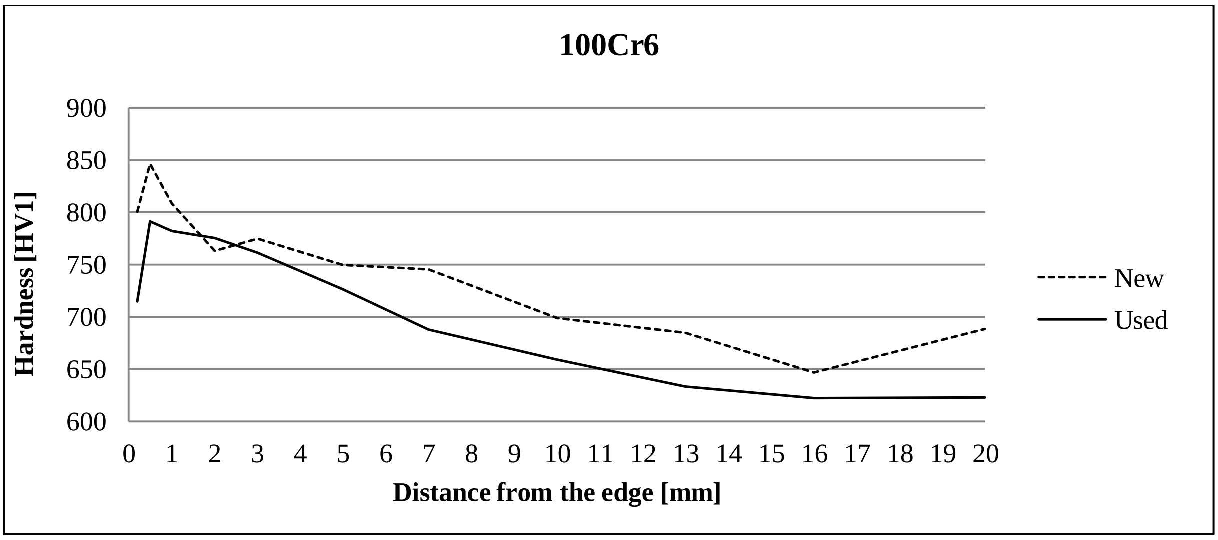

Figure 23 *Measured hardness on a 40 mm diameter test bar of 100Cr6 quenched in Klen Quench 140 from Company C at 75°C with agitation 0.2 m/s and an furnace temperature of 860°C.*

The rest of the Vickers Hardness Tests performed did not give any differences in hardness depending on condition. In figure 24 the hardness profiles for steel 16MnCr5 steel with diameter 40 mm quenched in QuenchWay 125 B from Company A can be seen. This figure is representative for the rest of the hardness measurements. The figure indicates a higher hardness closer to the edge of the steel piece compared to the core. The rest of the hardness profiles can be seen in Appendix E.

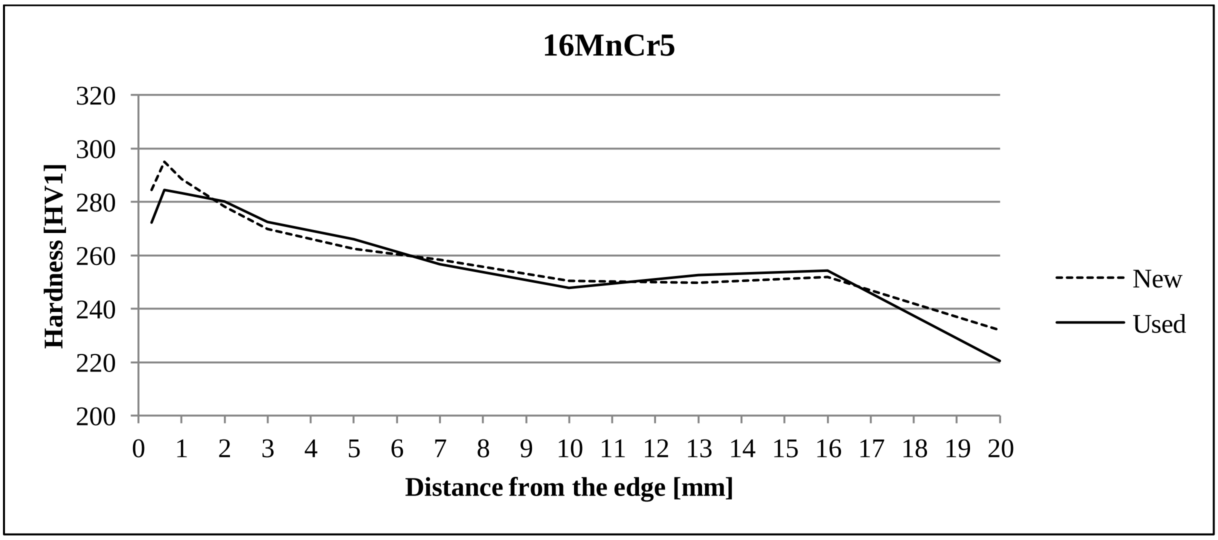

Figure 24 *Measured hardness on a 40 mm diameter test bar of 16MnCr5 quenched in QuenchWay 125 B from Company A at 130°C with agitation 0.2 m/s and an furnace temperature of 880°C.*

5 Analysis

Here is the analysis of the results from the measurements on the quenching oils. First a summary over the results from the analysis will be stated and then the analysis will be shown.

5.1 Summary

The general trend for all oils is that the Heat Removal Capacity and Hardening Power increases for increasing temperature. The analysis shows that for QuenchWay 125 B and Klen Quench 140 the Heat Removal Capacity and Hardening Power is increasing for increasing agitation and volume. The change in Heat Removal Capacity and Hardening Power for change in condition depends on the company the oil comes from, but for most oils the used oil has a lower cooling ability than the new oil.

QuenchWay 125 B from Company A has temperature and condition as significant parameters which mean that these parameters need to be properly set since they affect the cooling ability of the oil. For QuenchWay 125 B the company that the oil comes from affects the cooling ability, different production environments affect the oil in different ways.

Klen Quench 140 has temperature, condition and the interaction between them as significant which means that the parameter setting needs to be carefully selected. Small modifications can results in large effects on the cooling ability.

5.2 QuenchWay 125 B

The analysis of QuenchWay 125 B is divided depending on Company. First each company will be analyzed on its own and then an analysis containing both will be presented.

5.2.1 Company A

A General Linear Model was performed on one litre QuenchWay 125 B from Company A. In the method about the General Linear Model, Section 2.7, it is described how this was performed. If a selected parameter in the model was not significant it was removed. An example of this can be seen in figures 25 and 26 and table 2 and 3. The measurements are from both new and used QuenchWay 125 B at 70, 90 and 130°C. In figure 25 the first Residual Plot is shown together with the values in table 2. In figure 26 and table 3 the Residual Plot is shown when the model is redone and the sources condition and condition*temperature is removed. The values in both figures are measured in Heat Removal Capacity. In figure 27 the final Residual Plot is shown when it is measured in Hardening Power. The p-values from the models in figure 25, 26 and 27 are showing in table 2, 3 and 4 together with the R-Sq(adj) value that is indicating how much of the variation is covered by the model.

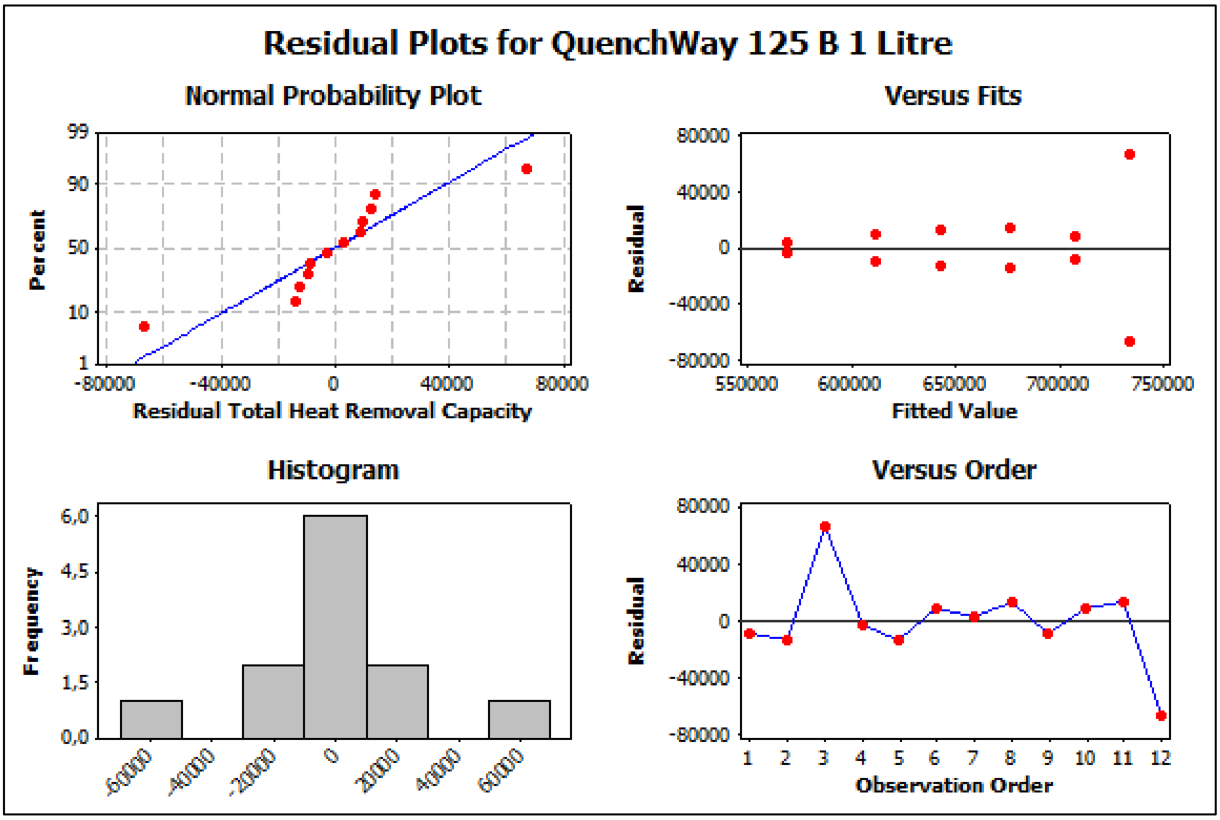

Figure 25 *The first Residual Plot for Heat Removal Capacity versus temperature and condition for 1 litre new and used QuenchWay 125 B from Company A without agitation,*

In the versus fits figure in figure 25 there is two sets of points, one above and one below the line. This is because each configuration of parameter settings had two measurements performed. The Versus Order plot in all Residual Plots is not significant since the observation order is not the order in which the measurements were performed.

Table 2 *The p-values and R-Sq(adj) for the first General Linear Model for Heat Removal Capacity versus temperature and condition for 1 litre new and used QuenchWay 125 B from Company A without agitation.*

Source	**p-value**
Temperature	0.012
Condition	0.199
Condition*Temperature	0.959
R-Sq(adj) = 61.69%	

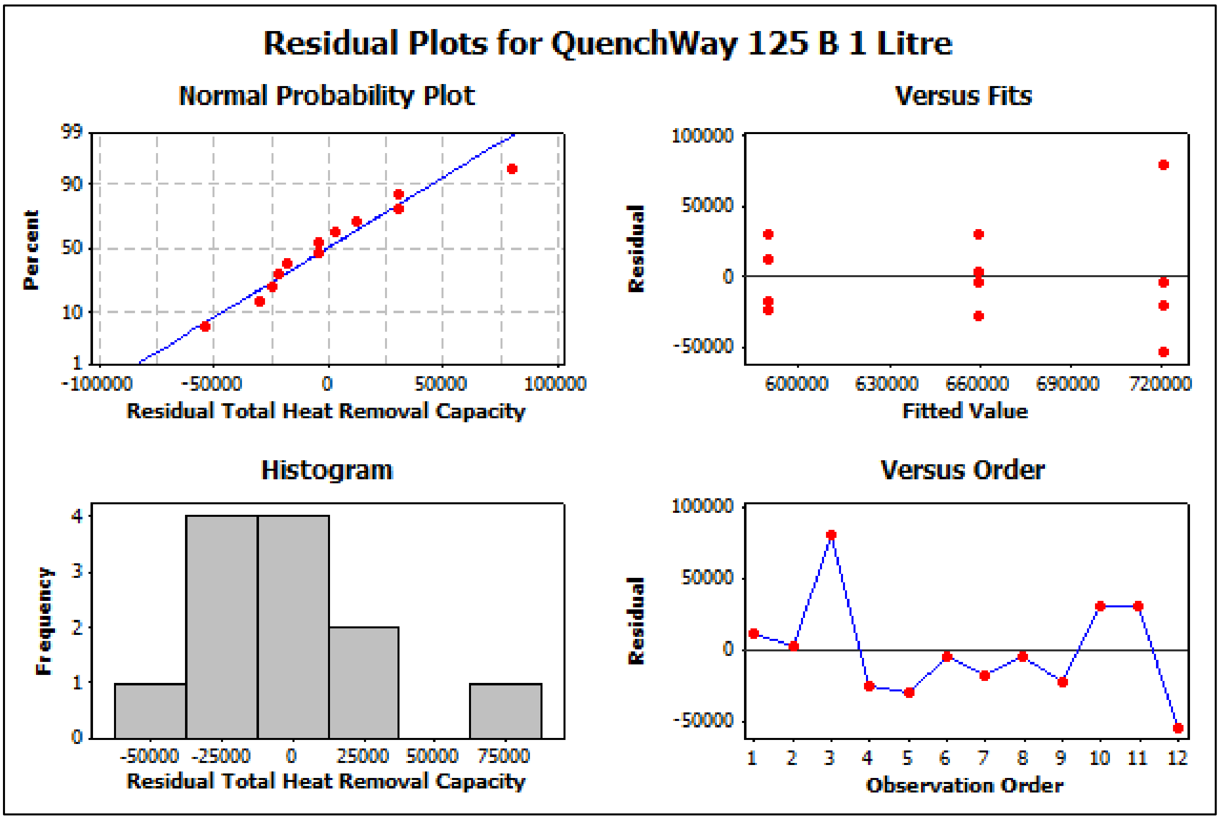

Figure 26 *The final Residual Plot for Heat Removal Capacity versus temperature and condition for 1 litre new and used QuenchWay 125 B from Company A without agitation.*

Table 3 *The p-values and R-Sq(adj) for the final General Linear Model for Heat Removal Capacity versus temperature and condition for 1 litre new and used QuenchWay 125 B from Company A without agitation.*

Source	p-value
Temperature	0.003
R-Sq(adj) = 65.22%	

One unusual observation in the model was found. An unusual observation is an observation that differs from the other values by for example a high residual. The observation was for used oil at 130°C. A lot of factors affects the compilation of curves when measured by the *SmartQuench* equipment and this one might have been affected differently than the others. Since no clear reason were found for the unusual observation it was not removed from the final model.

In the Versus Fits plot in figure 26 the three groups visualizes the three temperatures 70, 90 and 130˚C. The temperatures increases along the x-axis and shows that the measurements gets more uncertain for higher temperatures of the oil.

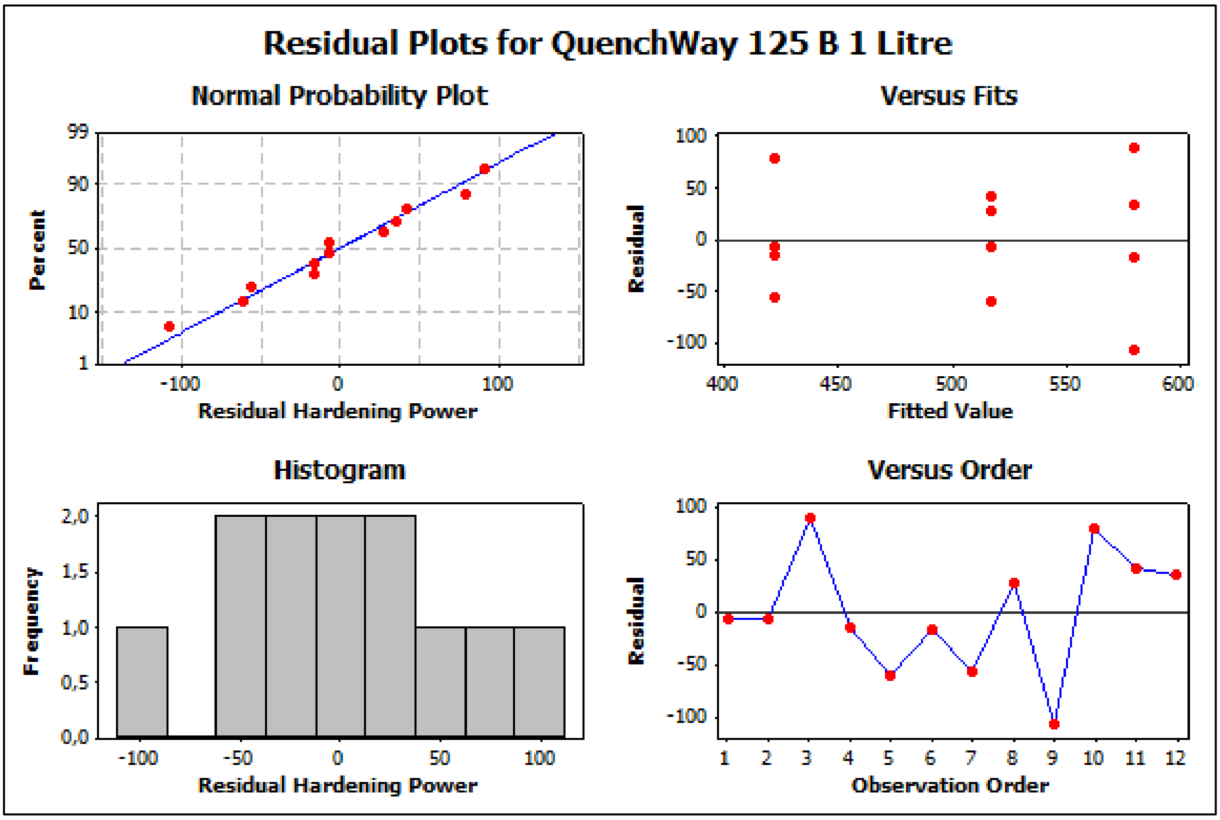

Figure 27 *Final Residual Plot for Hardening Power versus temperature and condition for 1 litre new and used QuenchWay 125 B from Company A without agitation.*

Table 4 *The p-values and R-Sq(adj) for the General Linear Model for Hardening Power versus temperature and condition for 1 litre new and used QuenchWay 125 B from Company A without agitation.*

Source	**p-value**
Temperature	0.027
Condition	0.006
R-Sq(adj) = 69.51%	

In figure 28 and 29 two Interaction Plots are showing the dependencies between temperature and condition. The corresponding Main Effect Plots can bee seen in Appendix C. The values in figure 28 are measured in Heat Removal Capacity and the values in figure 29 are measured in Hardening Power. With increasing temperature the cooling ability increases. Used oil has a higher cooling ability than new oil, this differs from oils from other companies where new oil has a higher cooling ability.

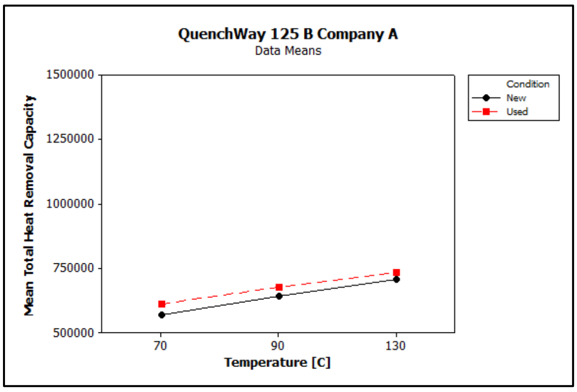

Figure 28 *Total Heat Removal Capacity depending on condition and temperature for 1 litre new and used QuenchWay 125 B from Company A without agitation.*

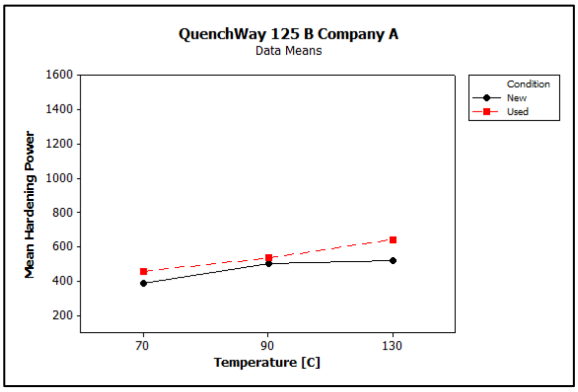

Figure 29 *Hardening Power depending on condition and temperature for 1 litre new and used QuenchWay 125 B from Company A without agitation.*

In figure 30 and 31 two Interaction Plots are showing the dependencies between agitation and condition. The measurements were performed on 10 litres QuenchWay 125 B from Company A. Measurements were performed on both new and used oil at 130°C with agitation 0.0, 0.2, 0.35 and 0.5 m/s. The values in figure 30 are measured in Heat Removal Capacity and in figure 31 they are measured in Hardening Power. With increasing agitation the cooling ability is increasing. Used oil from this company has a higher cooling ability than new. The corresponding main effect plots can bee seen in Appendix C together with two General Linear Models in Appendix D. The general linear model stated both condition and agitation as significant parameters.

In the General Linear Model, seen in Appendix D, unusual observations were found. In Figure 131 all but one unusual observation was for measures with agitation 0.35 or 0.5 m/s. This is because the software *SQIntegra* can calculate the T_{cp} value for the cooling rate curve incorrect for high agitations. The software calculates the value by third degree integrals, for high agitations the convection phase has no clear start which makes it difficult to calculate. For the unusual observations the calculated T_{cp} is much lower than the real value which gives the Hardening Power a higher value than it should have. They were removed from the final model since the reason for their behaviour is known. One unusual observation was found for used oil at zero agitation. The reason for this measurement is not known and was therefore not removed.

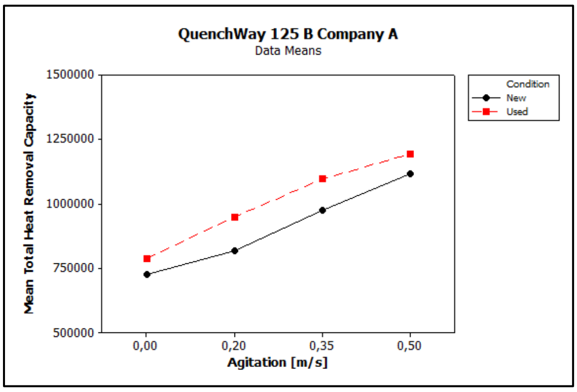

Figure 30 *Total Heat Removal Capacity depending on condition and agitation for 10 litres QuenchWay 125 B from Company A at 130°C.*

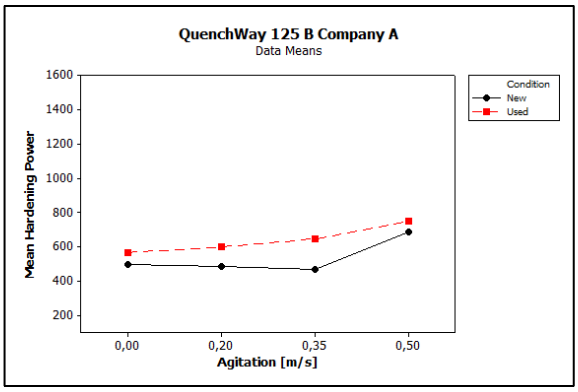

Figure 31 *Hardening Power depending on condition and agitation for 10 litres QuenchWay 125 B from Company A at 130°C.*

One last General Linear Model was performed on new and used QuenchWay 125 B from Company A. The corresponding Residual Plots can be seen in figure 32. Measurements were performed when the temperature was at 130°C and the volume was 1 or 10 litres. The values are measured in Heat Removal Capacity. In table 5 the p-values for the different factors are presented together with the R-Sq(adj) value that is indicating how much of the variation is covered by the model. The corresponding Residual Plot measured in Hardening Power can be seen in Appendix D.

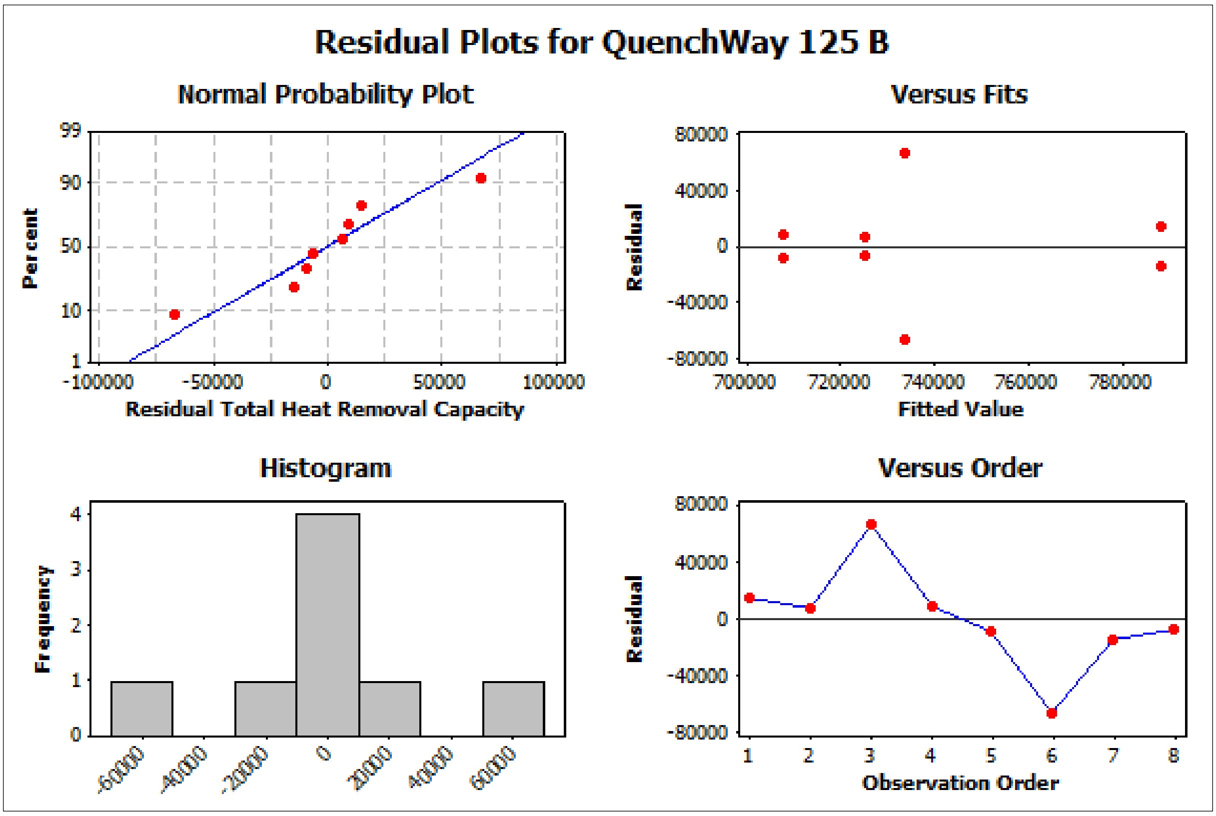

Figure 32 *Residual Plot for Heat Removal Capacity versus condition and volume for new and used QuenchWay 125 B from Company A at 130°C without agitation.*

Table 5 *The p-values and R-Sq(adj) for the General Linear Model for Heat Removal Capacity versus condition and volume for new and used QuenchWay 125 B from Company A at 130°C without agitation.*

Source	p-value
Condition	0.267
Volume	0.359
Condition * Volume	0.618
R-Sq(adj) = 0.39 %	

No sources were significant and a reason for that could be the high amount of measurement noise like variation from operator and equipment. This means that no conclusions about the oils cooling ability depending on condition and volume can be drawn. The corresponding Residual Plot measured in Hardening Power can be seen in Appendix D that show that the parameter condition is significant and has a p-value of 0.04 but the R-Sq(adj) is only 45%. This means that only 45% of the variation in the measurements is covered by the model.

In figure 33 and 34 two Scatter plots can be seen. In figure 33 it is for Heat Removal Capacity versus volume and in figure 34 it is Hardening Power versus volume. From the figures no general relationship between the measurements can be seen.

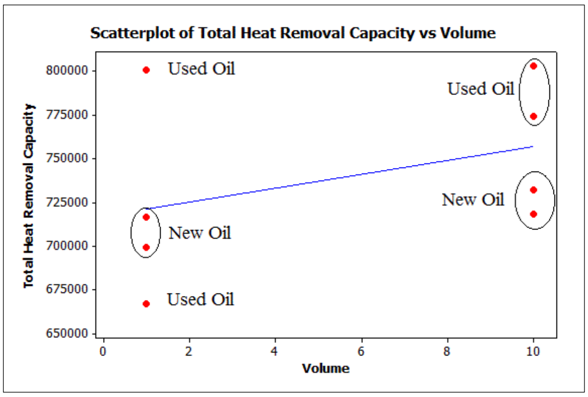

Figure 33 *Scatter plot for Heat Removal Capacity versus volume for new and used QuenchWay 125 B from Company A at 130°C without agitation.*

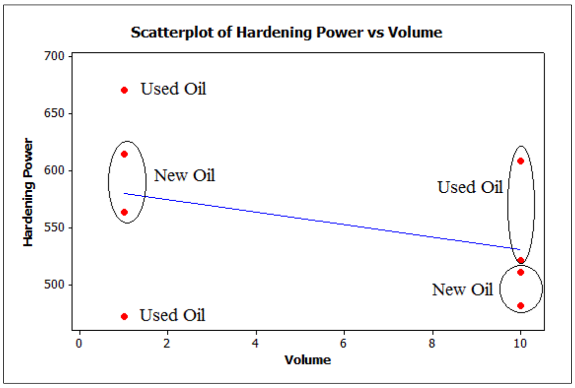

Figure 34 *Scatter plot for Hardening Power versus volume for new and used QuenchWay 125 B from Company A at 130°C without agitation.*

In figure 35 and 36 two Interaction Plots are showing the dependencies between volume and condition. The corresponding Main Effect Plots can bee seen in Appendix C. The values in figure 35 are measured in Heat Removal Capacity and in figure 36 they are measured in Hardening Power. When measuring in Heat Removal Capacity the cooling ability is increasing with increasing volume. When using Hardening Power instead the cooling ability is almost independent of the volume change. Used oil has higher cooling ability than new.

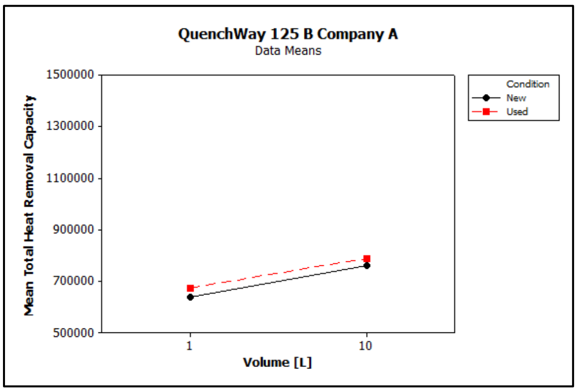

Figure 35 *Total Heat Removal Capacity depending on condition and volume for QuenchWay 125 B from Company A at 130°C without agitation.*

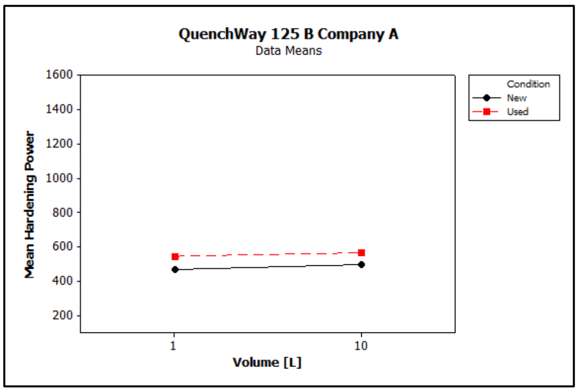

Figure 36 *Hardening Power depending on condition and volume for QuenchWay 125 B from Company A at 130°C without agitation.*

5.2.2 Company B

Analysis was performed on 1 litre of new and used QuenchWay 125 B from Company B. The amount of measurements was not enough to be able to perform a General Linear Model. The resulting Interaction Plots can be seen in figures 37 and 38 and the corresponding Main Effect Plots can be seen in Appendix C. The values in figure 37 are measured in Heat Removal Capacity and in figure 38 they are measured in Hardening Power. With increasing temperature the cooling ability is increasing for all oils except for used oil when the values are measured in Hardening Power. New oil has a higher cooling ability compared to used oil.

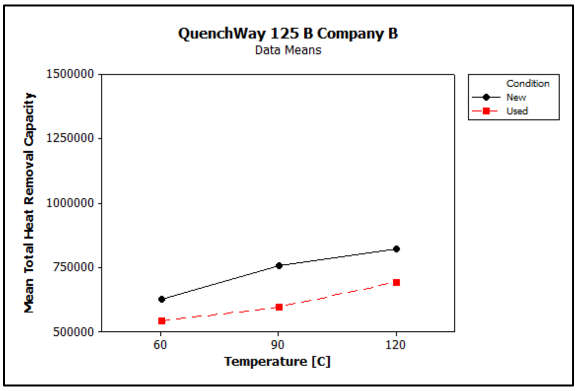

Figure 37 *Total Heat Removal Capacity depending on condition and temperature for 1 litre QuenchWay 125 B from Company B without agitation.*

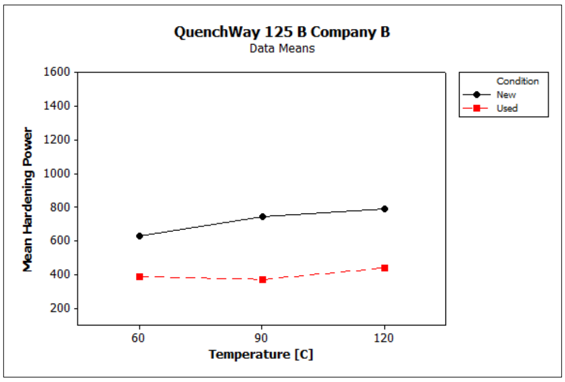

Figure 38 *Hardening Power depending on condition and temperature for 1 litre QuenchWay 125 B from Company B without agitation.*

5.2.3 Company A and B

An analysis was performed on 1 litre QuenchWay 125 B from both Company A and B to see the differences in behaviour between the different conditions of the oil. The resulting interaction plot can be seen in figure 39 and 40 for both Total Heat Removal Capacity and Hardening Power.

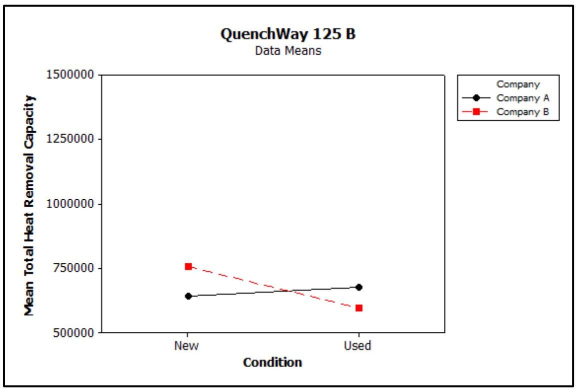

Figure 39 *Total Heat Removal Capacity depending on condition and company for 1 litre new and used QuenchWay 125 B at 90°C.*

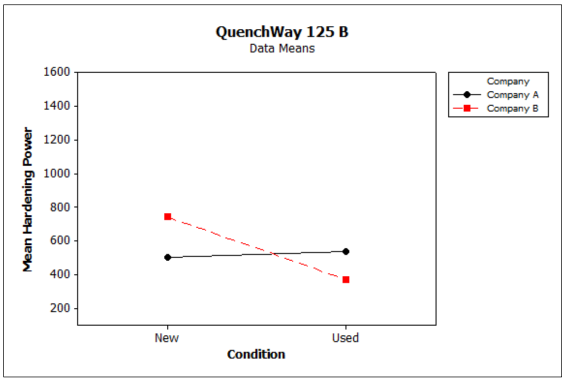

Figure 40 *Hardening Power depending on condition and company for 1 litre new and used QuenchWay 125 B at 90°C.*

The plots in figures 39 and 40 show an interaction between company and condition. For Company A the oil is less sensitive to the change in condition than the oil from Company B. The oil from Company B has a higher Heat Removal Capacity and Hardening Power for new oil compared to used oil. The oil from company A shows the opposite effect; it has higher Heat Removal Capacity and Hardening Power for the used oil. There is a difference between the measures of new oil from the different companies. The reason for this is outside this projects scope.

5.2.4 Klen Quench 140

A General Linear Model was performed on 1 litre Klen Quench 140 from Company C. The corresponding Residual Plots can be seen in figure 41 and 42. Measurements from both new and used oil at 75 and 95 °C were included in the model. The values in figure 41 are measured in Heat Removal Capacity and in figure 42 they are measured in Hardening Power. In table 6 and 7 the p-values for the different factors are presented together with the R-Sq(adj) value that is indicating how much of the variation is covered by the model.

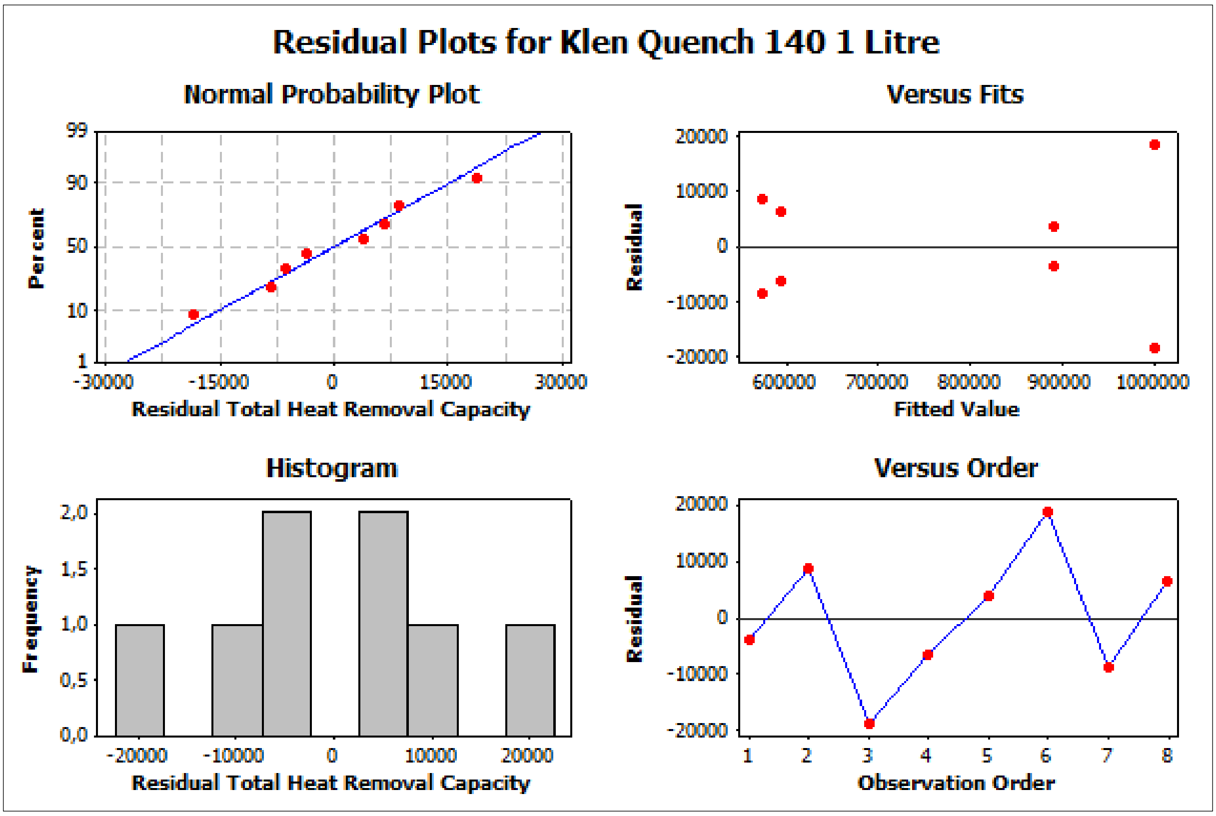

Figure 41 *Residual Plots for Heat Removal Capacity versus temperature and condition for 1 litre new and used Klen Quench 140 from Company C at 75 and 95°C without agitation.*

Table 6 *The p-values and R-Sq(adj) for the General Linear Model for Heat Removal Capacity versus temperature and condition for 1 litre new and used Klen Quench 140 from Company C at 75 and 95°C without agitation.*

Source	p-value
Temperature	0.000
Condition	0.004
Condition * Temperature	0.015
R-Sq(adj) = 99.40 %	

The Versus Fits plot in figure 41 shows two groups of points. The ones to the left are measurements on used oil and the ones to the right are new oil measurements. They show that the new oil is more sensitive to temperature change than used oil that seems to be more robust. The same behaviour can be seen in figure 42 for the Hardening Power measures.

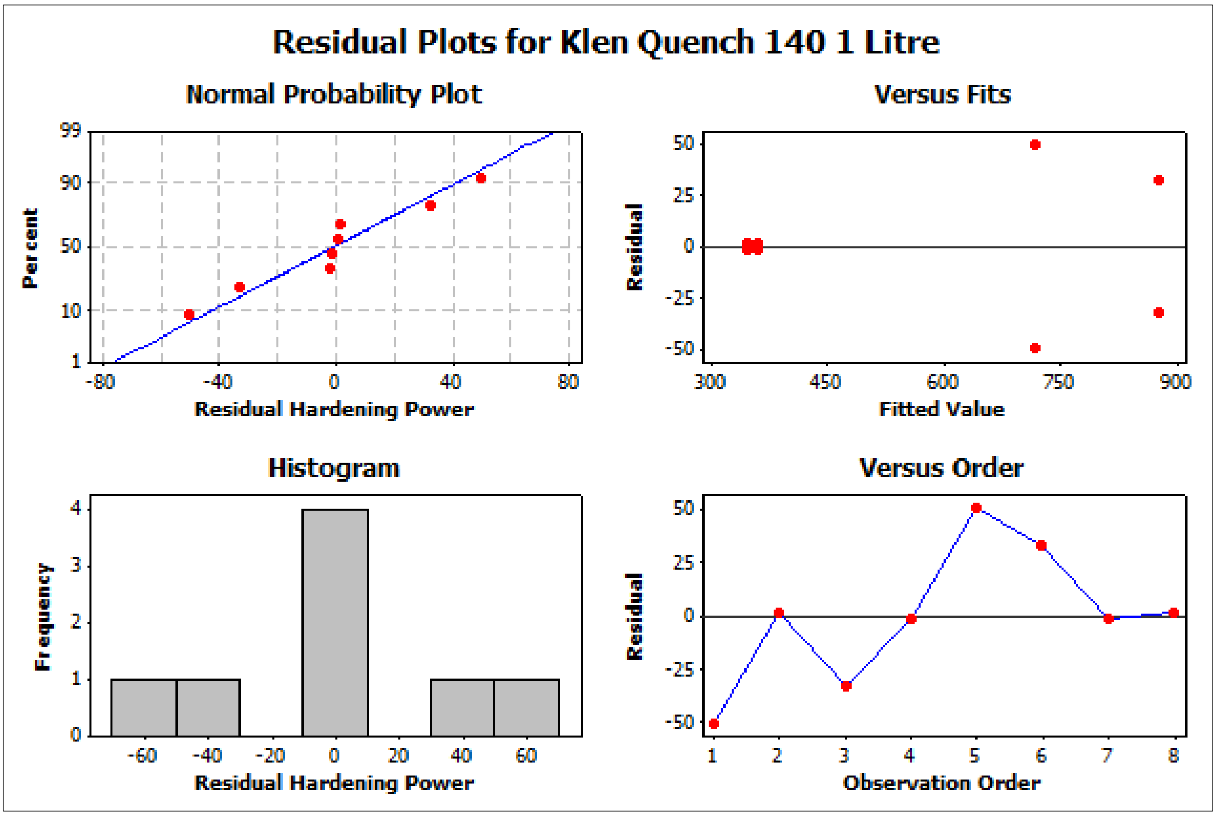

Figure 42 *Residual Plots for Hardening Power versus temperature and condition for 1 litre new and used Klen Quench 140 from Company C at 75 and 95°C without agitation.*

Table 7 *The p-values and R-Sq(adj) for the General Linear Model for Hardening Power versus temperature and condition for 1 litre new and used Klen Quench 140 from Company C at 75 and 95°C without agitation.*

Source	**p-value**
Temperature	0.074
Condition	0.000
Condition*Temperature	0.044
R-Sq(adj) = 97.04%	

In figure 43 and 44 two Interaction Plots are showing the dependence between condition and temperature. The corresponding Main Effect Plots can be seen in Appendix C. The values in figure 43 are measured in Heat Removal Capacity and in figure 44 they are measured in Hardening Power. An increase in temperature results in an increase in cooling ability. New oil has a higher cooling ability compared to used oil.

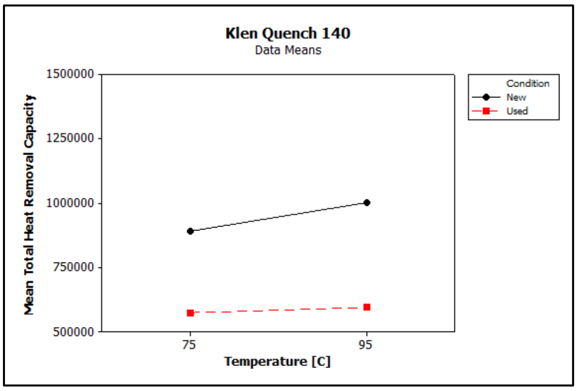

Figure 43 *Total Heat Removal Capacity depending on condition and temperature for 1 litre new and used Klen Quench 140 from Company C at 75 and 95°C without agitation.*

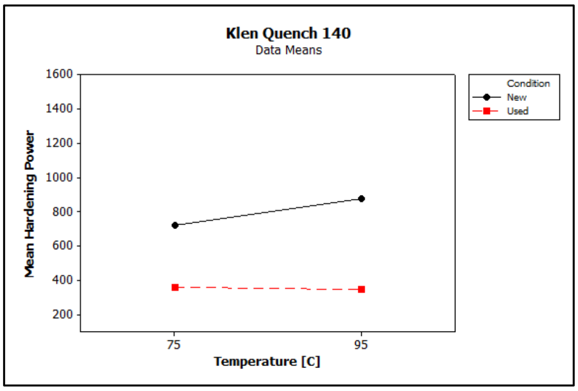

Figure 44 *Hardening Power depending on condition and temperature for 1 litre new and used Klen Quench 140 from Company C at 75 and 95°C without agitation.*

In figure 45 to 50 six different Interaction Plots are showing the dependencies between temperature, agitation and condition. The measurements were performed on 10 litres Klen Quench 140 from Company C. Measurements were performed on both new and used oil at 75 and 95°C with agitation 0.0, 0.2, 0.35, and 0.5 m/s. The corresponding Main Effect Plots and Residual Plots can be seen in Appendix D. The values in figure 45, 47 and 49 are measured in Heat Removal Capacity and figure 46, 48 and 50 are measured in Hardening Power. An increase in temperature gives a decrease in cooling ability except for new oil when measuring in Heat Removal Capacity. An increase in agitation gives an increase in cooling ability. New oil has a higher cooling ability compared to used oil.

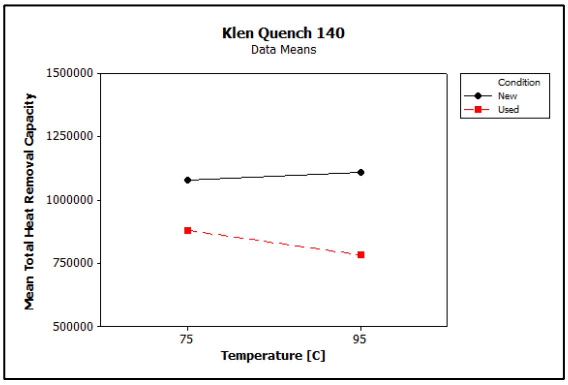

Figure 45 *Total Heat Removal Capacity depending on condition and temperature for 10 litres new and used Klen Quench 140 from Company C with agitation.*

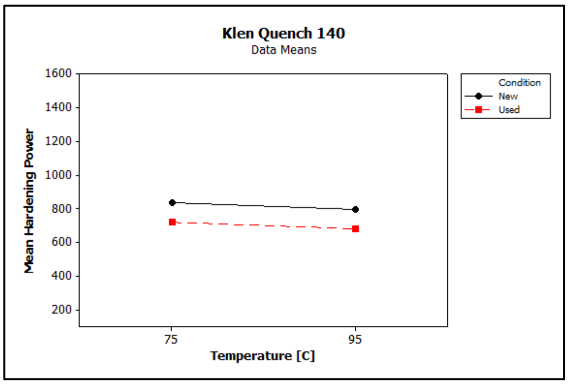

Figure 46 *Hardening Power depending on condition and temperature for 10 litres new and used Klen Quench 140 from Company C with agitation.*

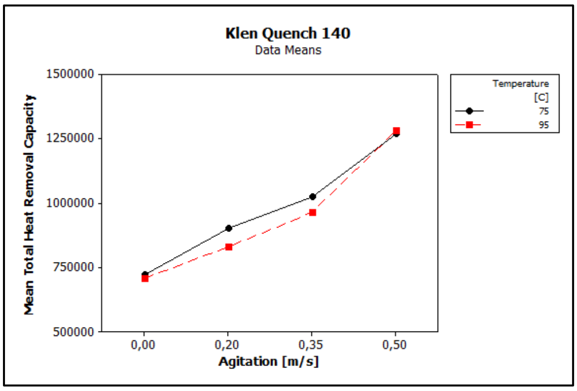

Figure 47 *Total Heat Removal Capacity depending on temperature and agitation for 10 litres new and used Klen Quench 140 from Company C.*

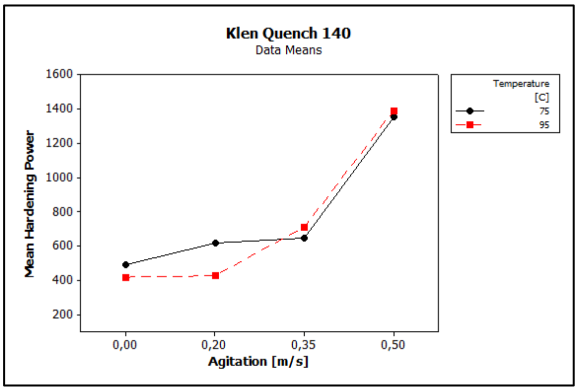

Figure 48 *Hardening Power on temperature and agitation for 10 litres new and used Klen Quench 140 from Company C.*

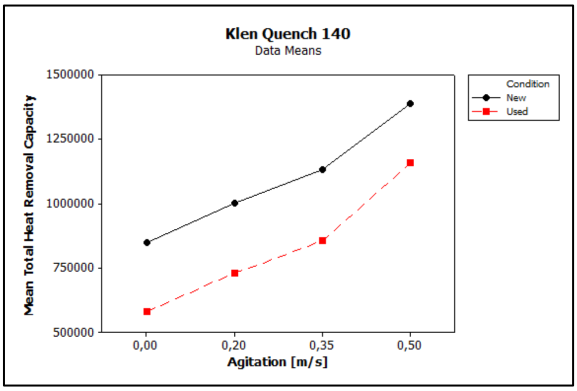

Figure 49 *Total Heat Removal Capacity depending on condition and agitation for 10 litre Klen Quench 140 from Company C at 75 and 95°C.*

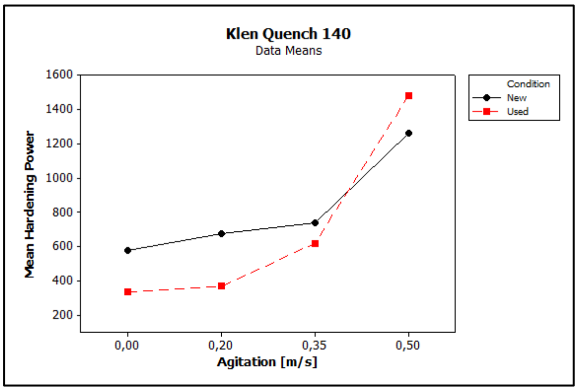

Figure 50 *Hardening Power depending on condition and agitation for 10 litre Klen Quench 140 from Company C at 75 and 95°C.*

In figure 51 and 52 two Interaction Plots are showing the dependencies between temperature and volume. The measurements were performed on new Klen Quench 140 from Company C. Measurements were performed when the temperature was either 75 or 95 °C and volume was either 1 or 10 litres. The corresponding Main Effect Plots can be seen in Appendix D together with the corresponding Residual Plots in Appendix D. The values in figure 51 are measured in Heat Removal Capacity and in figure 52 they are measured in Hardening Power. With increasing volume and temperature the cooling ability is increasing except for oils at 95°C measured with Hardening Power.

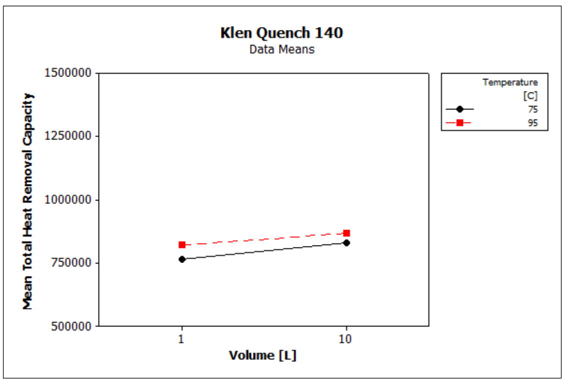

Figure 51 *Total Heat Removal Capacity depending on temperature and volume for 1 and 10 litres new Klen Quench 140 from Company C at 75 and 95°C.*

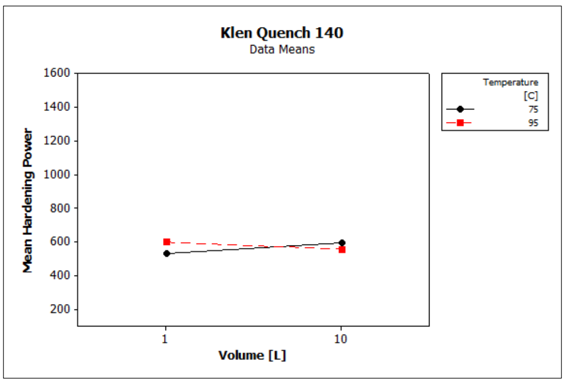

Figure 52 *Total Hardening Power depending on temperature and volume for 1 and 10 litres Klen Quench new 140 from Company C at 75 and 95°C.*

5.3 Isorapid 277 HM

Analyses to see how Isorapid 277 HM is depending on temperature, condition and company were performed, see interaction plots in figure 53-58. The corresponding Main Effect Plots can be seen in Appendix C. The values in figure 53, 55 and 57 are measured in Heat Removal Capacity and the values in figure 54, 56 and 58 are measured in Hardening Power. Isorapid 277 HM was collected from both Company B and D. All curves show an increase in cooling with a rise in temperature. The new oils from both companies' show a higher cooling ability than the used one.

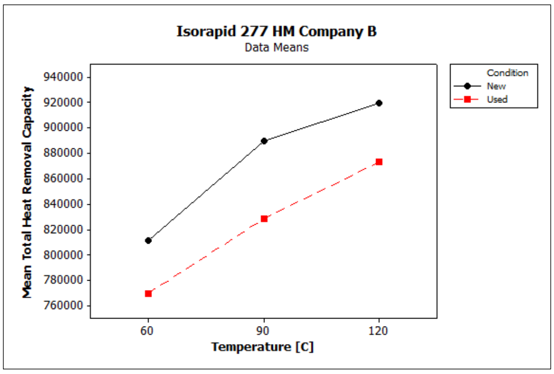

Figure 53 *Total Heat Removal Capacity depending on temperature and condition for 1 litre new and used Isorapid 277 HM from Company B without agitation.*

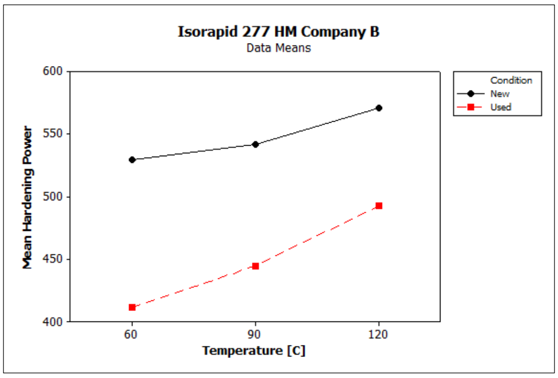

Figure 54 *Hardening Power depending on temperature and condition for 1 litre new and used Isorapid 277 HM from Company B without agitation.*

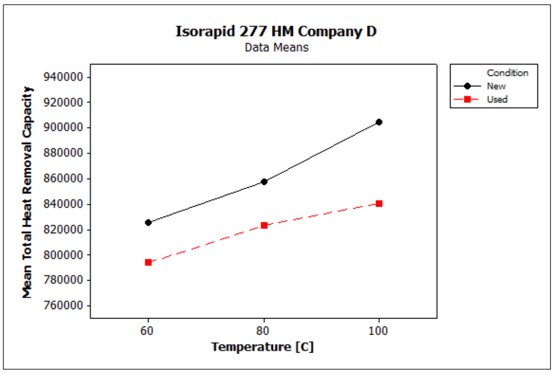

Figure 55 *Total Heat Removal Capacity depending on temperature and condition for 1 litre new and used Isorapid 277 HM from Company D without agitation.*

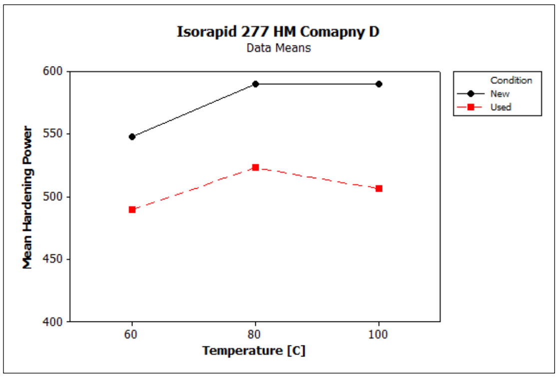

Figure 56 *Hardening Power depending on temperature and condition for 1 litre new and used Isorapid 277 HM from Company D without agitation.*

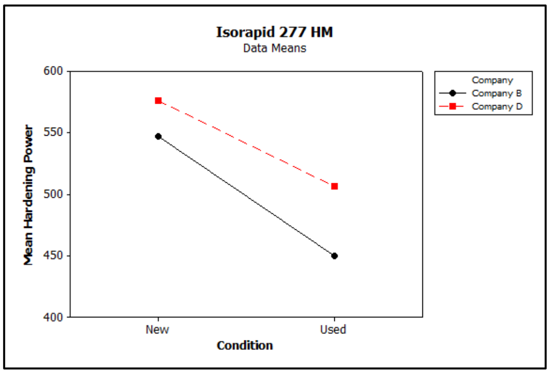

Figure 57 *Total Heat Removal Capacity depending on temperature and company for 1 litre new and used Isorapid 277 HM from Company D without agitation.*

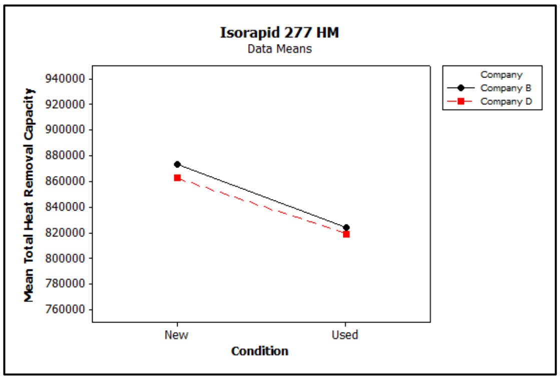

Figure 58 *Hardening Power depending on temperature and company for 1 litre new and used Isorapid 277 HM from Company D without agitation.*

5.4 Durixol W72

Measurements to see how Durixol W72 from Company E is depending on temperature and condition was performed, see the Interaction Plots in figure 59 and 60. The corresponding Main Effect Plots can bee seen in Appendix C. The values in figure 57 are measured in Heat Removal Capacity and in figure 58 it is measured in Hardening Power. The temperature was varying between 60, 80 and 100°C for new and used Durixol W72. Both figures indicate an increase in Heat Removal Capacity and Hardening Power when increasing temperature. When measuring with Total Heat Removal Capacity new oil showed larger cooling ability. When measuring in Hardening Power used oil showed a larger cooling ability compared to new.

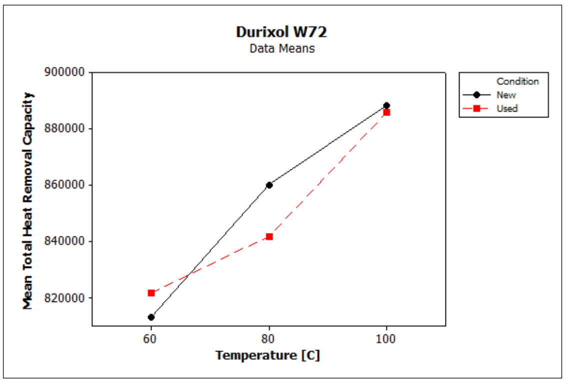

Figure 59 *Total Heat Removal Capacity depending on temperature for 1 litre new and used Durixol W72 from Company E without agitation.*

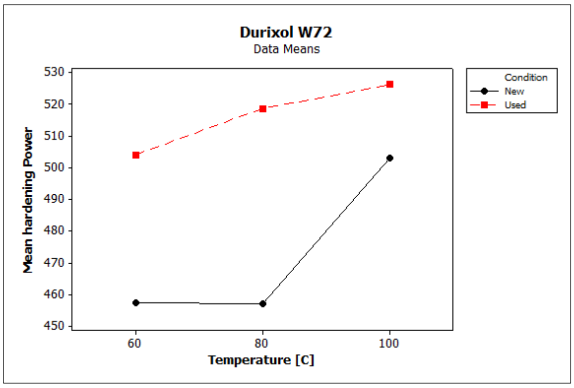

Figure 60 *Hardening Power depending on temperature for 1 litre new and used Durixol W72 from Company E without agitation.*

5.5 Correlation between Heat Removal Capacity and Hardening Power

The chosen values to evaluate the cooling ability of the oils are the Heat Removal Capacity and the Hardening Power. To see how they correlate, Fitted Line Plots were performed. QuenchWay 125 B from Company A and Klen Quench 140 from Company C have enough values to get a good overview of the correlation. Their Fitted Line Plots can be seen in figure 61 and figure 62 below. They show that there is correlation between the two values. Their Pearson number is 0.862 and 0.824 and states that when the Hardening Power increases so does the Heat Removal Capacity.

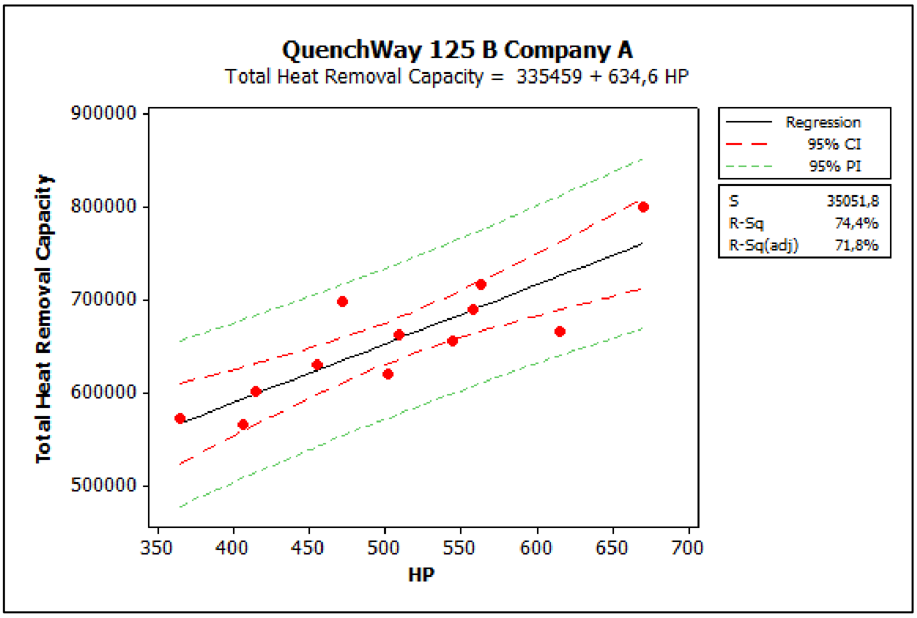

Figure 61 *Fitted Line Plot for Heat Removal Capacity versus Hardening Power for 1 litre QuenchWay125 B.*

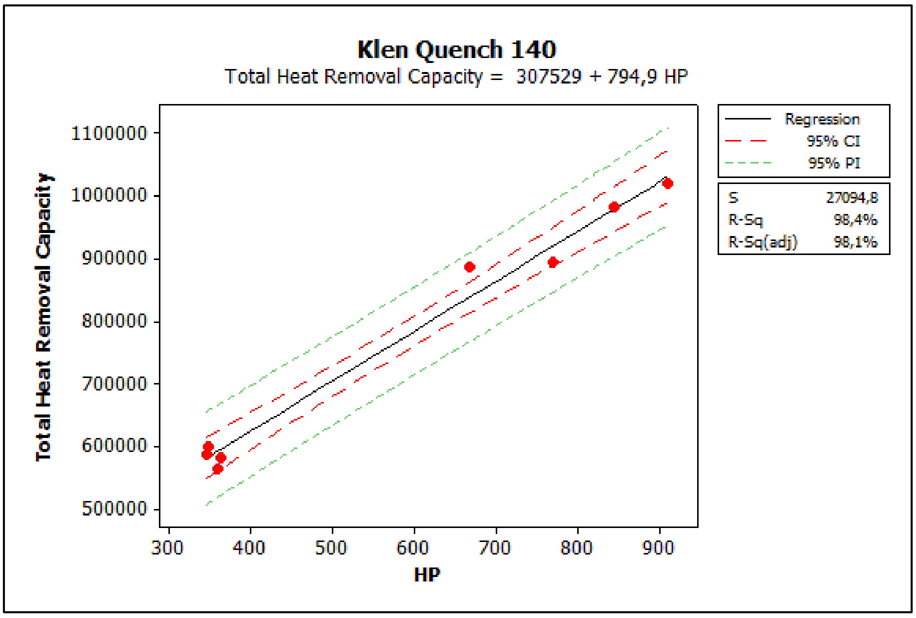

Figure 62 *Fitted Line Plot for Heat Removal Capacity versus Hardening Power for 1 litre Klen Quench 140.*

6 Discussion

The results from the measurements of cooling curves performed during this thesis seem to be according to the hypothesis stated under theory. An increase in temperature, agitation or volume increases the Heat Removal Capacity and Hardening Power. When looking at condition of the oil no general trend is seen. After using the oil in production the cooling ability is affected and three of the four tested oils show a decrease in cooling ability. Some oils become more robust against changes in parameters by the change in condition. The reason for the different outcomes for change in condition seems to be because of the different production conditions leading to e.g. variation of the contamination and cracking of the oils.

This report adds to the research within the field of heat treatments by increasing the number of measured cooling curves and calculated Heat Transfer Coefficient curves. The knowledge about the equipment *ivf SmartQuench®* and *SQIntera* has been increasing. Few limitations were found and recommendations for improvements were performed.

The method used in this project has been following the DMAIC method of Six Sigma. Under a larger project it is helpful to have some guidelines that can be followed. Different tools have been used and in the beginning this was especially helpful for defining the project. The Measurement System Analysis changed the way the measurements were performed and improved the reliability of the results. Without this the clear trends from the analysis could have been harder to detect. Early in the project a lot of focus was put on finding the characteristic values of the process. This made it easier to understand how the results from the measurements were going to be compared and analyzed. Some tools from Six Sigma were not applicable on the project but overall the method has been useful.

For analysing the results the statistical software Minitab for Six Sigma has been used. It has helped handle and analyse the large amount of data collected. Different statistical tools have been used which visually and numerically describes the data making the results easy to interpret.

There is always a risk of generating experimental uncertainties. The Measurement System Analysis performed on the equipment used for compiling cooling curves showed that the equipment is depending on the operator. The way the measurements are executed is affecting the resulting cooling curves. The software *SQIntegra* is used for calculating Heat Transfer Coefficients and Hardening Power. The software has a risk of misreading the value T_{cp} when the transition between the boiling and convection phase in the cooling curve is unclear, resulting in a miscalculated Hardening Power.

The results were measured in both Heat Removal Capacity and Hardening Power. Hardening Power is commonly mentioned in literature but the measure Heat Removal Capacity was developed during the project. This implies that there might be some uncertainties regarding its reliability. A correlation analysis was performed on the two measures for the collected data. It was seen that there were a correlation and that when one of them increased the other one did as well. This lead to the conclusion that the not so commonly used measure Heat Removal Capacity is valid to use in cooling ability investigations of mineral oils.

The compilation of data has not been performed according to a Design of Experiments method. This causes the measurements to take more time than needed and all effects of the parameters were not evaluated in all ways possible. To get a better compilation of data that is covering all effects of the parameters a Design of Experiments should have been performed. This to ensure that all the important information is collected from the measurements. The collected data was not enough to perform a General Linear Model for some of the oils. With measurements performed according to a Design of Experiments the statistical analyze would be able to evaluate more factors.

Future research could be performed in this field. One of the limitations in this report was how the oil on a chemical level changed after it had been used in production. A chemical analysis of the oil together with an investigation of the production environment is a suggestion for further investigation. An additional parameter that could be investigated is the viscosity of the oil.

According to literature there is a peak in cooling ability at a certain temperature. Below and above this temperature the cooling ability is lower. Where this peak occurs at for the different oils have not been investigated. The different oils have recommended using temperatures that are given from the manufacturer. An investigation on how the oils are behaving outside compared to inside the using temperature could be performed.

More hardening of steel can be performed to see how the different parameters other than condition are affecting the results. Beside Vickers Hardness Test further evaluations of the hardened steel can be investigated. With a light optical microscope it is possible to investigate the microstructure. An analysis of how the calculated hardness and microstructure of hardened steel from the software *SQInterga* can also be performed.

7 Conclusions

The four tested oils show significant differences in cooling ability. When increasing the temperature the Hardening Power and Heat Removal Capacity increase. The same trend is seen for increased agitation and volume. After the oil has been used in production the cooling ability is changed. Both increase and decrease in cooling ability is seen for the different oils after being used in production. These results are according to the stated hypothesis in Section 2.10.

The result from the Measurement System Analysis indicates that when performing tests on oils with different characteristic different operators can perform the tests. When only smaller changes on the oils are tested, like an increase in temperature all the tests needs to be performed by the same operator. This means that no benchmark can be done.

When different parameters of the oils are changed the characteristic values Hardening Power and Heat Removal Capacity are affected. The two values are varying in the same way and there is correlation between them. This means that the earlier unused Heat Removal Capacity can be used for measuring the cooling ability of a mineral oil.

Hardening of steel bars in new and used QuenchWay 125 B from Company A and Klen Quench 140 from Company C was performed. All but one of the resulting hardenings showed no confirmation on the differences in cooling ability for new and used oil could be seen. The cooling abilities of the oils were enough for reaching the maximum hardness for both steels. But the steel bars with a diameter of 40 mm that was quenched in Klen Quench 140 showed results that confirmed the results in Heat Removal Capacity and Hardening Power for the oil. The new oil gave a higher hardness than the used oil.

The software *SQIntegra* can misread values like T_{cp} from the cooling curve. This results in an incorrect calculated value for Hardening Power. The misreading can occur for cooling curves recorded using high agitation.

The method DMAIC of Six Sigma has been helpful to use during the project. The Define Phase made the understanding and the definition of the project clearer. The Measurement System Analysis increased the understanding of the measurement equipment. In the Analyze Phase the statistical software Minitab made the results clearer and easy to interpret.

8 Recommendations

In the User's Manual for *ivf SmartQuench*® some additional information could be added. This is to make sure the measurements are good and reliable. When measuring oils that are similar one operator should perform all measurements. Inexperienced operators should perform each measurement at least two times. This is to assure the result and to make sure the measurement system is stable.

The calculated T_{cp} from the software *SQIntegra* should be controlled against the cooling rate curve to see if it is correctly calculated. Another way of calculating this value could be considered.

The oils are affected differently after being used in production. It is important to investigate how the oils cooling ability is changing. If the cooling ability of the oil is changed drastically it may change the metal being quenched. This can result in residual stresses, cracks, need for rework etc, which is not good in a sustainable system.

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10 Appendix A

Six Sigma tools theory. The used tools in the project are here described and shown.

SIPOC

SIPOC stands for Supplier, Input, Process, Output and Customer. It is a table that states all of those categories. When doing SIPOC, start from “Output” and state what this process should deliver, is it information, a product or service? From this go to the column “Customers” and state the ones that will be the receivers of the outputs of the project. After that go back to the “Output” column and indicate what the customers require from each output, which measure should be improved. Here it is important that together reach one measure to improve and focus on before moving on with the other parts of the table. In the next step the process should be identified and where it starts and ends should be put in. From there the inputs that are required in the process should be stated and then identify the suppliers to them in the “Supplier” column. Last column is the second column under “Inputs” where the requirements for each input should be stated.

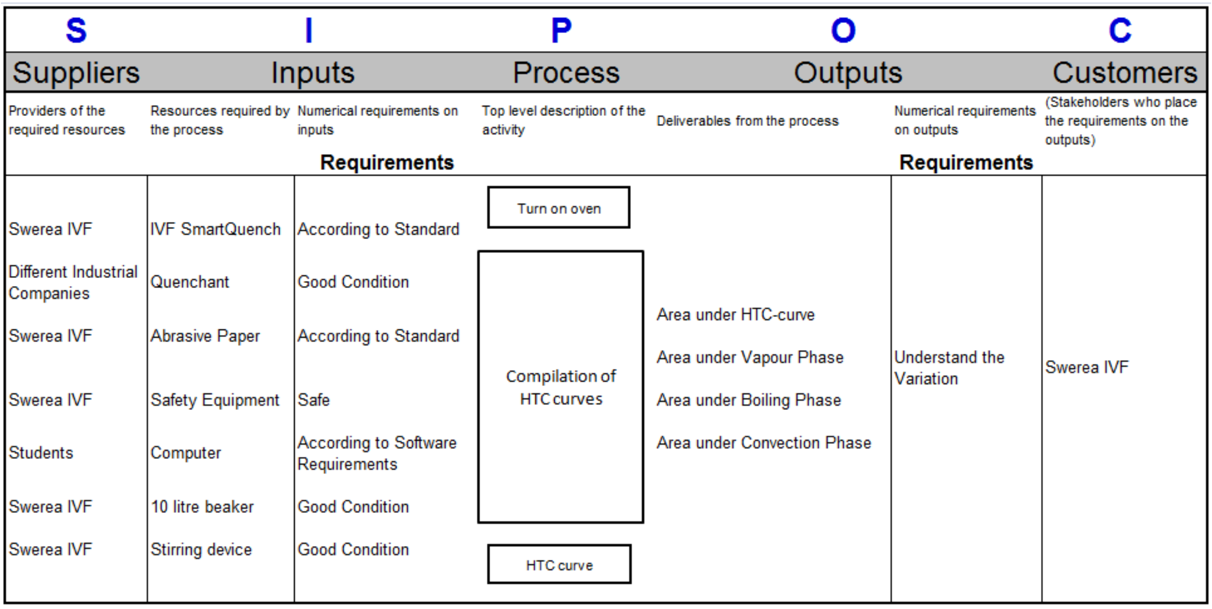

Figure 63 *SIPOC for the project.*

AIM

AIM stands for Affinity-Interrelationship Method and it is a problem solving tool. The affinity diagram and the interrelationship diagraph are two out of the 7 management tools AIM is based on. It shows different factors that are affecting the big Y and how they are related to each other. Also a grading on how important the different factors are is showing.

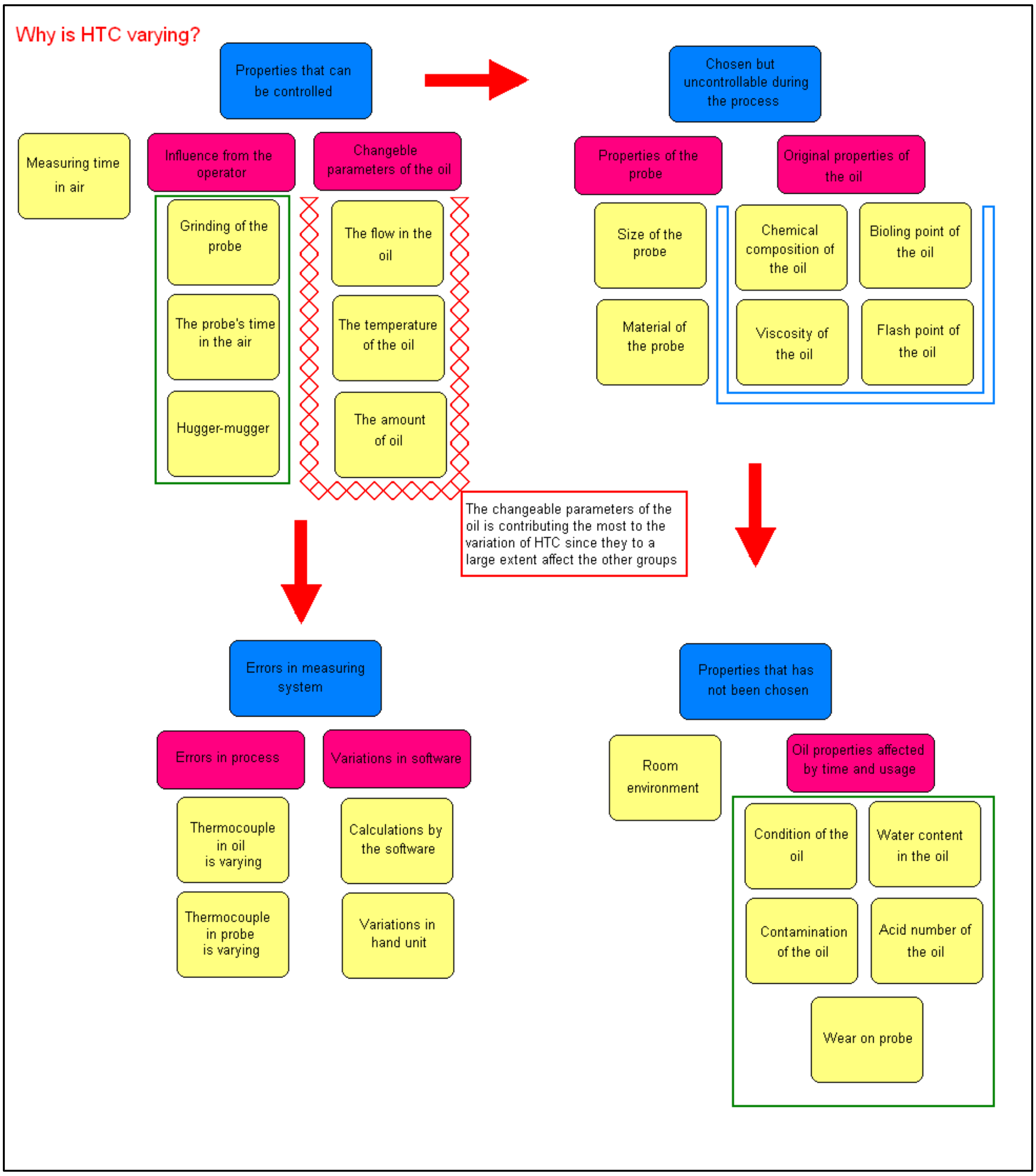

Figure 64 *Affinity-Interrelationship Method for the project.*

Fishbone Diagram

The fishbone diagram shows the causes for a problem or effect. Measurements, Operator, Environment, Oil and Probe are all major factors affecting the variation of HTC. The sub-causes are connected to the major causes with small arrows.

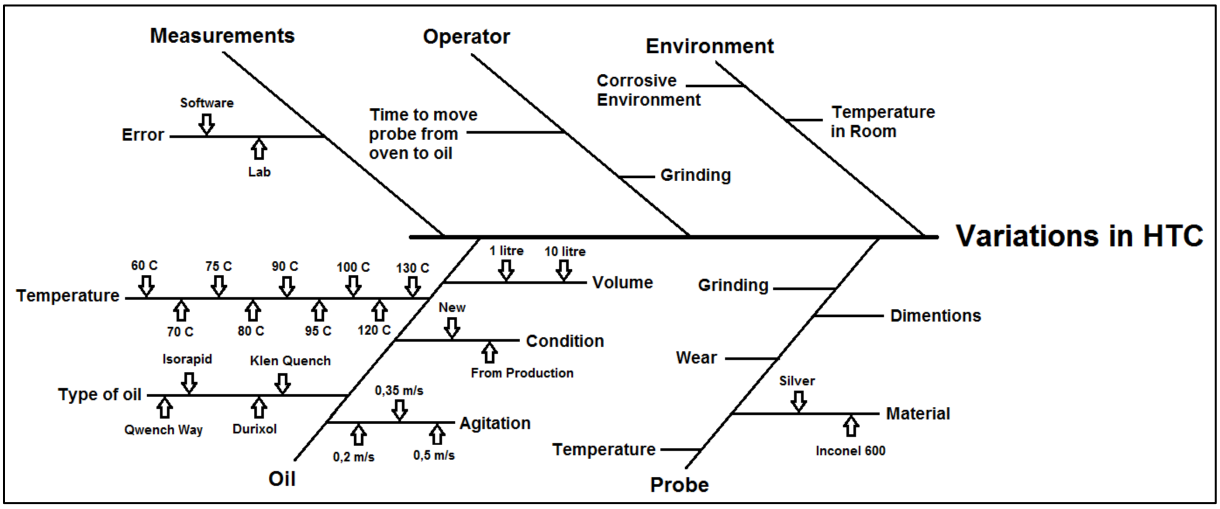

Figure 65 *Fishbone diagram showing what is causing a variation in Heat Transfer Coefficient*

11 Appendix B

Here the remaining cooling, cooling rate and HTC curves will be shown.

Temperature

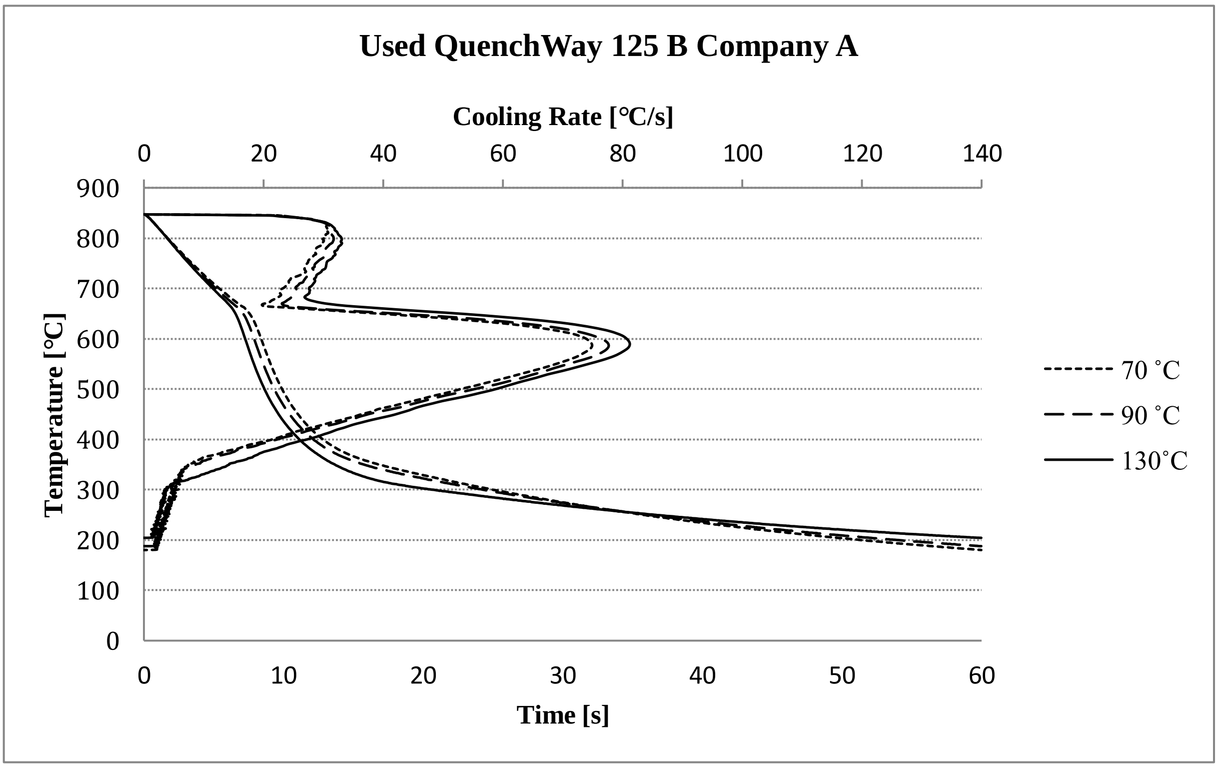

Figure 66 *Cooling and cooling rate curve showing the temperature dependence for 1 litre QuenchWay 125 B from Company A without agitation.*

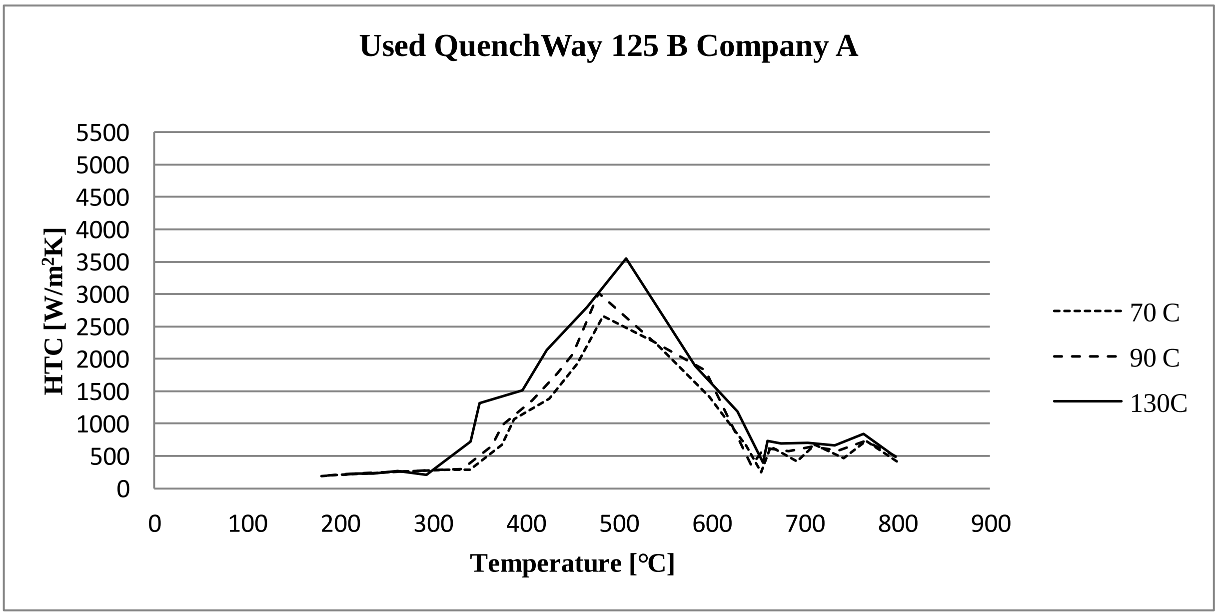

Figure 67 *Heat Transfer Coefficient curve showing temperature dependence for 1 litre QuenchWay 125 B from Company A without agitation.*

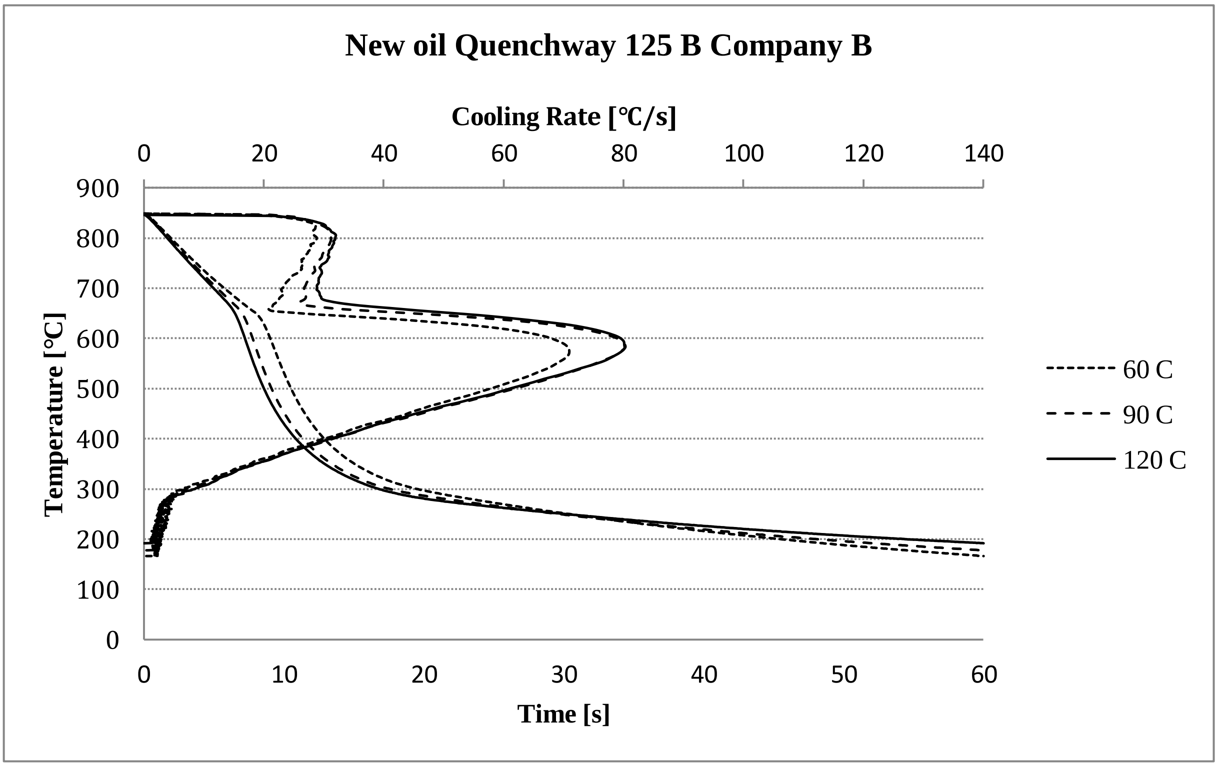

Figure 68 *Cooling and cooling rate curve showing the temperature dependence for 1 litre QuenchWay 125 B from Company B without agitation.*

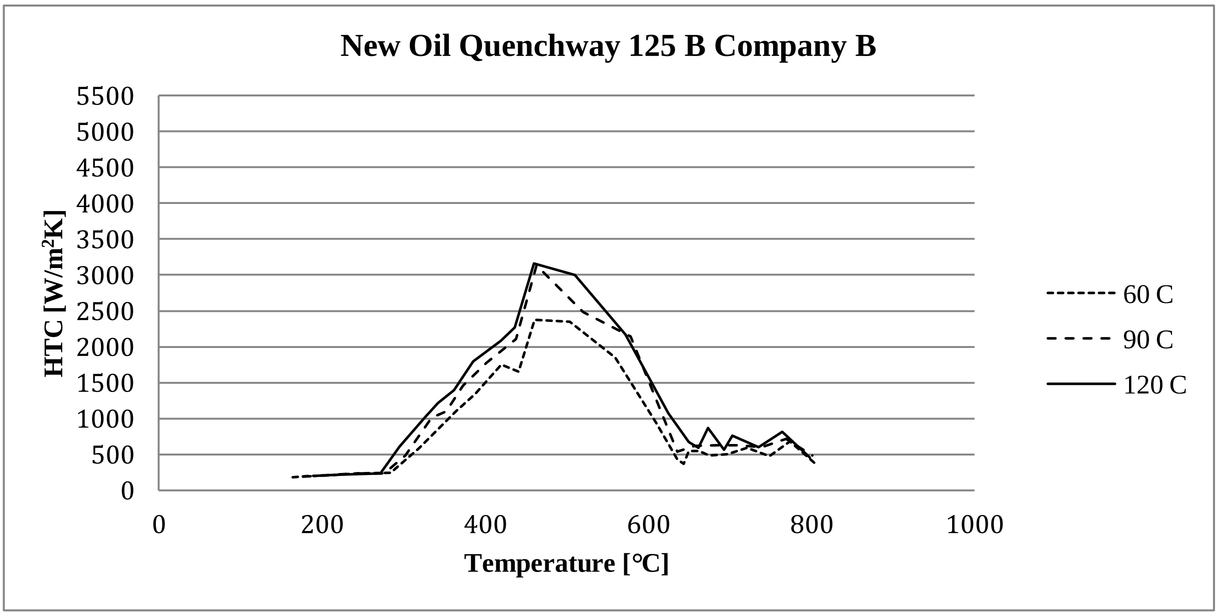

Figure 69 *Heat Transfer Coefficient curve showing temperature dependence for 1 litre QuenchWay 125 B from Company B without agitation.*

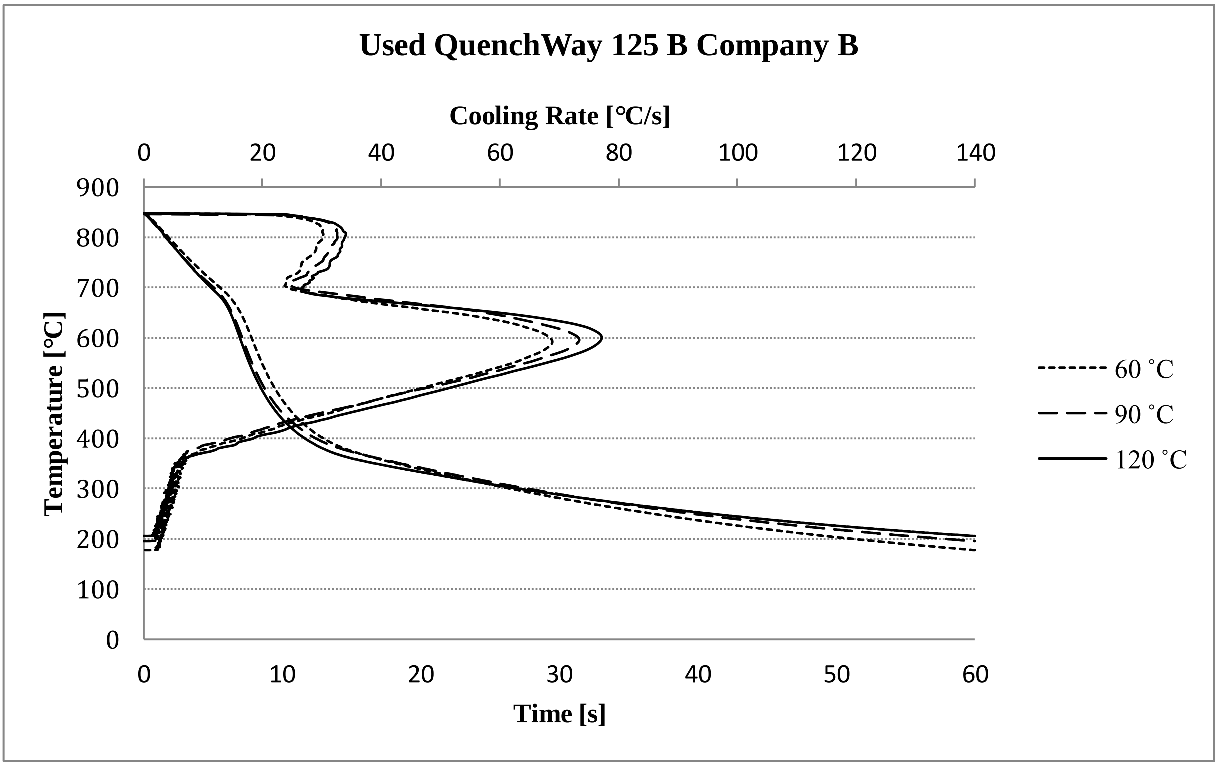

Figure 70 *Heat Transfer Coefficient curve showing temperature dependence for 1 litre QuenchWay 125 B from Company B without agitation.*

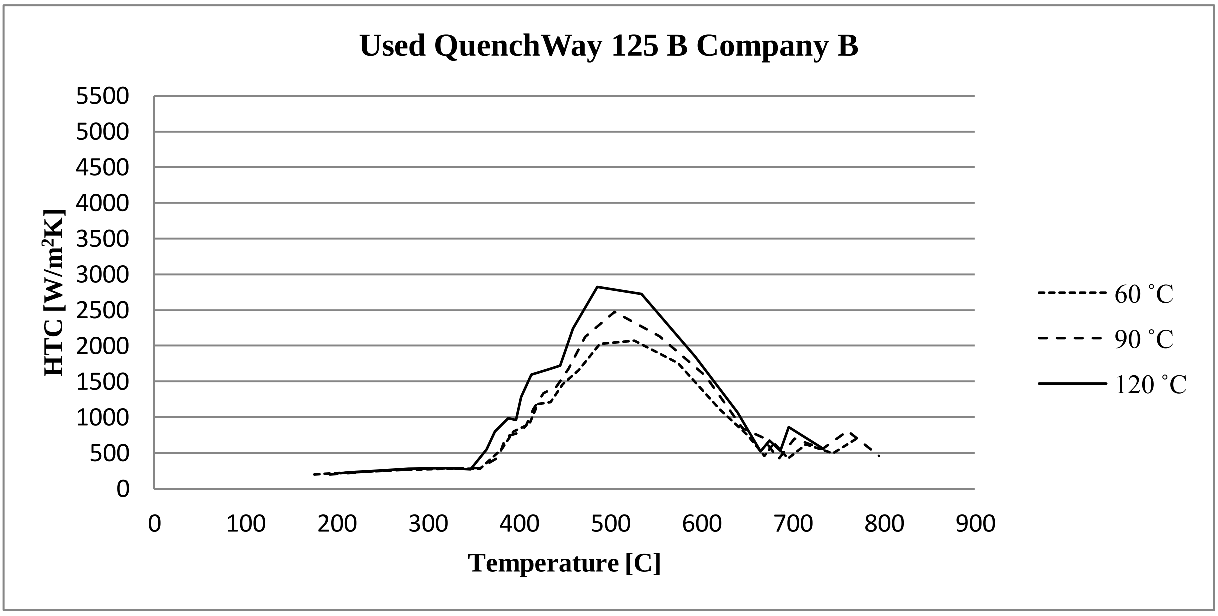

Figure 71 *Heat Transfer Coefficient curve showing temperature dependence for 1 litre QuenchWay 125 B from Company B without agitation.*

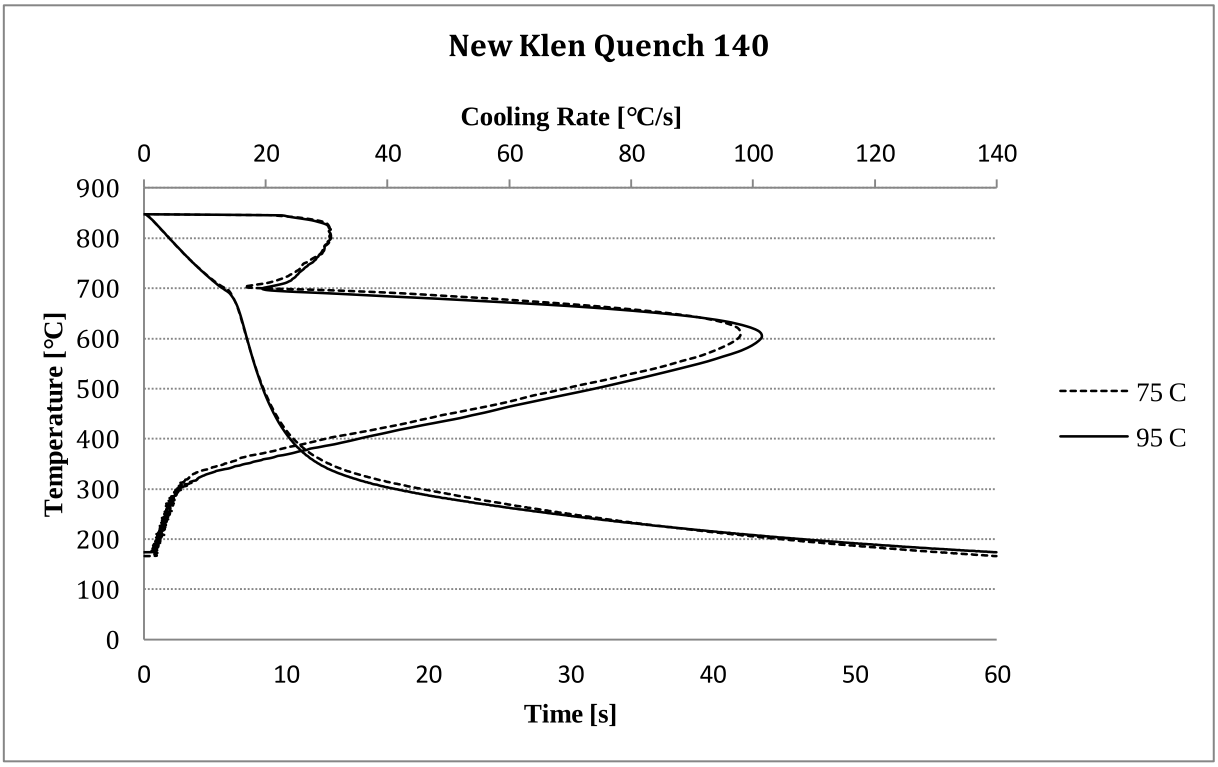

Figure 72 *Cooling and cooling rate curve showing the temperature dependence for 1 litre Klen Quench 140 from Company C without agitation.*

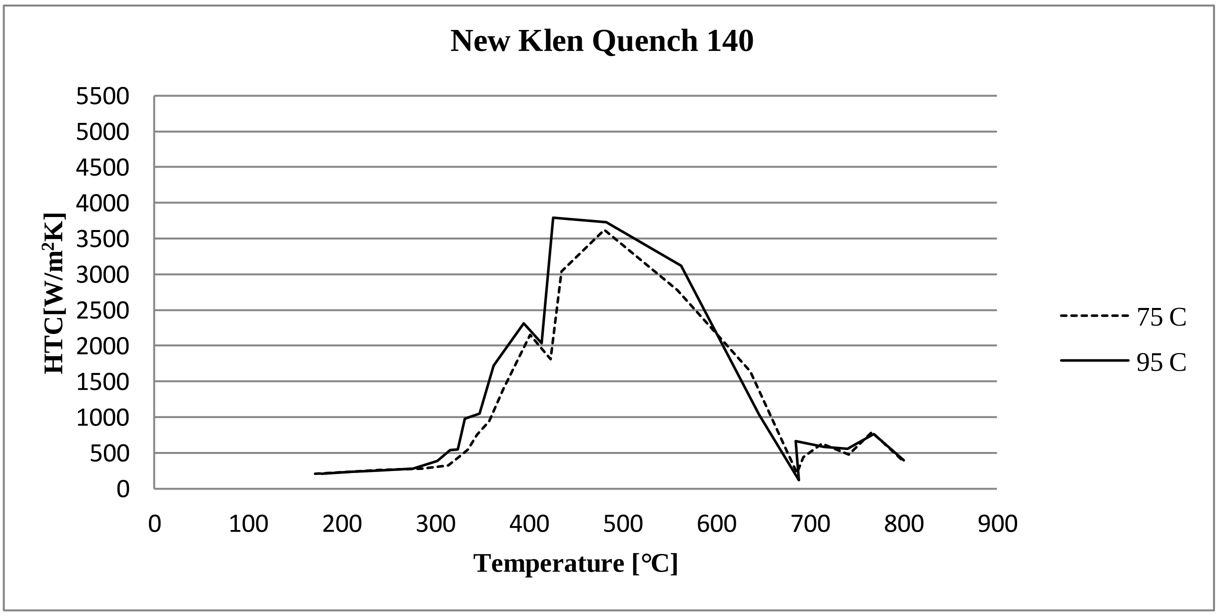

Figure 73 *Heat Transfer Coefficients curve showing temperature dependence for 1 litre Klen Quench 140 from Company C without agitation.*

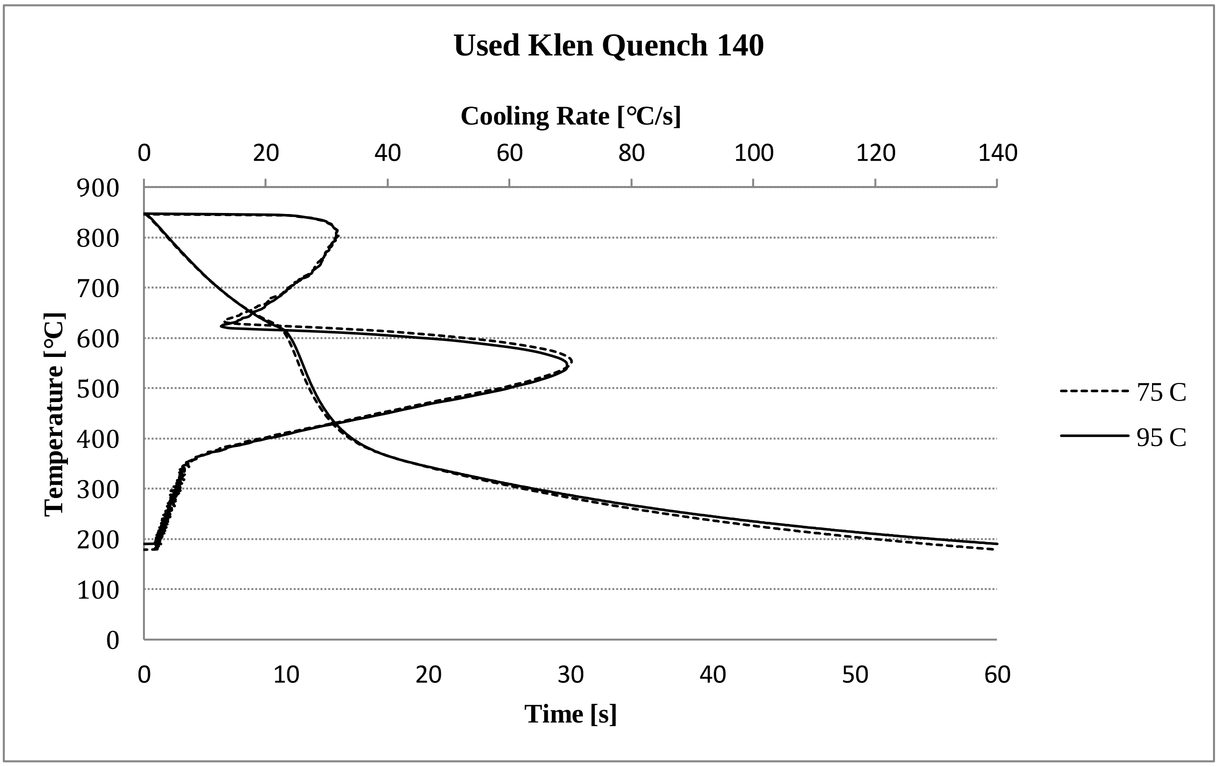

Figure 74 *Cooling and cooling rate curve showing the temperature dependence for 1 litre Klen Quench 140 from Company C without agitation.*

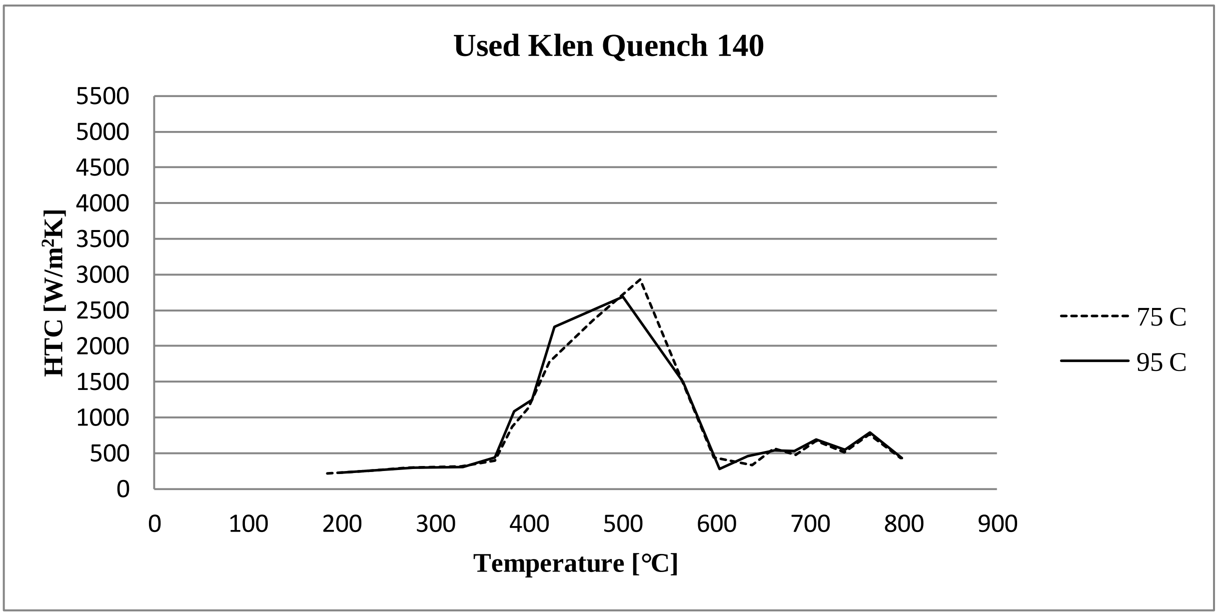

Figure 75 *Heat Transfer Coefficients curve showing temperature dependence for 1 litre Klen Quench 140 from Company C without agitation.*

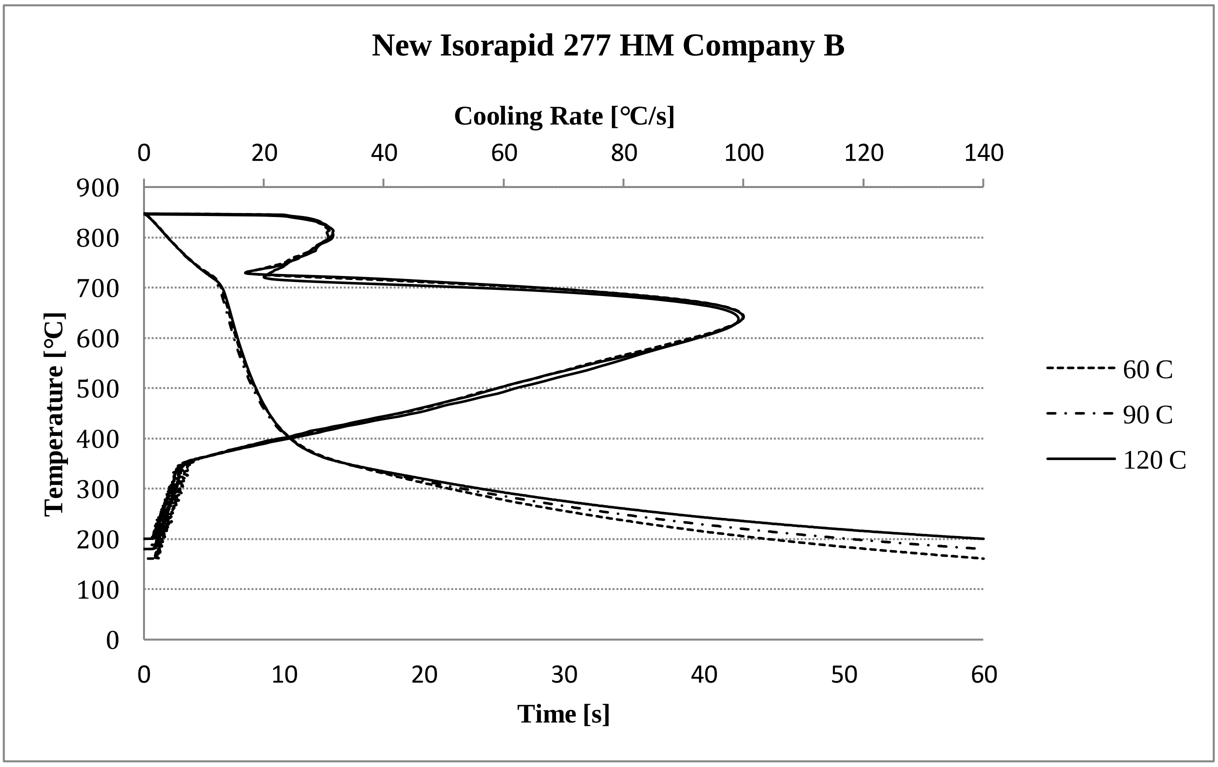

Figure 76 *Cooling and cooling rate curve showing the temperature dependence for 1 litre Isorapid 277 HM from Company B without agitation.*

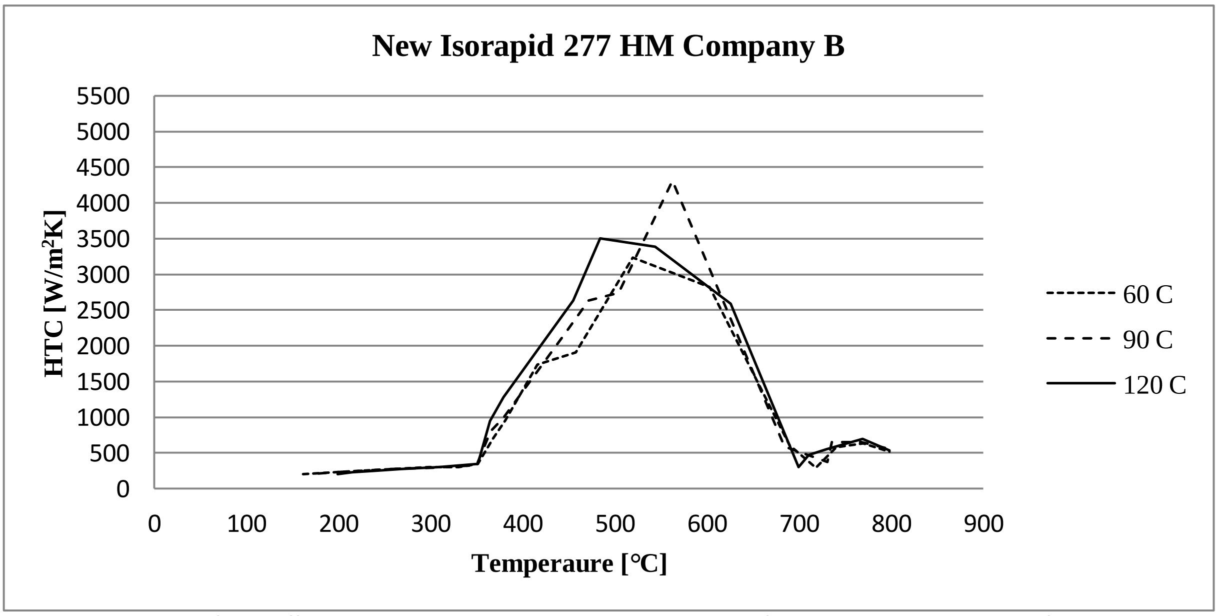

Figure 77 *Heat Transfer Coefficients curve showing temperature dependence for 1 litre Isorapid 277 HM from Company B without agitation.*

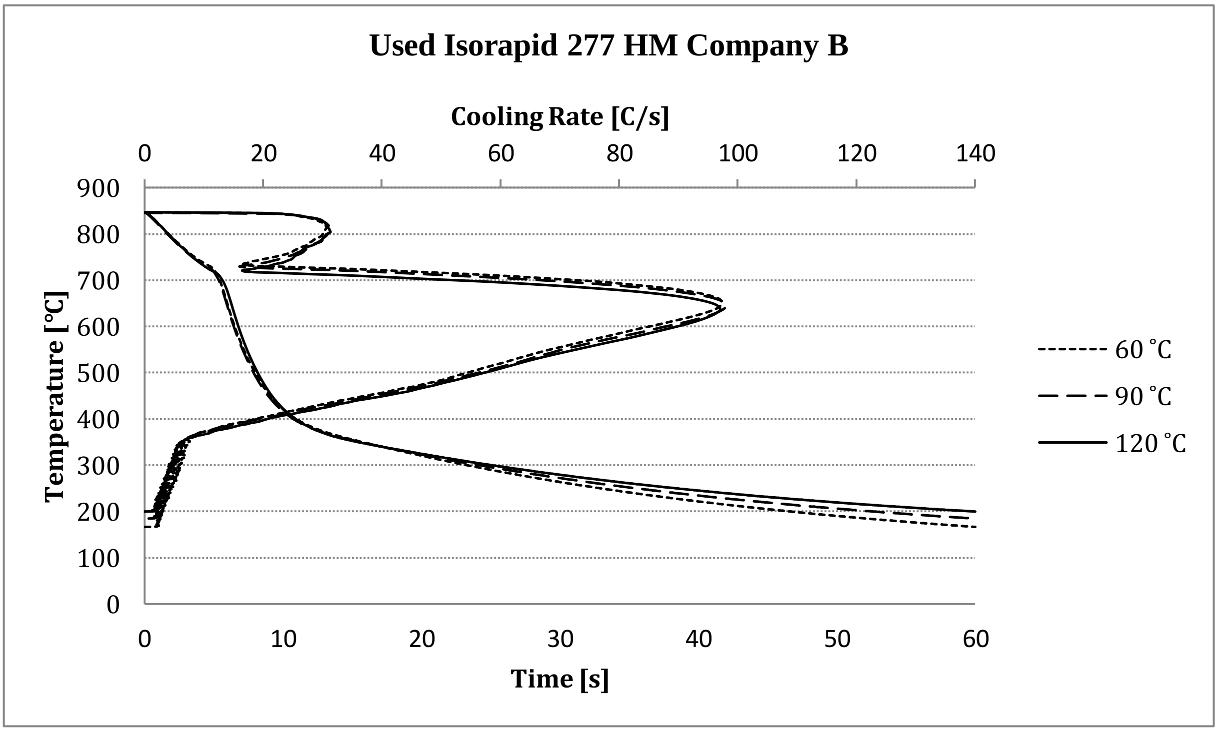

Figure 78 *Cooling and cooling rate curve showing the temperature dependence for 1 litre Isorapid 277 HM from Company B without agitation.*

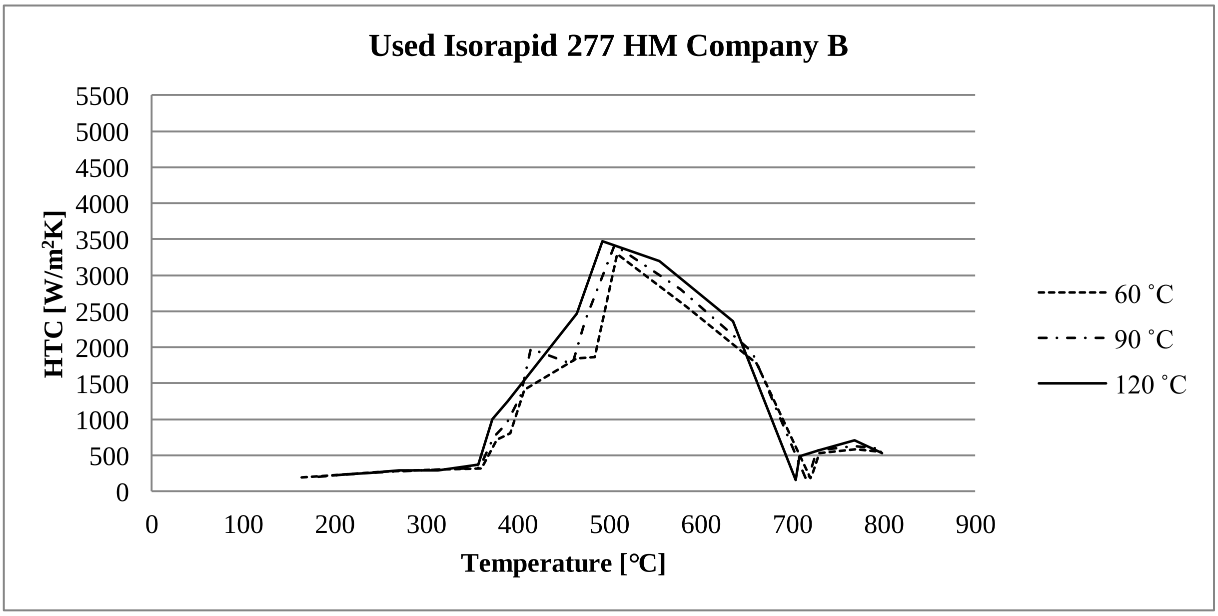

Figure 79 *Heat Transfer Coefficients curve showing temperature dependence for 1 litre Isorapid 277 HM from Company B without agitation.*

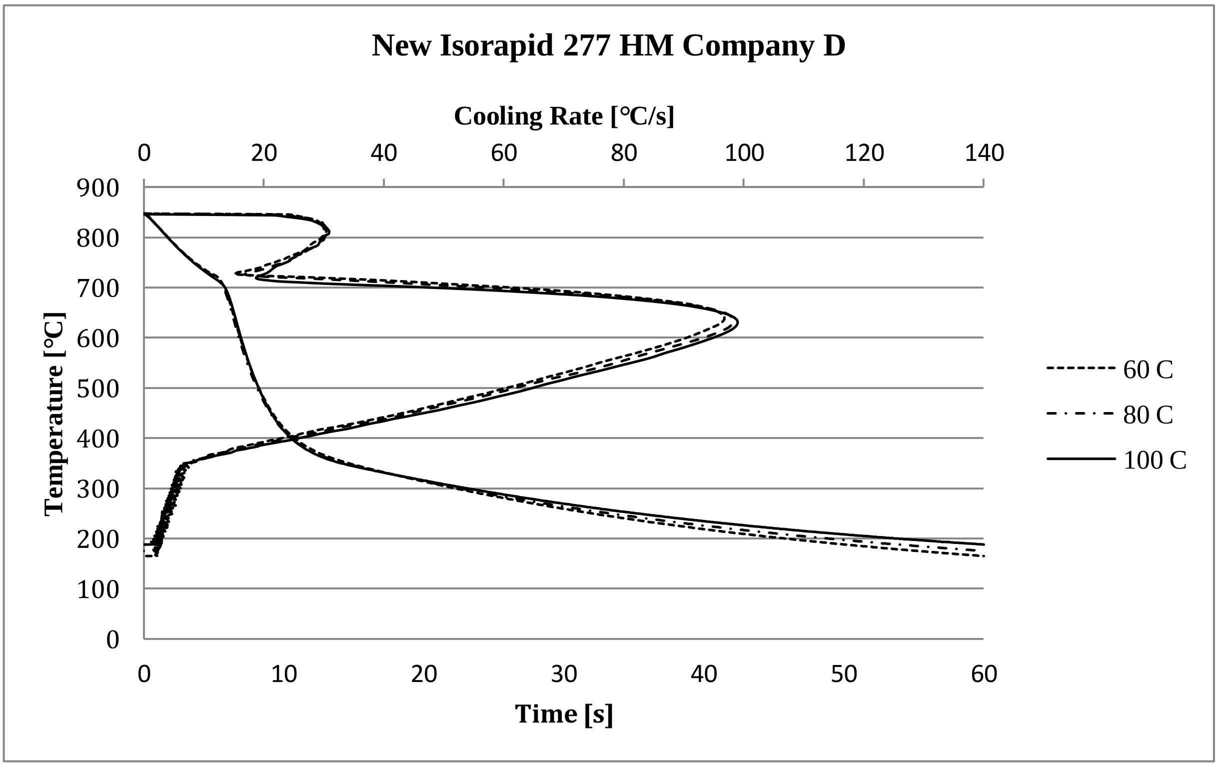

Figure 80 *Cooling and cooling rate curve showing the temperature dependence for 1 litre Isorapid 277 HM from Company D without agitation.*

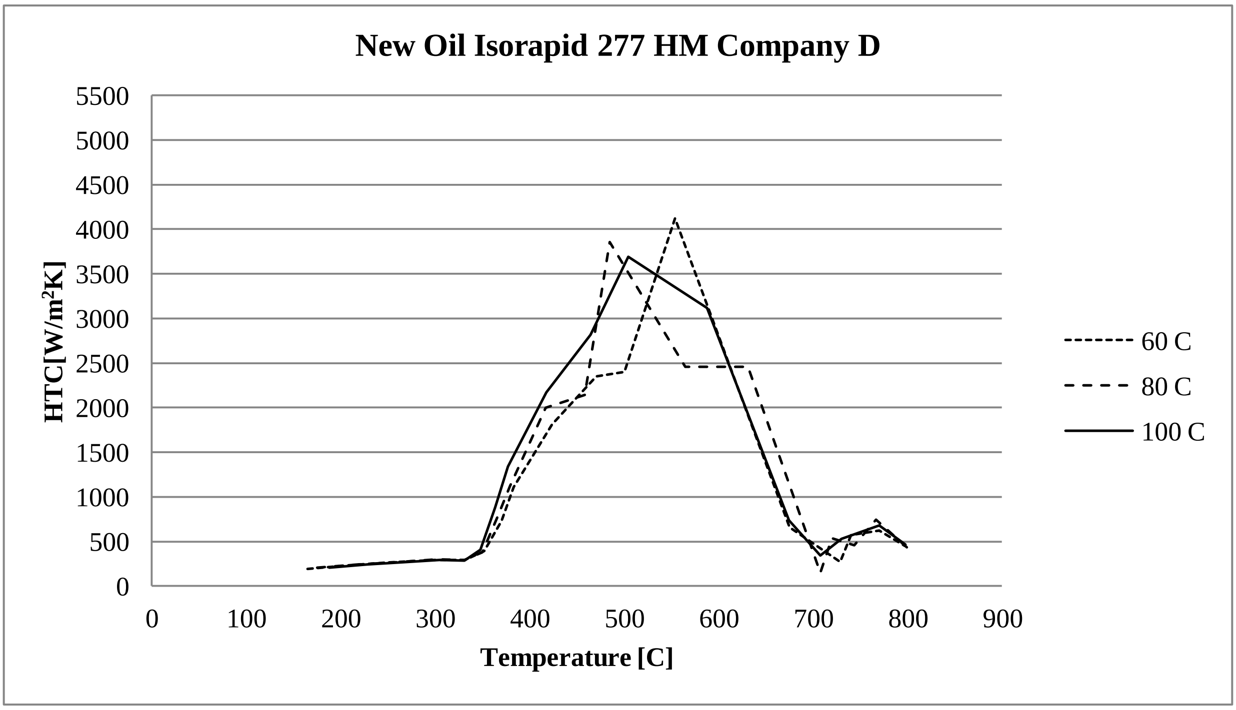

Figure 81 *Heat Transfer Coefficients curve showing temperature dependence for 1 litre Isorapid 277 HM from Company D without agitation.*

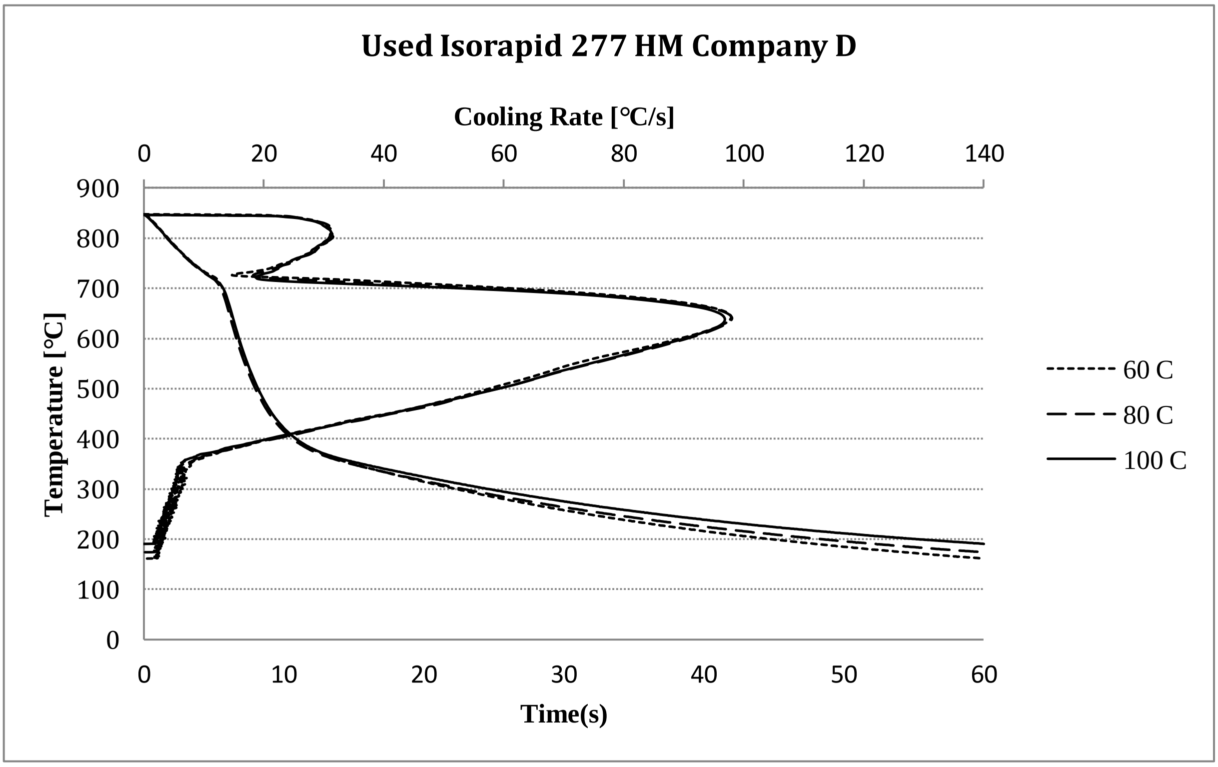

Figure 82 *Cooling and cooling rate curve showing the temperature dependence for 1 litre Isorapid 277 HM from Company D without agitation.*

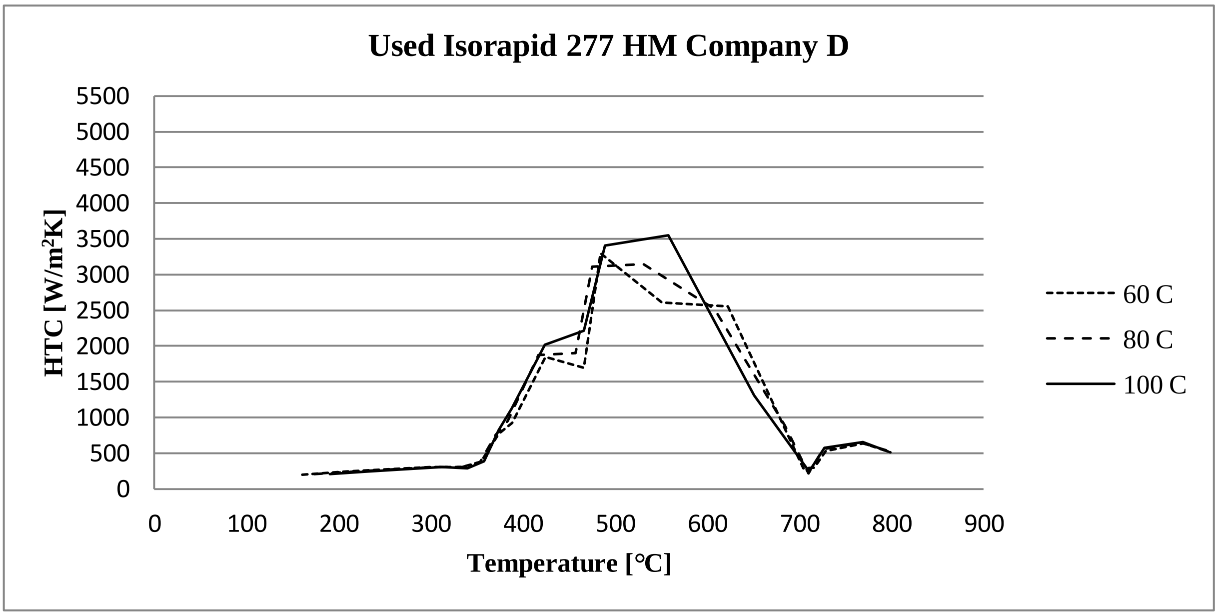

Figure 83 *Heat Transfer Coefficients curve showing temperature dependence for 1 litre Isorapid 277 HM from Company D without agitation.*

Agitation

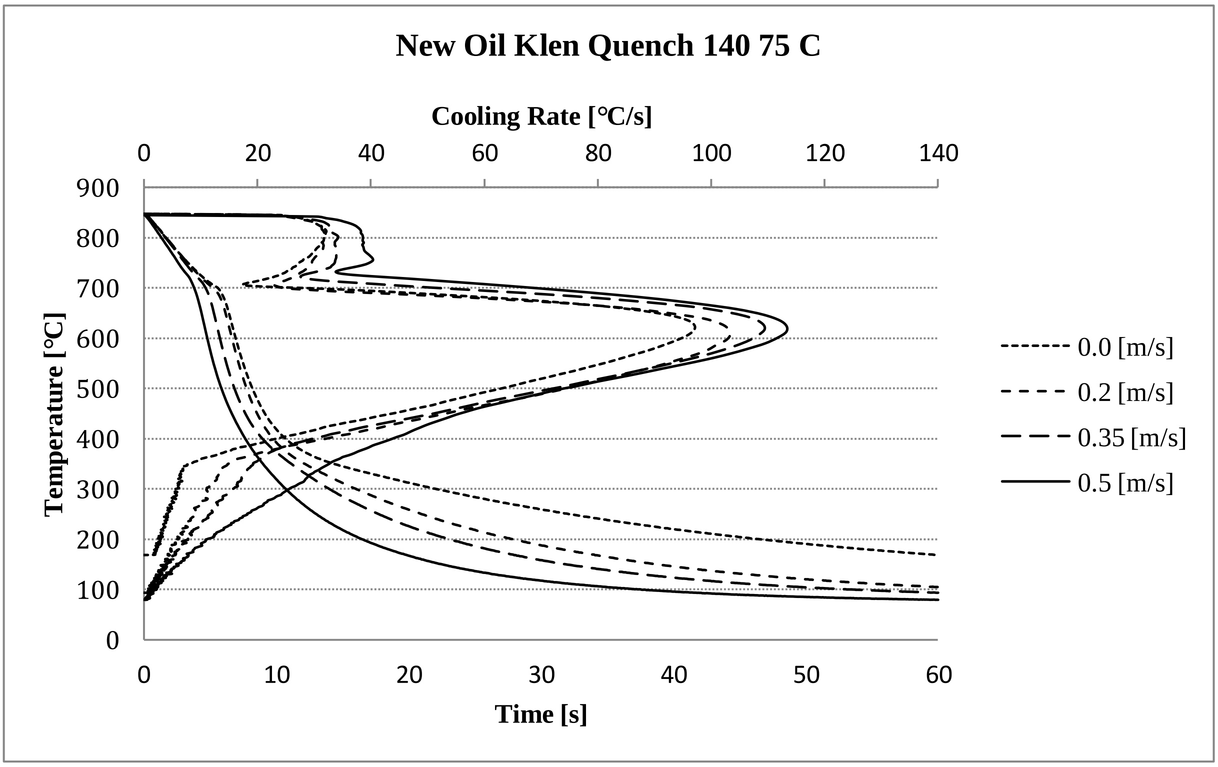

Figure 84 *Cooling and cooling rate curve showing the temperature dependence for 10 litres Klen Quench 140 from Company C at 75°C.*

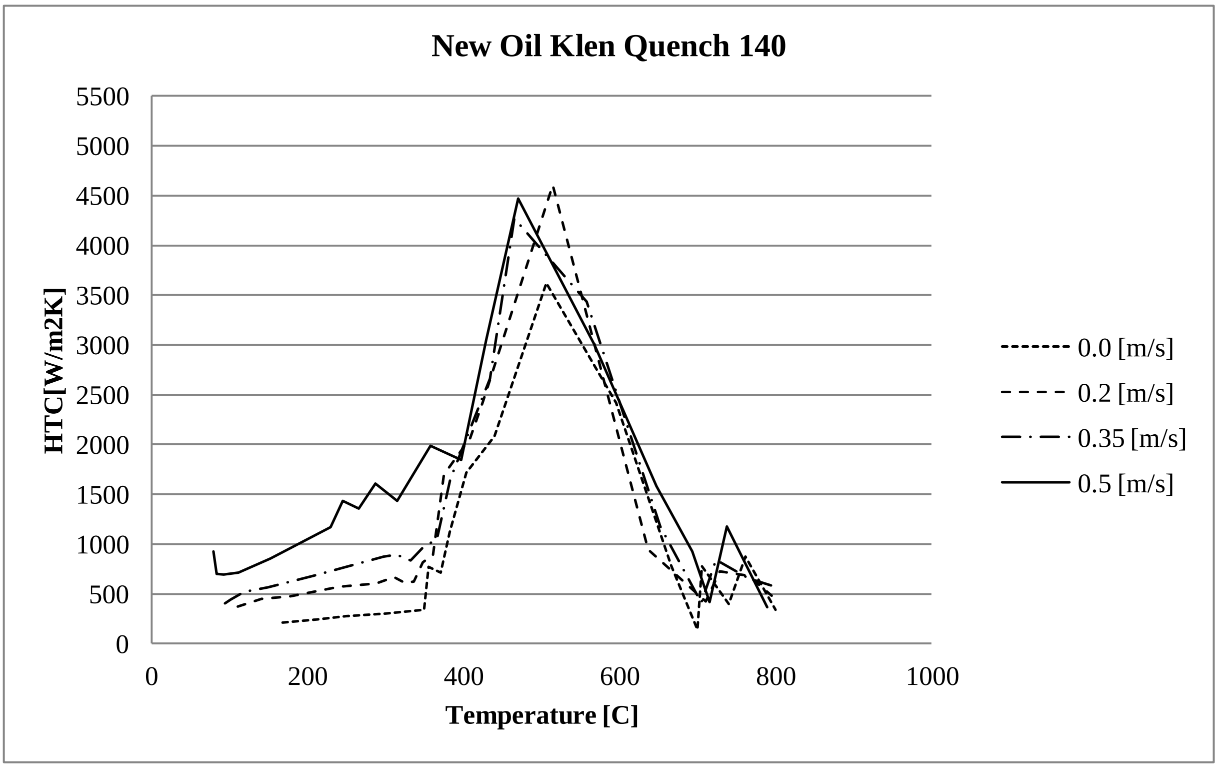

Figure 85 *Heat Transfer Coefficients curve showing temperature dependence for 10 liters Klen Quench 140 from Company C at 75°C.*

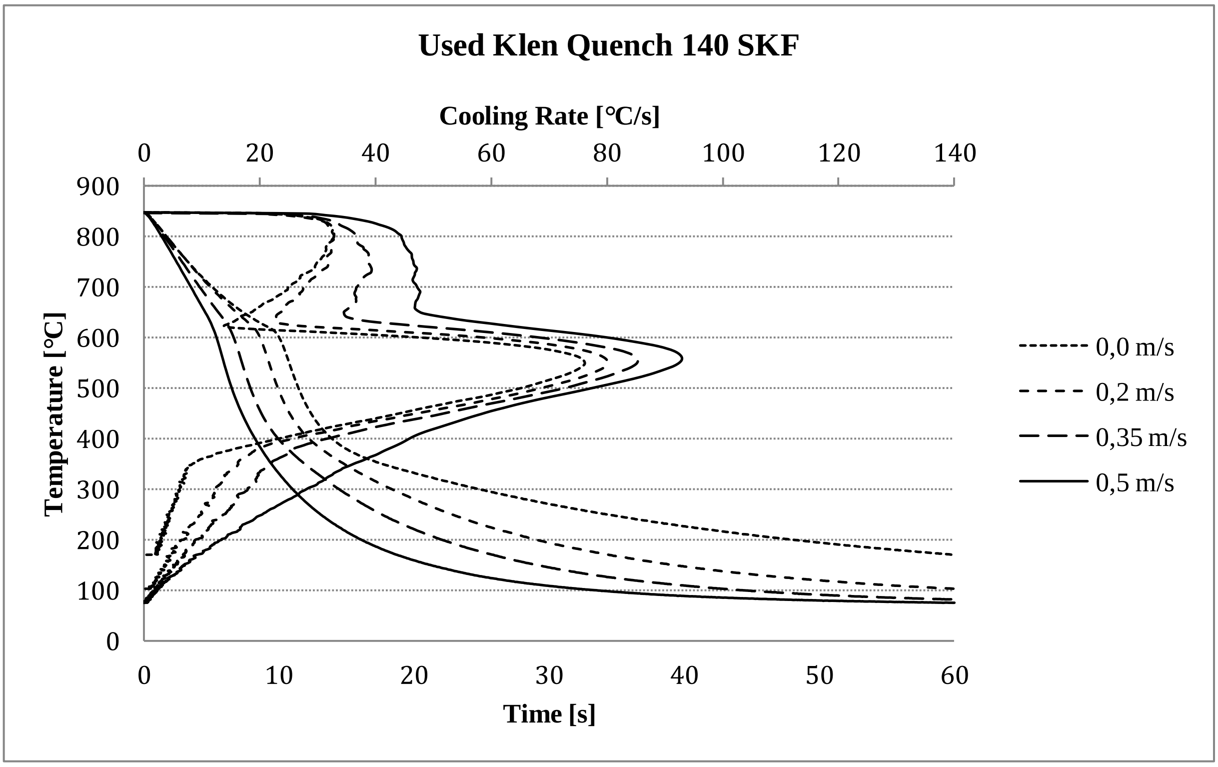

Figure 86 *Cooling and cooling rate curve showing the temperature dependence for 10 litres Klen Quench 140 from Company C at 75°C.*

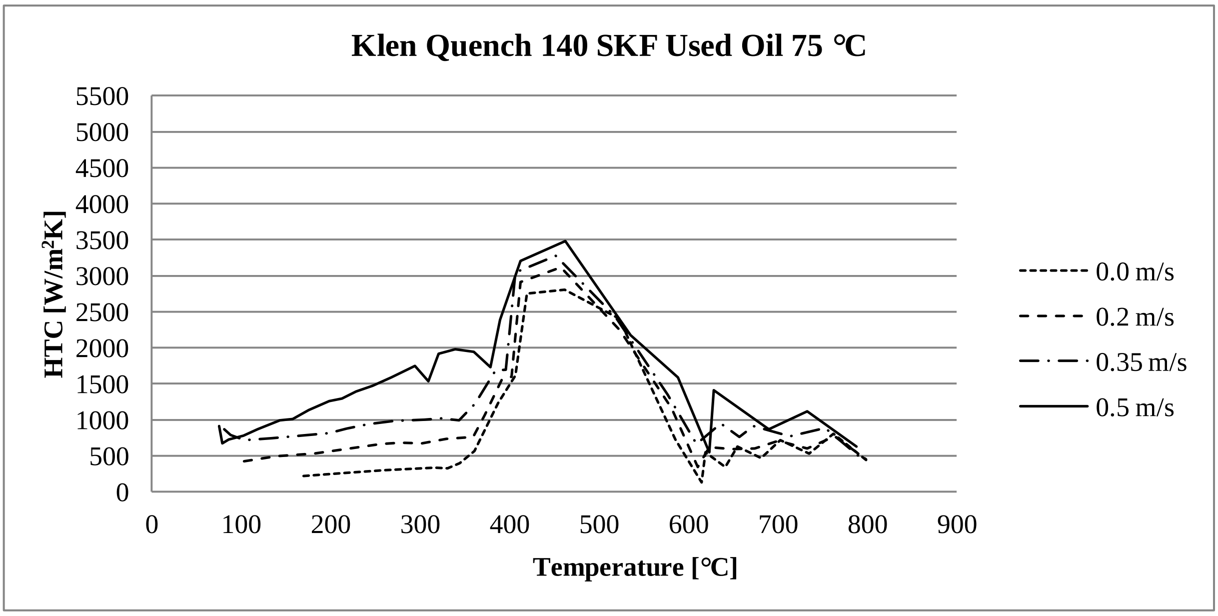

Figure 87 *Heat Transfer Coefficients curve showing temperature dependence for 10 litres Klen Quench 140 from Company C at 75°C.*

Condition

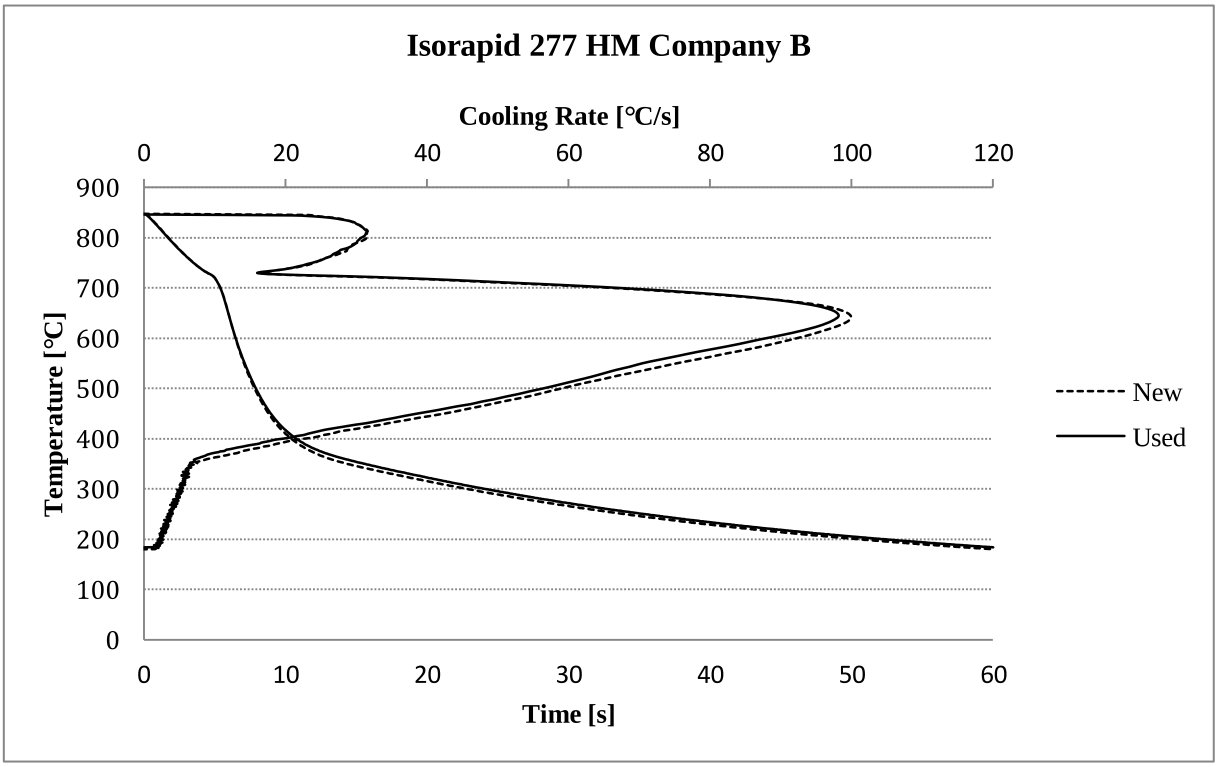

Figure 88 *Cooling and cooling rate curve showing the condition dependence for 1 litre Isorapid 277 HM from Company B at 90°C.*

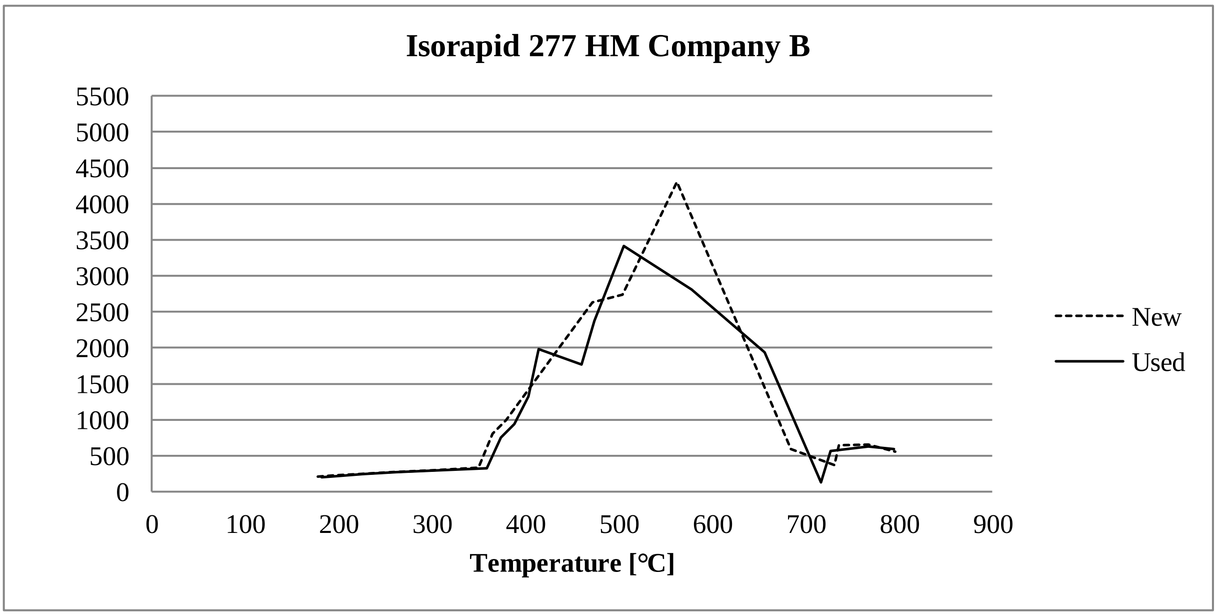

Figure 89 *Heat Transfer Coefficients curve showing condition dependence for 1 litre Isorapid 277 HM from Company B at 90°C.*

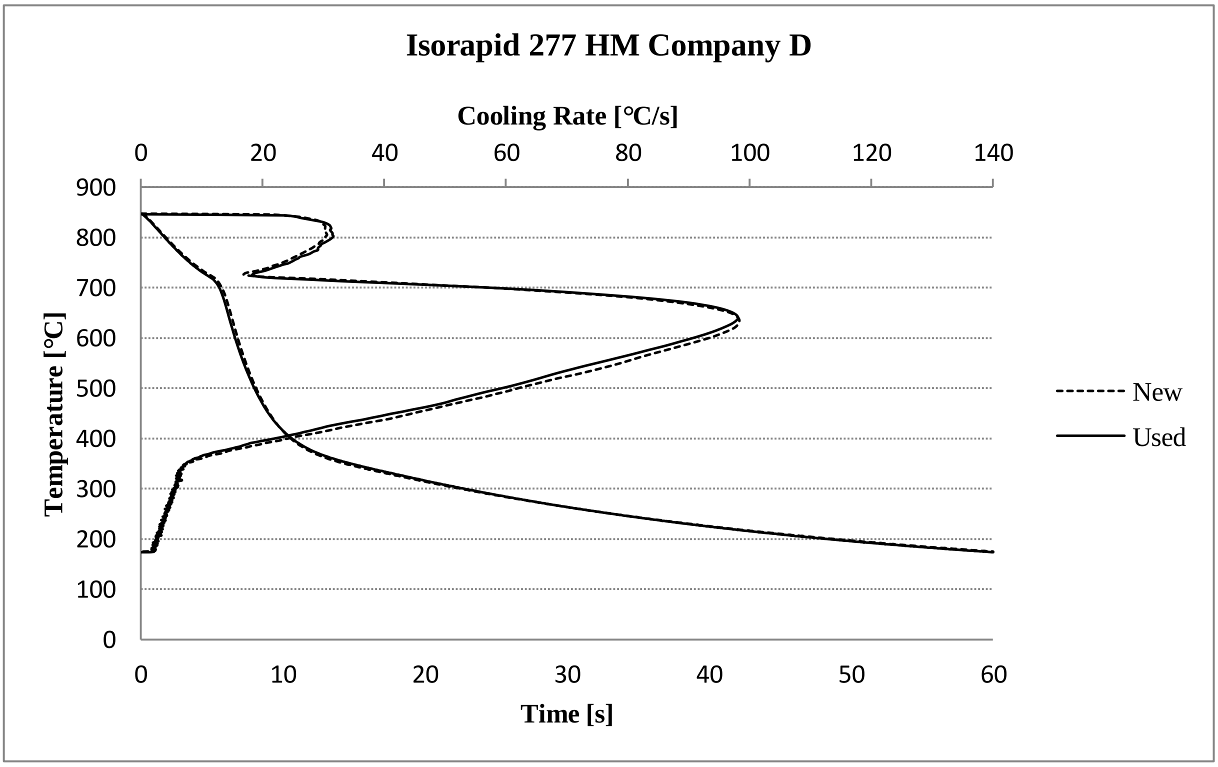

Figure 90 *Cooling and cooling rate curve showing the condition dependence for 1 litre Isorapid 277 HM from Company D at 80°C.*

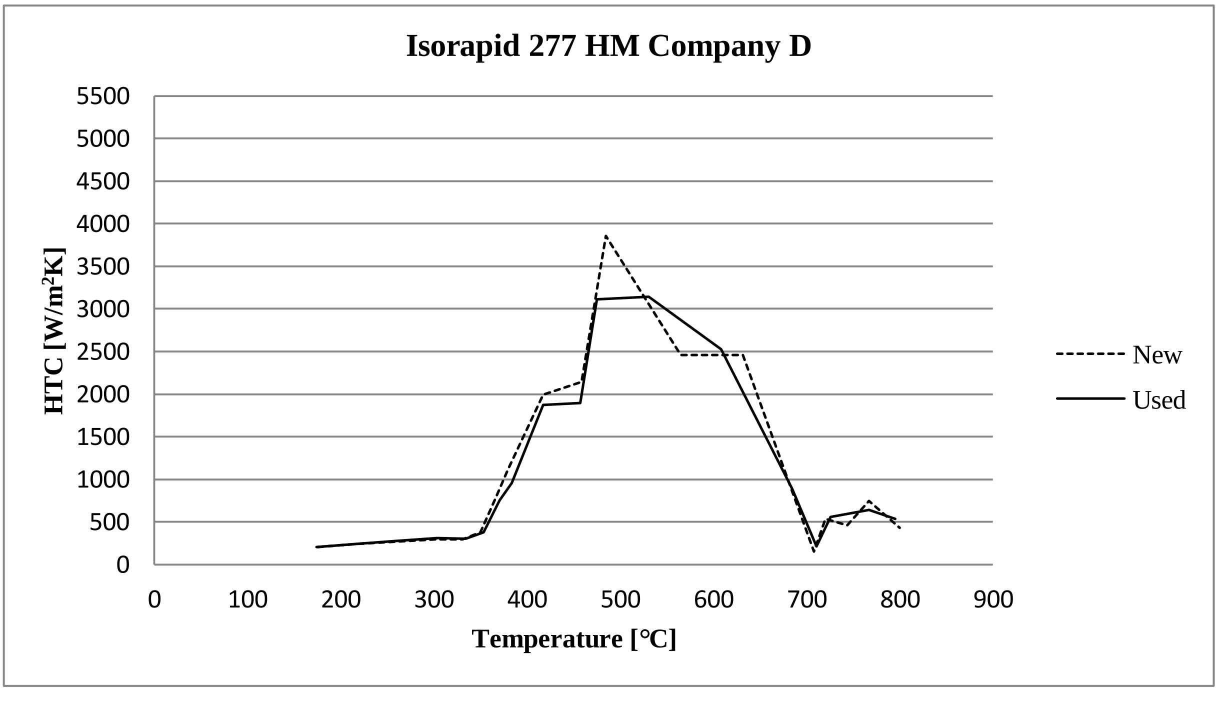

Figure 91 *Heat Transfer Coefficients curve showing condition dependence for 1 litre Isorapid 277 HM from Company D at 80°C.*

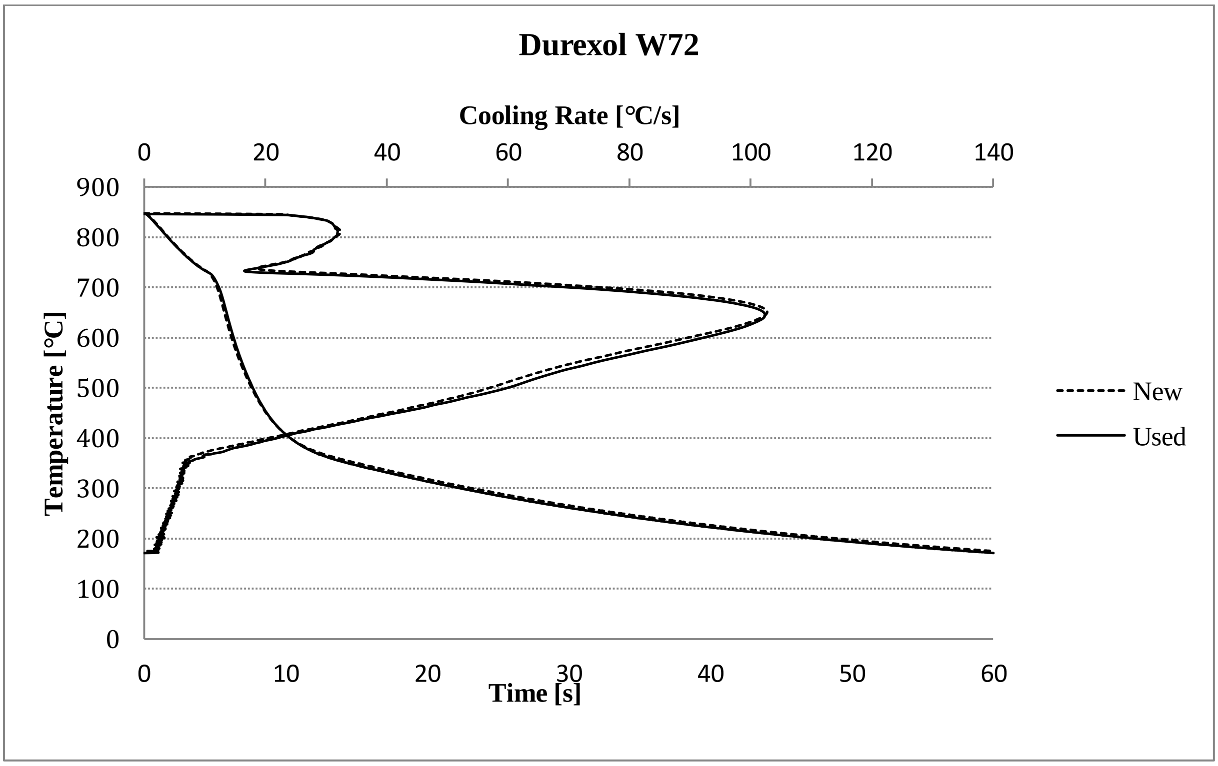

Figure 92 *Cooling and cooling rate curve showing the condition dependence for 1 litre Durixol W72 from Company E at 80°C.*

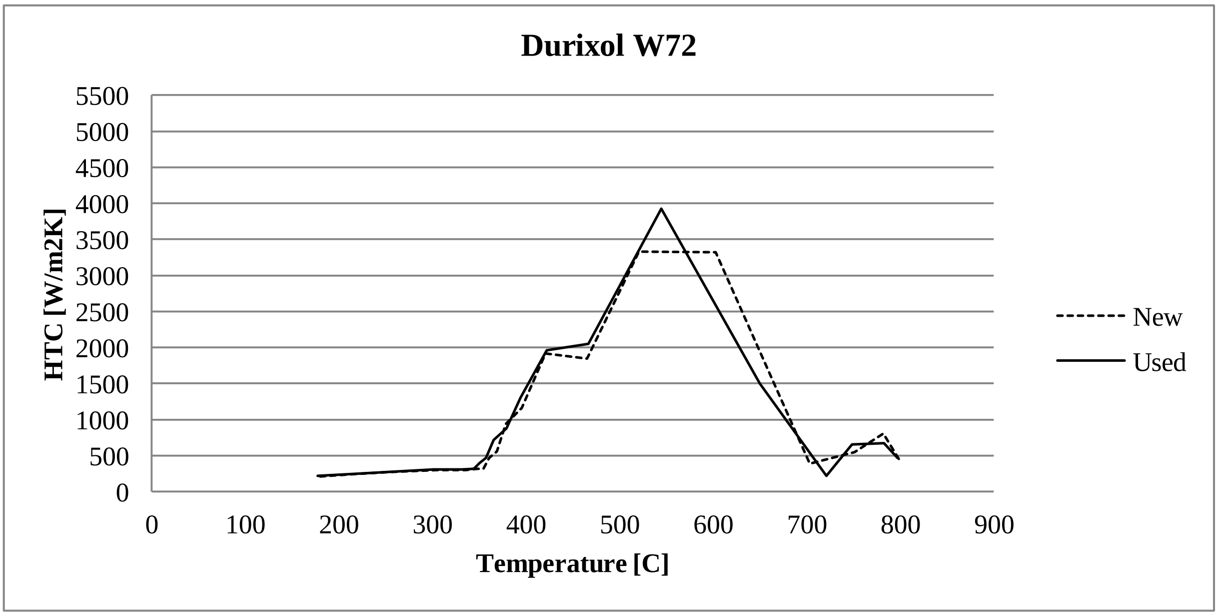

Figure 93 *Heat Transfer Coefficients curve showing condition dependence for 1 litre Durixol W72 from Company E at 80°C.*

Volume

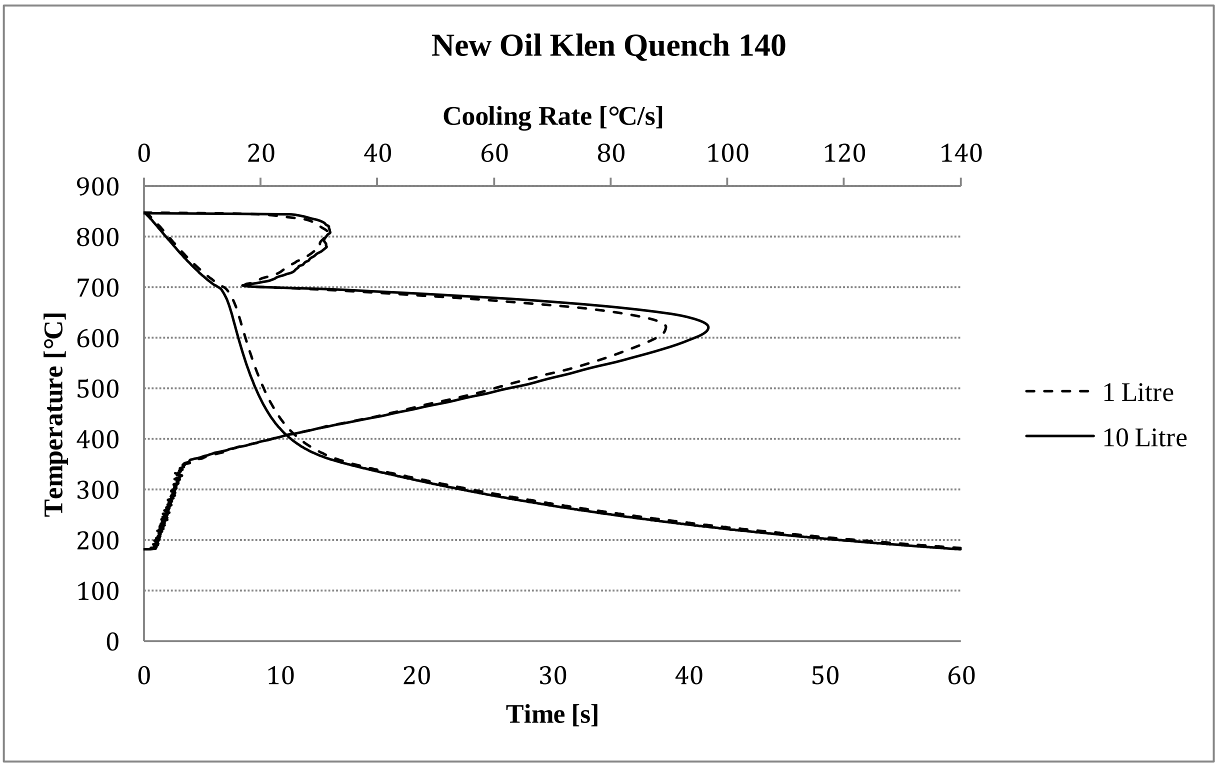

Figure 94 *Cooling and cooling rate curve showing the volume dependence for Klen Quench 140 from Company C at 75°C without agitation.*

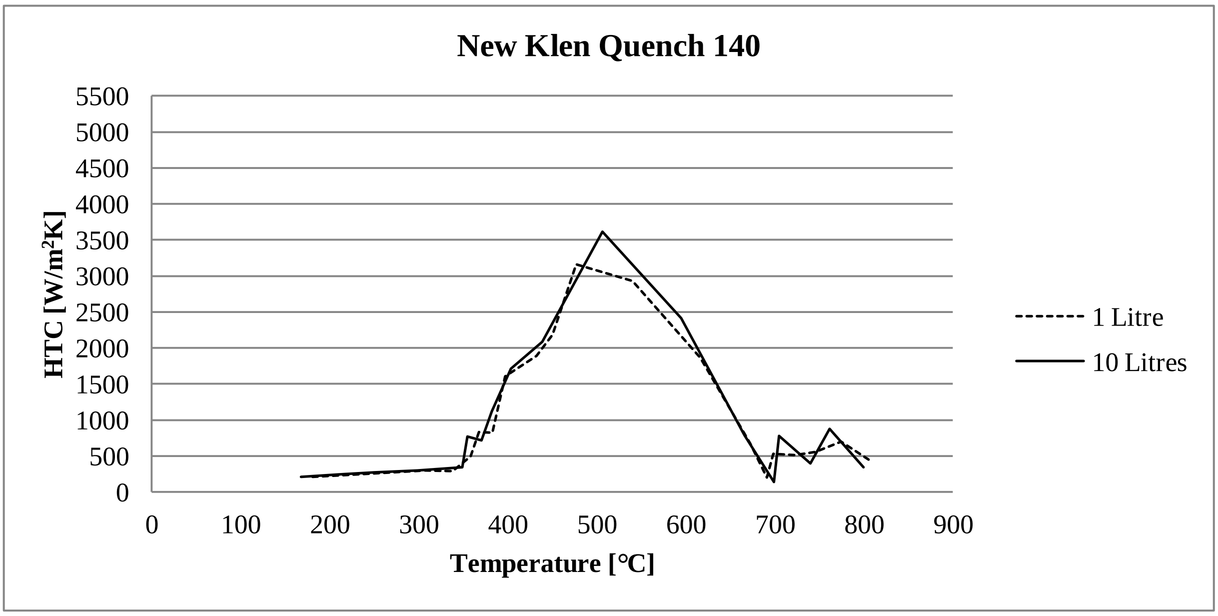

Figure 95 *Heat Transfer Coefficients curve showing the volume dependence for Klen Quench 140 from Company C at 75°C without agitation.*

12 Appendix C

Main Effects plots for the performed General Linear Models.

QuenchWay 125 B

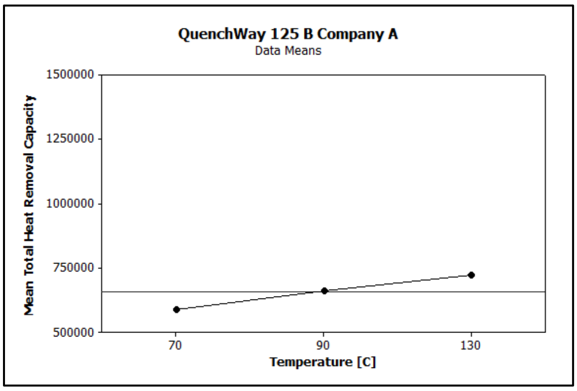

Figure 96 *Total Heat Removal Capacity depending on temperature for 1 litre new and used QuenchWay 125 B from Company A without agitation.*

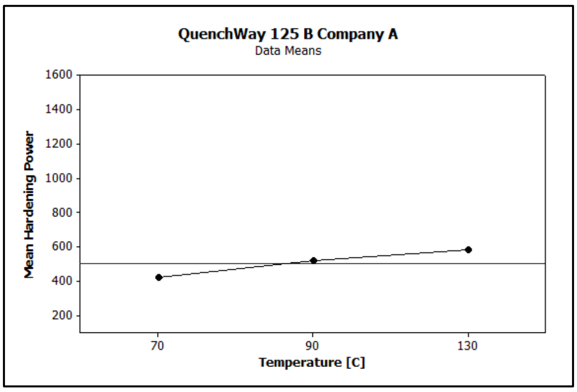

Figure 97 *Hardening Power depending on temperature for 1 litre new and used QuenchWay 125 B from Company A without agitation.*

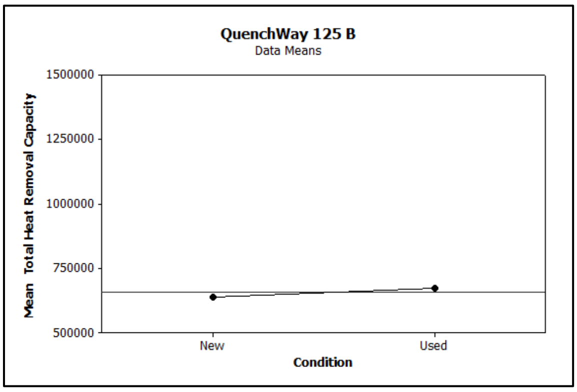

Figure 98 *Total Heat Removal Capacity depending on condition for 1 litre new and used QuenchWay 125 B from Company A without agitation.*

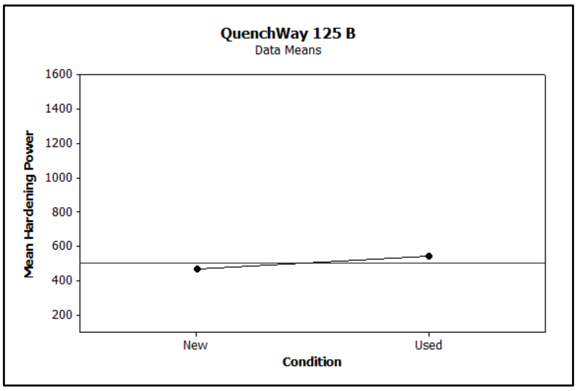

Figure 99 *Hardening Power depending on condition for 1 litre new and used QuenchWay 125 B from Company A without agitation.*

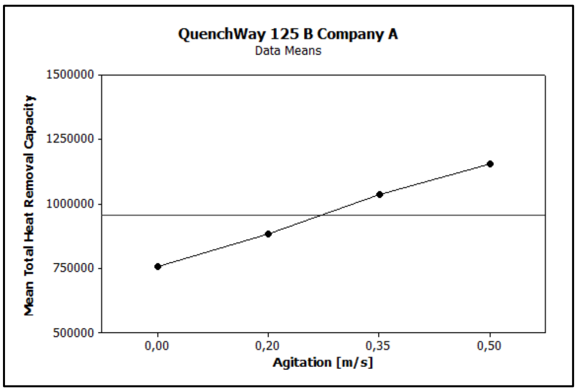

Figure 100 *Total Heat Removal Capacity depending on agitation for 10 litres new and used QuenchWay 125 B from Company A at 130°C.*

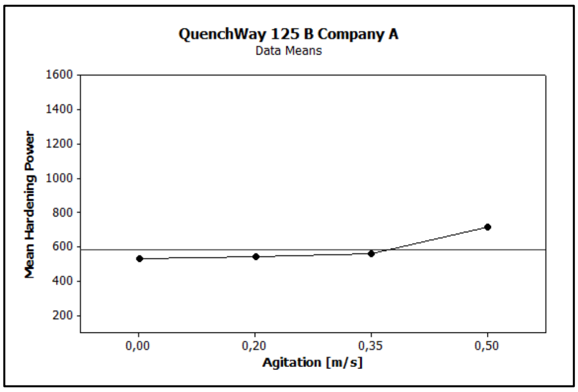

Figure 101 *Hardening Power depending on agitation for 10 litres new and used QuenchWay 125 B from Company A at 130°C.*

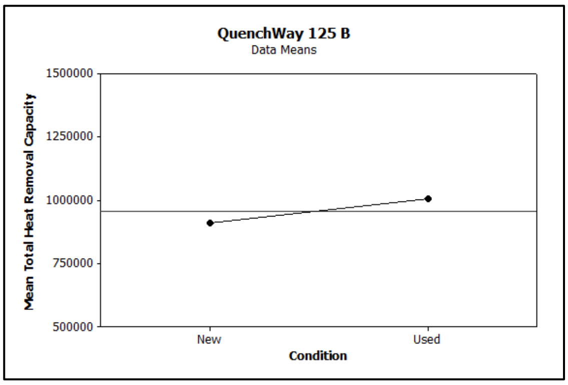

Figure 102 *Total Heat Removal Capacity depending on condition for 10 litres QuenchWay 125 B from Company A at 130°C with agitation.*

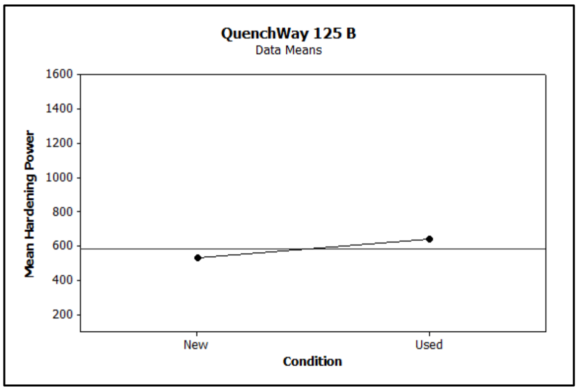

Figure 103 *Hardening Power depending on condition for 10 litres QuenchWay 125 B from Company A at 130°C with agitation.*

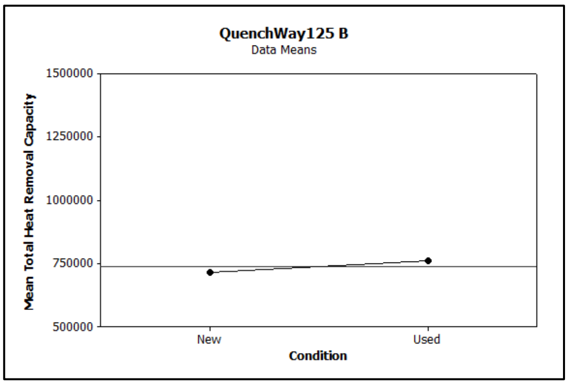

Figure 104 *Total Heat Removal Capacity depending on condition for 1 and 10 litres QuenchWay 125 B from Company A at 130°C without agitation.*

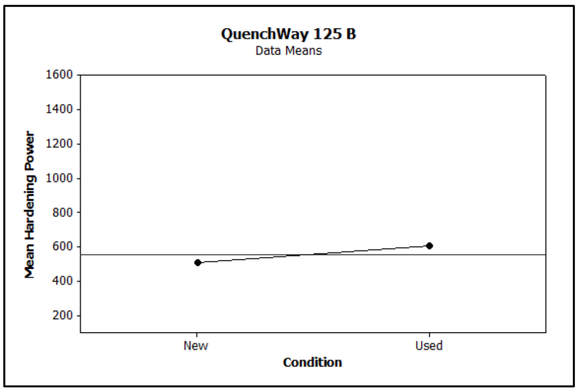

Figure 105 *Hardening Power depending on condition for 1 and 10 litres QuenchWay 125 B from Company A at 130°C without agitation.*

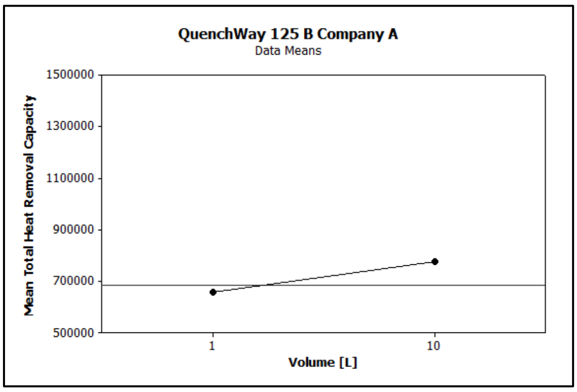

Figure 106 *Total Heat Removal Capacity depending on volume for new and used QuenchWay 125 B from Company A at 130°C without agitation.*

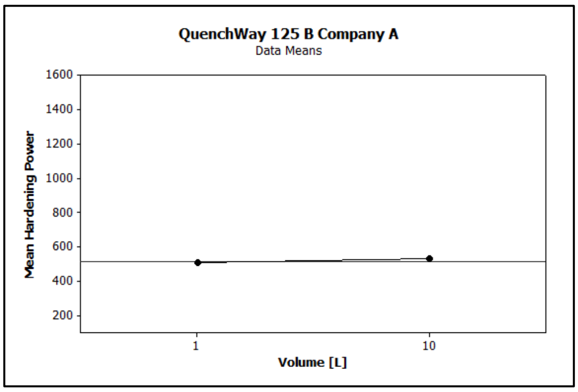

Figure 107 *Hardening Power depending on volume for new and used QuenchWay 125 B from Company A at 130°C without agitation.*

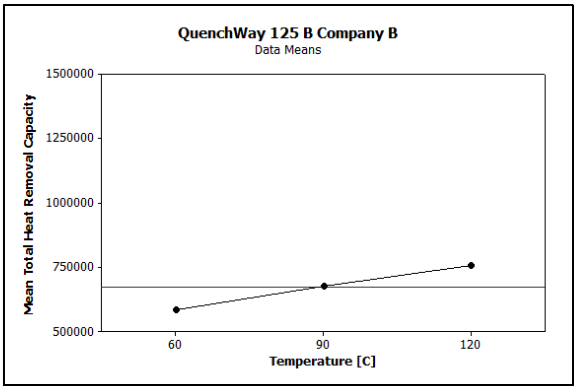

Figure 108 *Total Heat Removal Capacity depending on temperature for 1 litre new and used QuenchWay 125 B from Company B without agitation.*

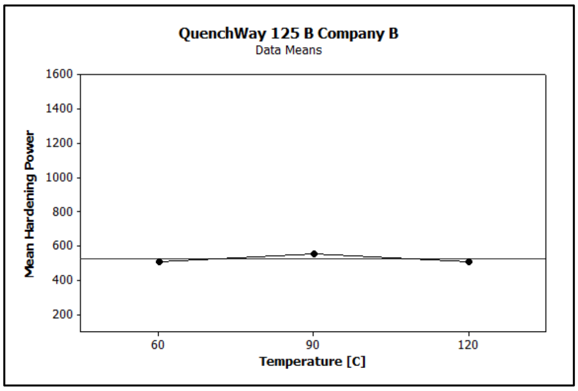

Figure 109 *Hardening Power depending on temperature for 1 litre new and used QuenchWay 125 B from Company B without agitation.*

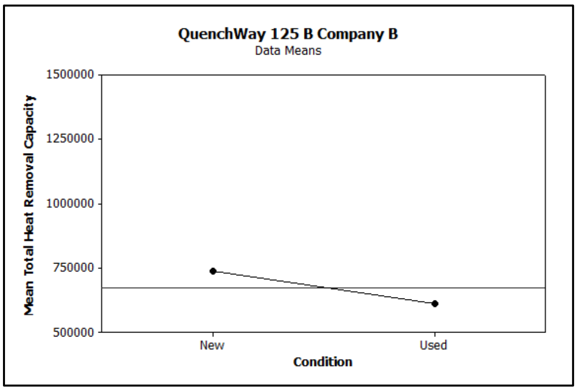

Figure 110 *Total Heat Removal Capacity depending on condition for 1 litre QuenchWay 125 B from Company B without agitation.*

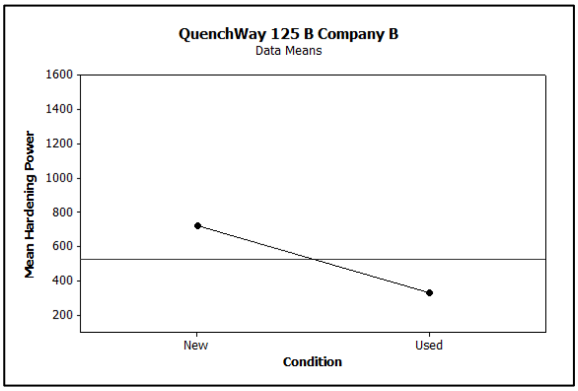

Figure 111 *Hardening Power depending on condition for 1 litre QuenchWay 125 B from Company B without agitation.*

Klen Quench 140

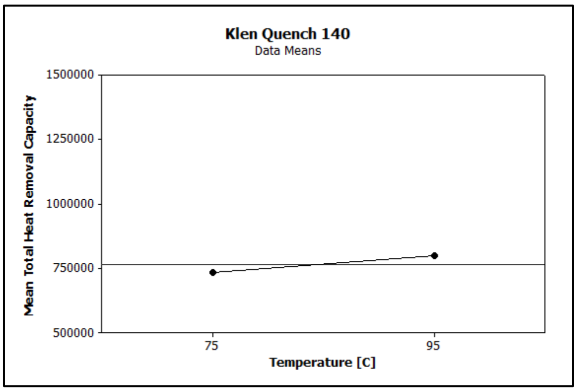

Figure 112 *Total Heat Removal Capacity depending on temperature for 1 litre new and used Klen Quench 140 from Company C without agitation.*

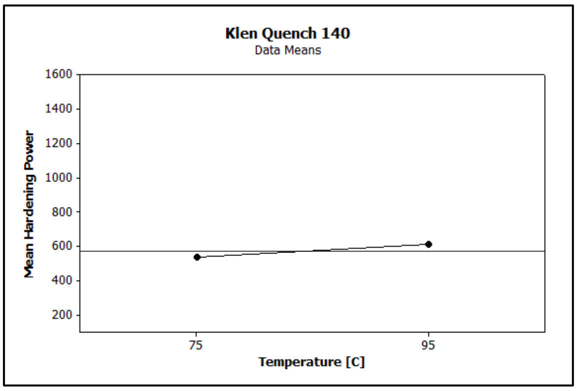

Figure 113 *Hardening Power depending on temperature for 1 litre new and used Klen Quench 140 from Company C without agitation.*

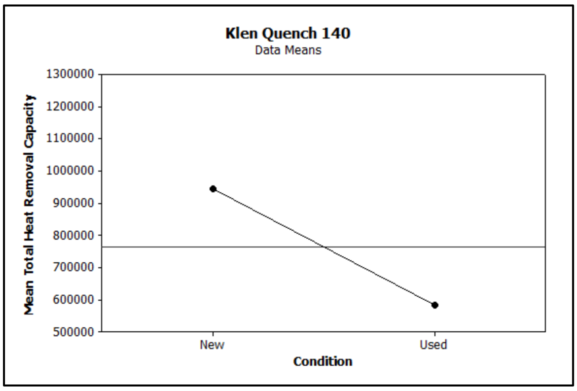

Figure 114 *Total Heat Removal Capacity depending on condition for 1 litre new and used Klen Quench 140 from Company C without agitation.*

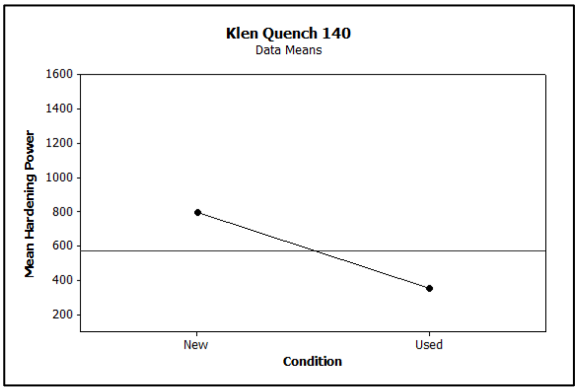

Figure 115 *Hardening Power depending on condition for 1 litre new and used Klen Quench 140 from Company C without agitation.*

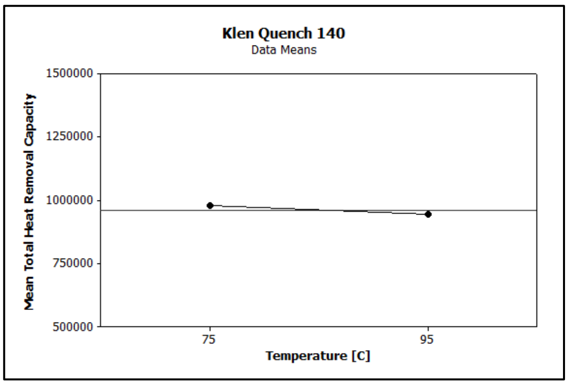

Figure 116 *Total Heat Removal Capacity depending on temperature for 10 litres new and used Klen Quench 140 from Company C without agitation.*

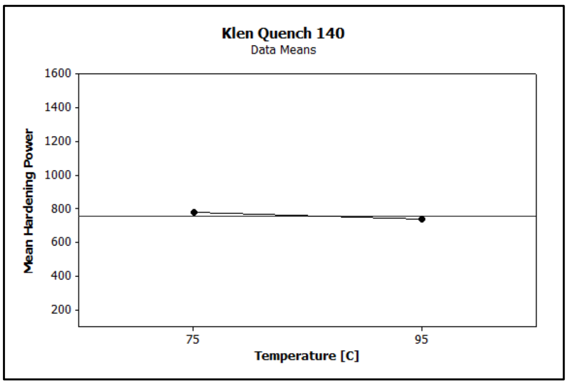

Figure 117 *Hardening Power depending on temperature for 10 litres new and used Klen Quench 140 from Company C without agitation.*

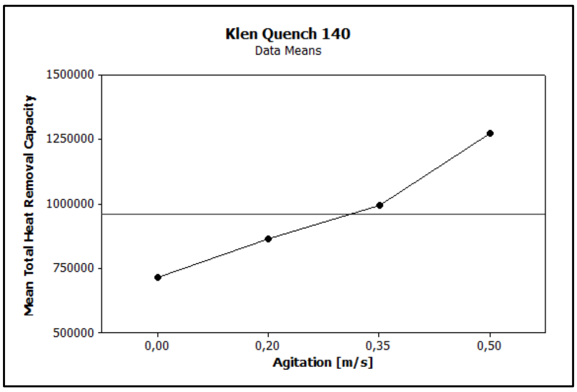

Figure 118 *Total Heat Removal Capacity depending on agitation for 10 litres new and used Klen Quench 140 from Company C at 75 and 95°C.*

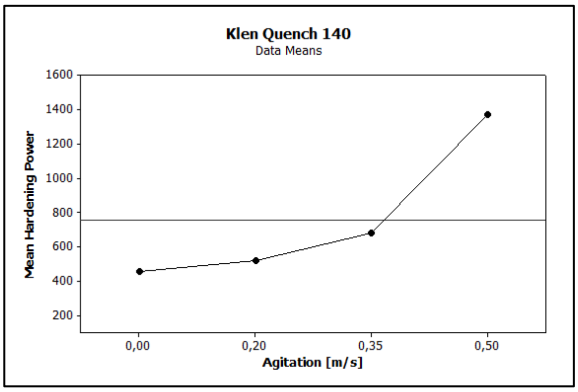

Figure 119 *Hardening Power depending on agitation for 10 litres new and used Klen Quench 140 from Company C for 75 at 95°C.*

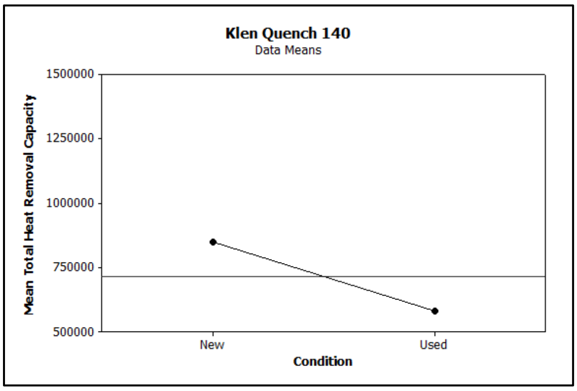

Figure 120 *Total Heat Removal Capacity depending on condition for 10 litres new and used Klen Quench 140 from Company C without agitation.*

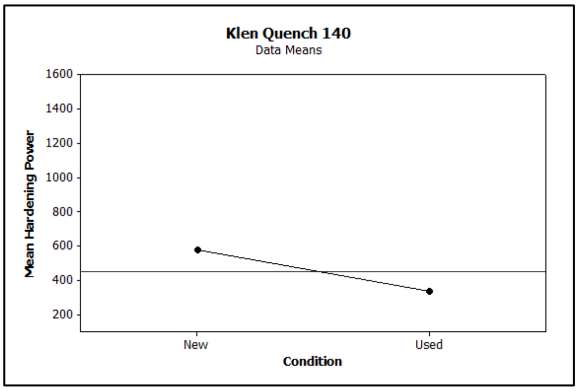

Figure 121 *Hardening Power depending on condition for 10 litres new and used Klen Quench 140 from Company C without agitation.*

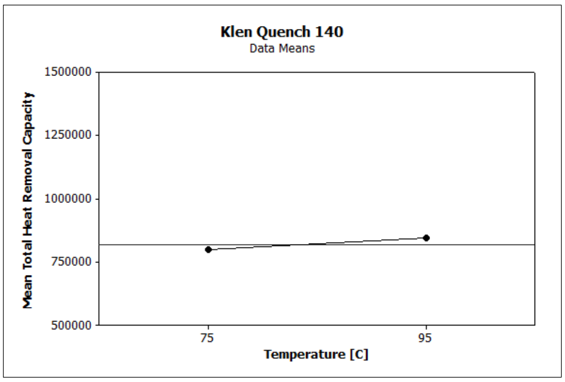

Figure 122 *Total Heat Removal Capacity depending on temperature for 1 and 10 litres new Klen Quench 140 from Company C at 75 and 95°C without agitation.*

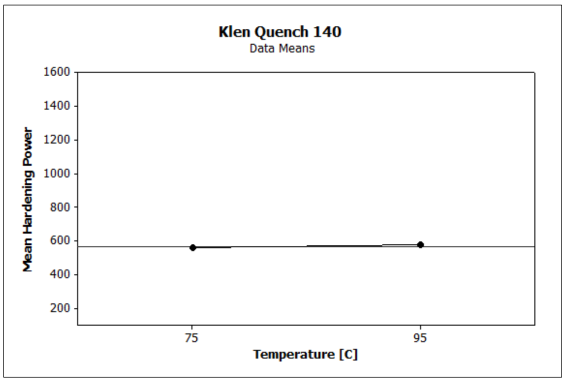

Figure 123 *Total Hardening Power depending on temperature for 1 and 10 litres new Klen Quench 140 from Company C at 75 and 95°C without agitation.*

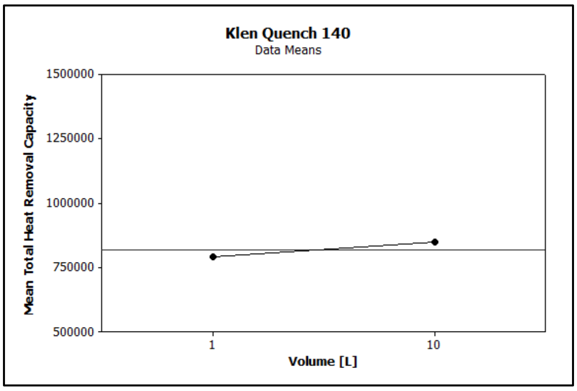

Figure 124 *Total Heat Removal Capacity depending on volume for new Klen Quench 140 from Company C at 75 and 95°C without agitation.*

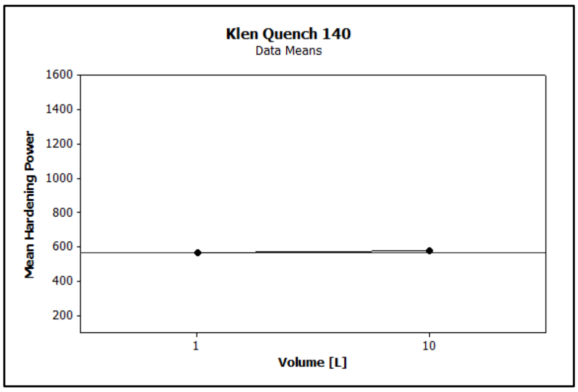

Figure 125 *Hardening Power depending on volume for new Klen Quench 140 from Company C at 75 and 95°C without agitation.*

Durixol W72

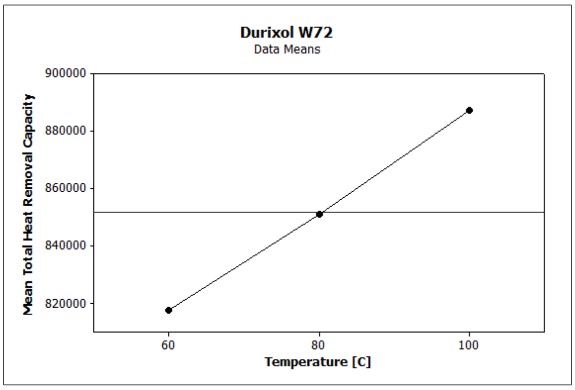

Figure 126 *Total Heat Removal Capacity depending on condition for 1 litre Durixol W72 from Company E with temperature 60, 80 and 100°C without agitation.*

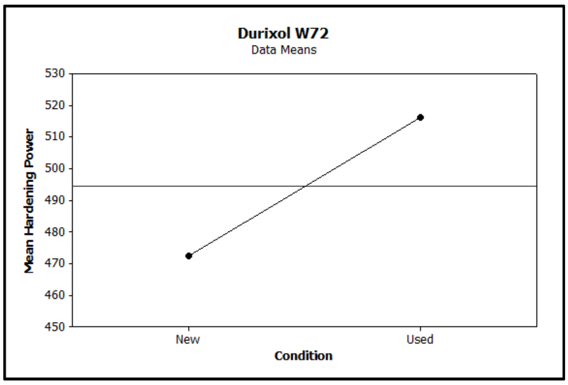

Figure 128 *Hardening Power depending on temperature for 1 litre new and used Durixol W72 from Company E without agitation.*

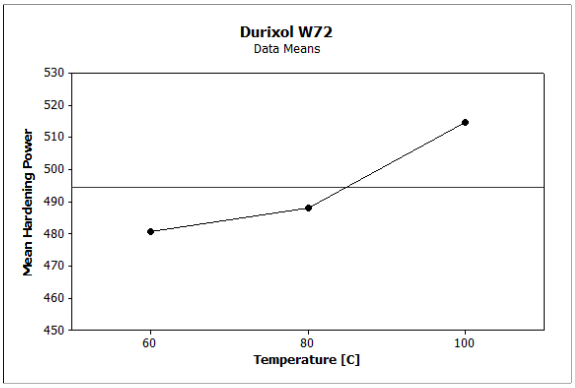

Figure 127 *Hardening Power depending on condition for 1 litre Durixol W72 from Company E with temperature 60, 80 and 100°C without agitation.*

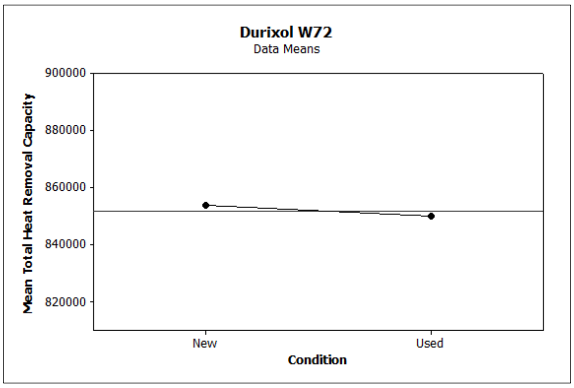

Figure 129 *Total Heat Removal Capacity depending on temperature for 1 litre new and used Durixol W72 from Company E without agitation.*

13 Appendix D

A General Linear Model was performed on 10 litres QuenchWay 125 B from Company A, see the corresponding Residual Plots in figure 130 and 131. Measurements were performed on both new and used oil at 130 °C with agitation 0.0, 0.2, 0.35 and 0.5 m/s. The values in figure 130 are measured in Total Heat Removal Capacity and in figure 131 they are measured in Hardening Power. In table 8 and 9 the p-values for the different factors are presented together with the R-Sq(adj) value that is indicating how much of the variation is covered by the model.

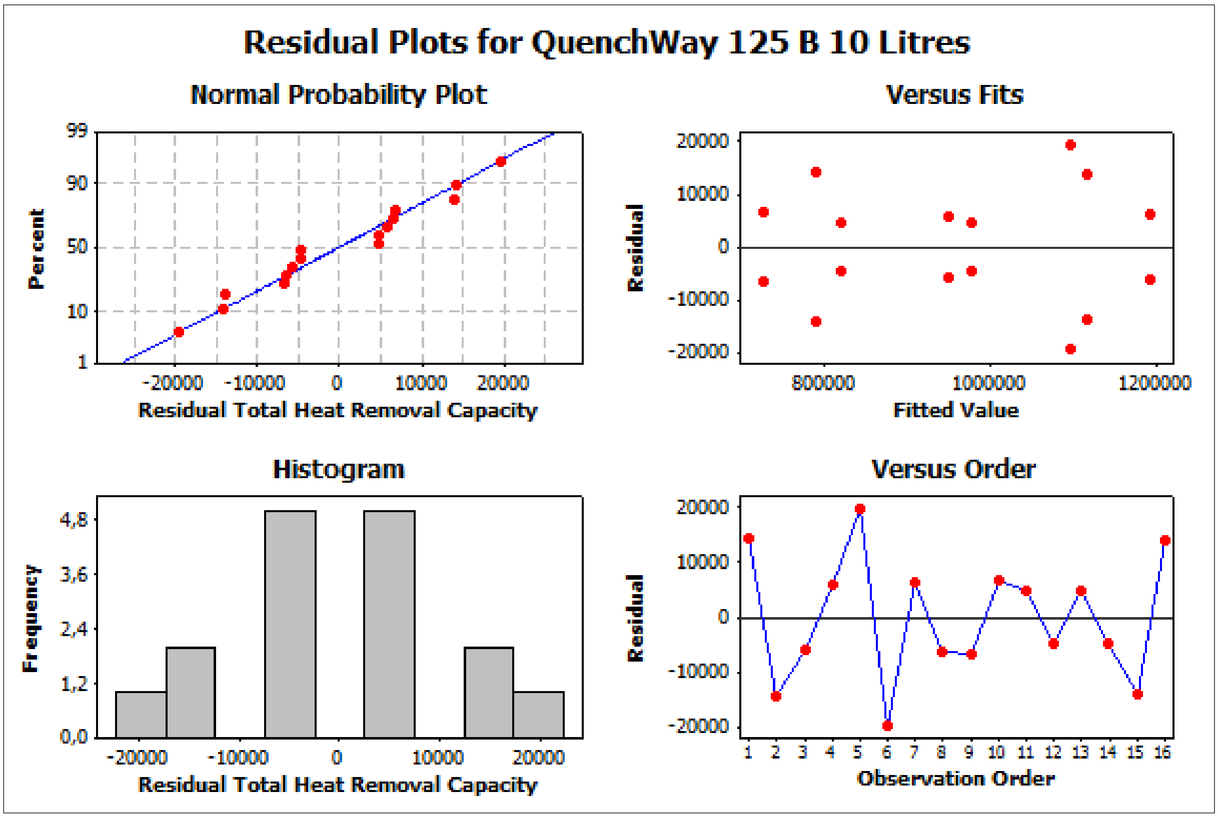

Figure 130 *Residual Plot for Heat Removal Capacity versus temperature and agitation for 10 litres new and used QuenchWay 125 B from Company A at 130°C.*

Table 8 *The p-values and R-Sq(adj) for the General Linear Model for Heat Removal Capacity versus condition and agitation for 10 litres new and used QuenchWay 125 B from Company A at 130 °C.*

Source	p-value
Condition	0.000
Agitation	0.000
Condition*Agitation	0.040
R-Sq(adj) = 99.12%	

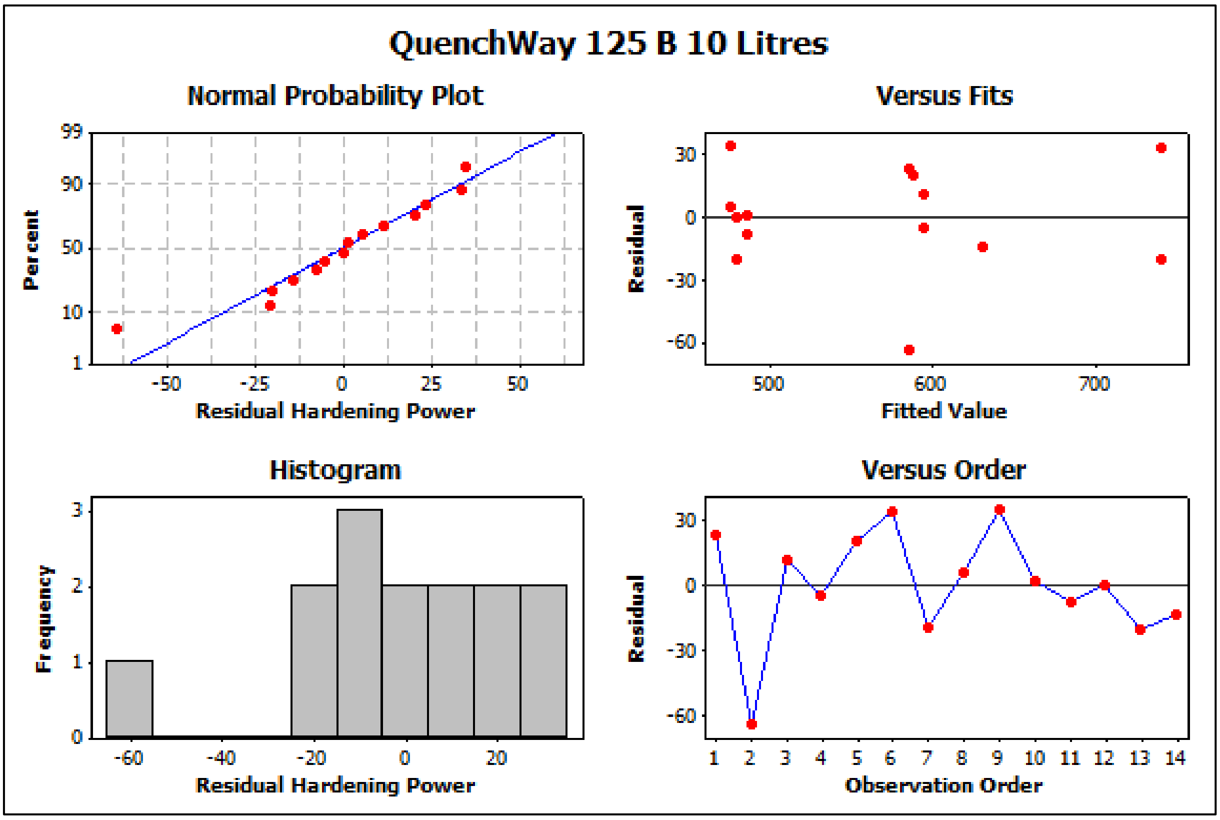

Figure 131 *Residual Plot for Hardening Power versus condition and agitation for 10 litres new and used QuenchWay 125 B from Company A at 130°C.*

Table 9 *The p-values and R-Sq(adj) for the General Linear Model for Hardening Power versus condition and agitation for 10 litres new and used QuenchWay 125 B from Company A at 130°C.*

Source	p-value
Condition	0.000
Agitation	0.000
R-Sq(adj) = 89.56%	

A General Linear Model was performed on new and used QuenchWay 125 B from Company A. The corresponding Residual Plots can be seen in figure 3. Measurements were performed when the temperature was at 130°C and the volume were varying between 1 and 10 litres. The values are measured in Hardening Power. In table 10 the p-values for the different factors are presented together with the R-Sq(adj) value that is indicating how much of the variation is covered by the model.

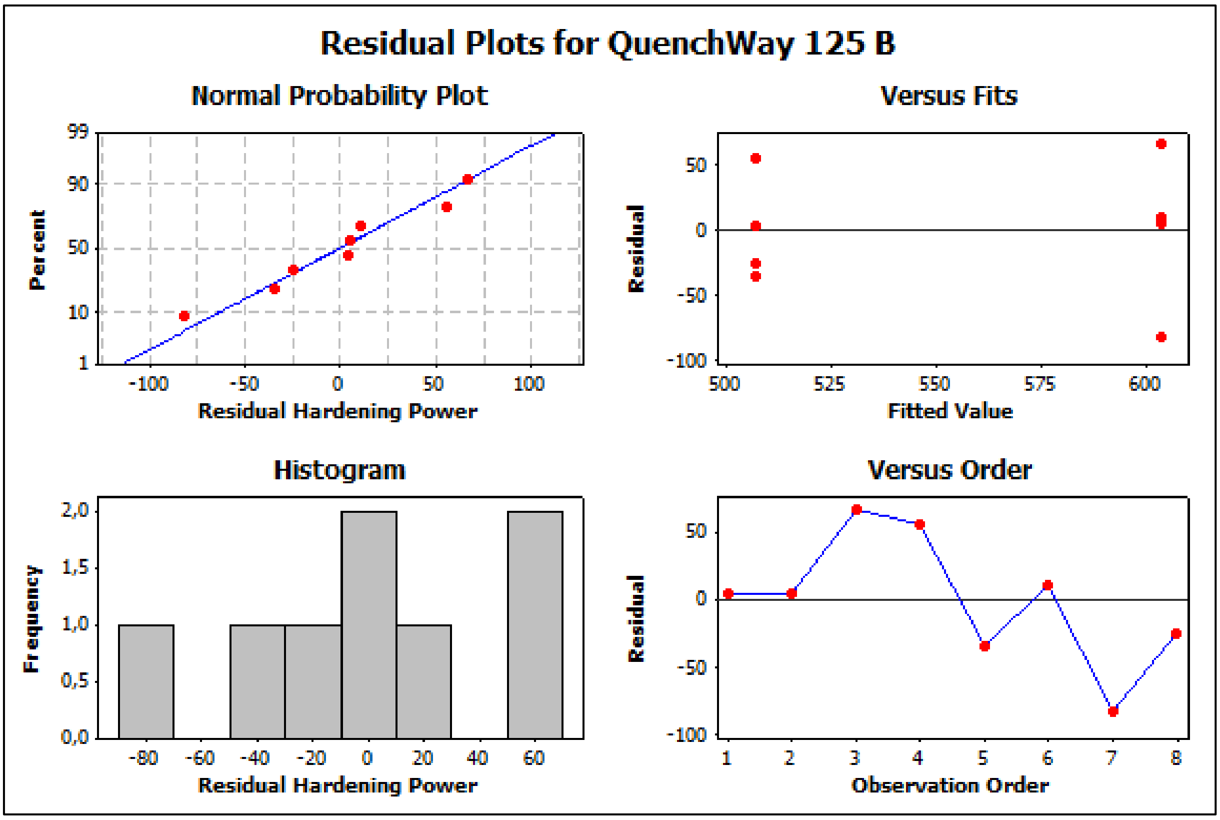

Figure 132 *Residual Plot for Hardening Power versus condition and volume for new and used QuenchWay 125 B from Company A at 130°C without agitation.*

Table 10 *The p-values and R-Sq(adj) for the General Linear Model for Hardening Power versus condition and volume for QuenchWay 125 B from Company A at 130°C without agitation.*

Source	p-value
Condition	0.040
R-Sq(adj) = 45.38%	

A General Linear Model was performed on 10 litres Klen Quench 140 from Company C, see the corresponding Residual Plots in figure 133 and 134. Measurements were performed on both new and used oil at 75 and 95°C with agitation 0.0, 0.2, 0.35, and 0.5 m/s. The values in figure 133 are measured in Heat Removal Capacity and in figure 134 they are measured in Hardening Power. In table 11 and 12 the p-values for the different factors are presented together with the R-Sq(adj) value that is indicating how much of the variation is covered by the model.

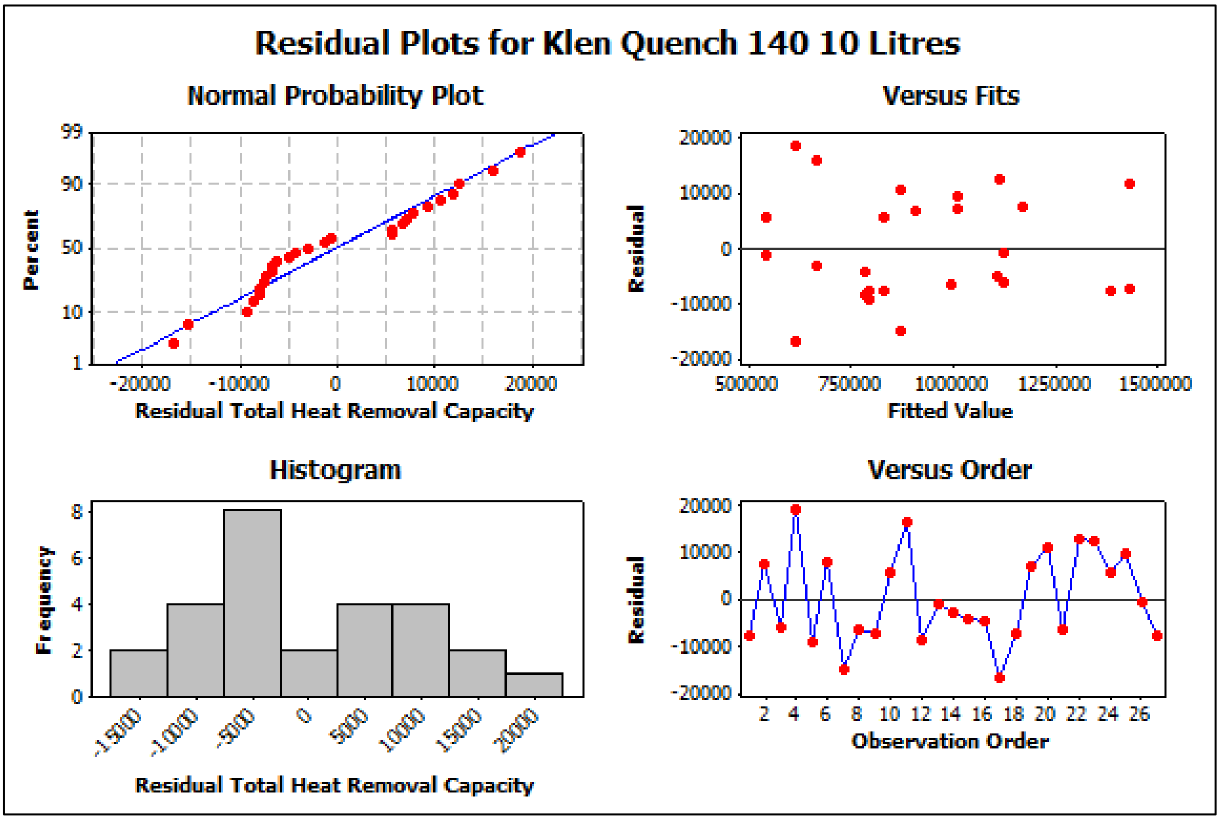

Figure 133 *Residual Plots for Total Heat Removal Capacity versus temperature, agitation and condition for 10 litres Klen Quench 140 from Company C.*

Table 11 *The p-values and R-Sq(adj) for the General Linear Model for Total Heat Removal Capacity versus temperature, agitation and condition for ten litres Klen Quench 140 from Company C.*

Source	**p-value**
Temperature	0.000
Agitation	0.000
Condition	0.000
Condition*Temperature	0.000
Temperature*Agitation	0.000
R-Sq(adj) = 99.78%	

Two unusual observations for the model were found. One was for new oil at 75°C with agitation 0.5 m/s. The second one was from measurements on new oil at 95°C with agitation 0.35 m/s. The reason for this is the same reason as said for QuenchWay 125 B. The software calculates the T_{cp} value wrong. The observations were removed for the final model.

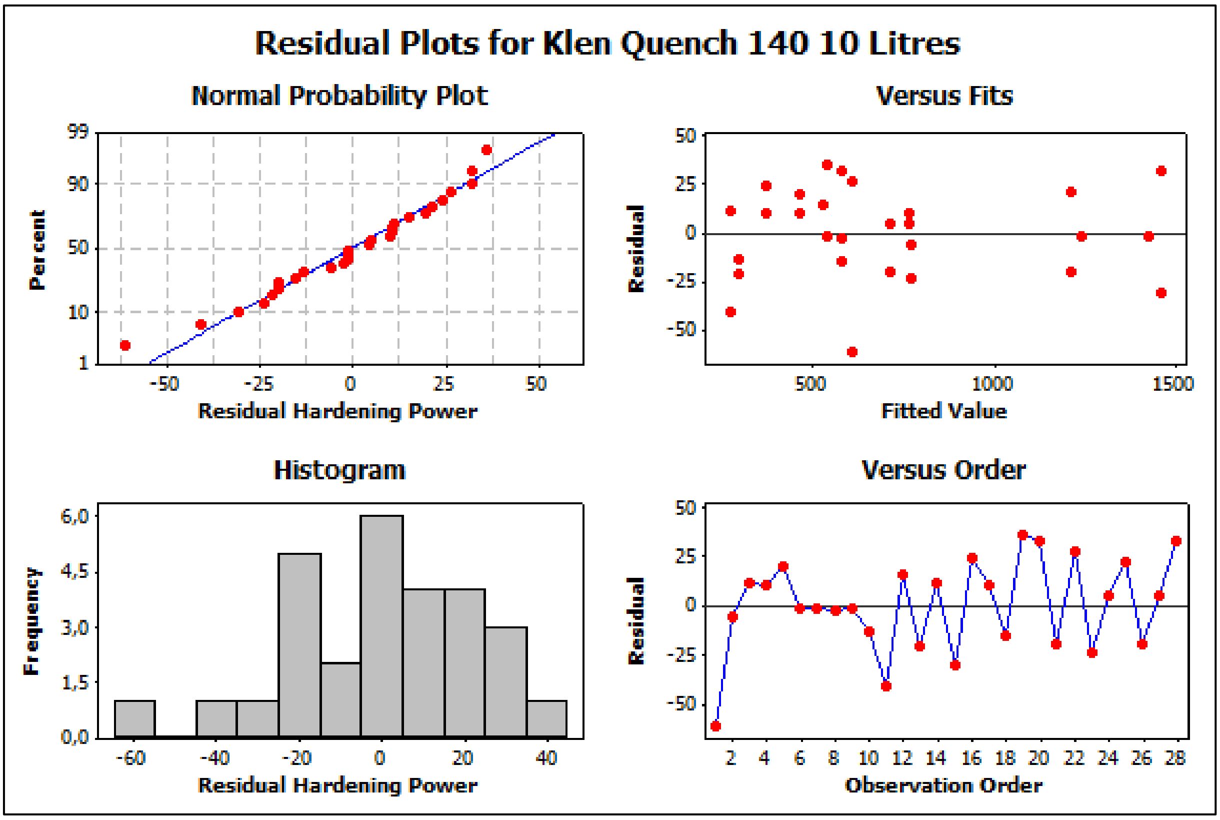

Figure 134 *Residual Plots for Hardening Power versus temperature, agitation and condition for 10 litres Klen Quench 140 from Company C.*

Table 12 *The p-values and R-Sq(adj) for the General Linear Model for Hardening Power versus temperature, agitation and condition for 10 litres Klen Quench 140 from Company C.*

Source	p-value
Temperature	0.000
Agitation	0.000
Condition	0.000
Temperature*Agitation	0.000
Condition*Agitation	0.000
R-Sq(adj) = 99.32%	

One unusual observation for Hardening Power versus temperature, agitation and condition was found. It was for 75 ˚C new oil at 0 m/s. The reason for this observation was not found and it was not removed from the final model.

A General Linear Model was performed on new Klen Quench 140 from Company C. The corresponding Residual Plots can be seen in figure 135 and 136. Measurements were performed when the temperature was either 75 or 95 °C and volume was either 1 or 10 litres. The values in figure 135 are measured in Heat Removal Capacity and in figure 136 it is measured in Hardening Power. In table 13 and 14 the p-values for the different factors are presented together with the R-Sq(adj) value that is indicating how much of the variation is covered by the model.

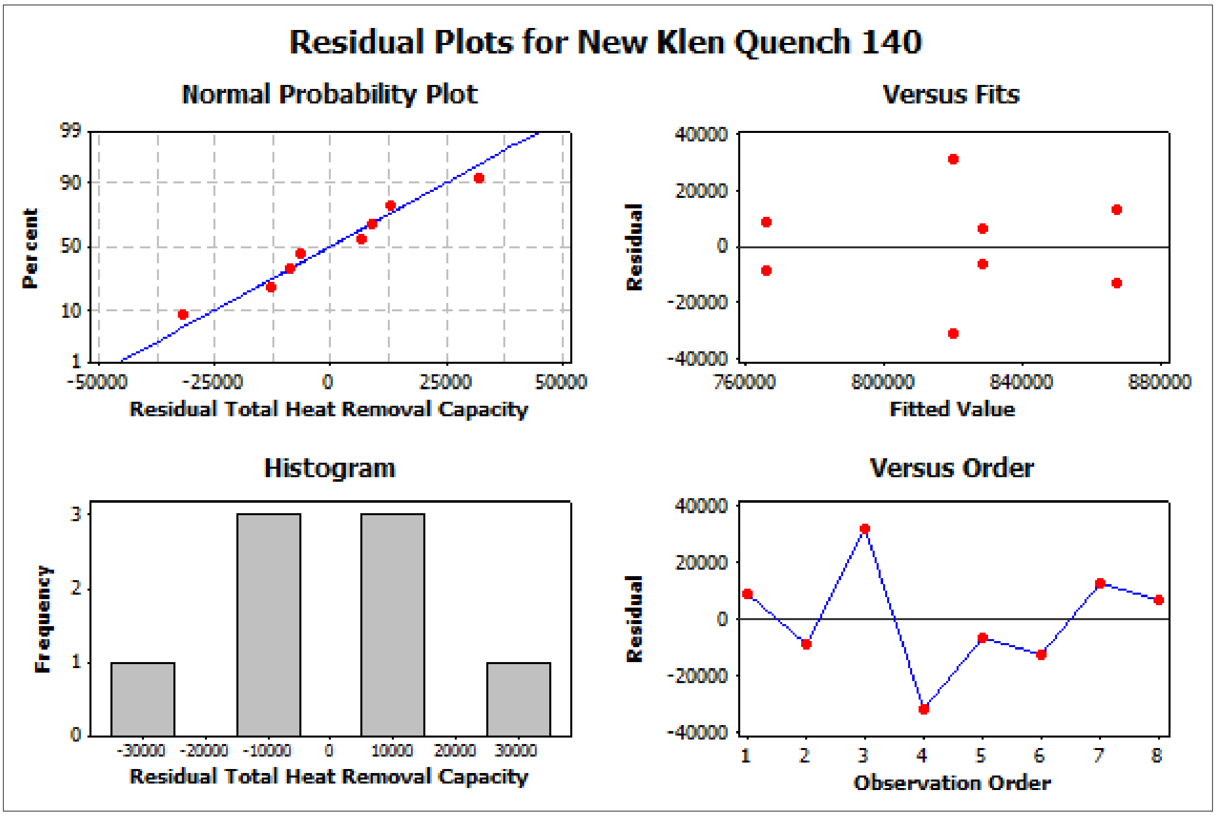

Figure 135 *Residual Plots for Heat Removal Capacity versus temperature and volume for 1 and 10 litres new Klen Quench 140 from Company C at 75 and 95°C without agitation.*

Table 13 *The p-values and R-Sq(adj) for the General Linear Model for Heat Removal Capacity versus temperature and volume for 1 and 10 litres new Klen Quench 140 from Company C at 75 and 95°C without agitation.*

Source	**p-value**
Temperature	0.037
Volume	0.021
R-Sq(adj) = 70.89%	

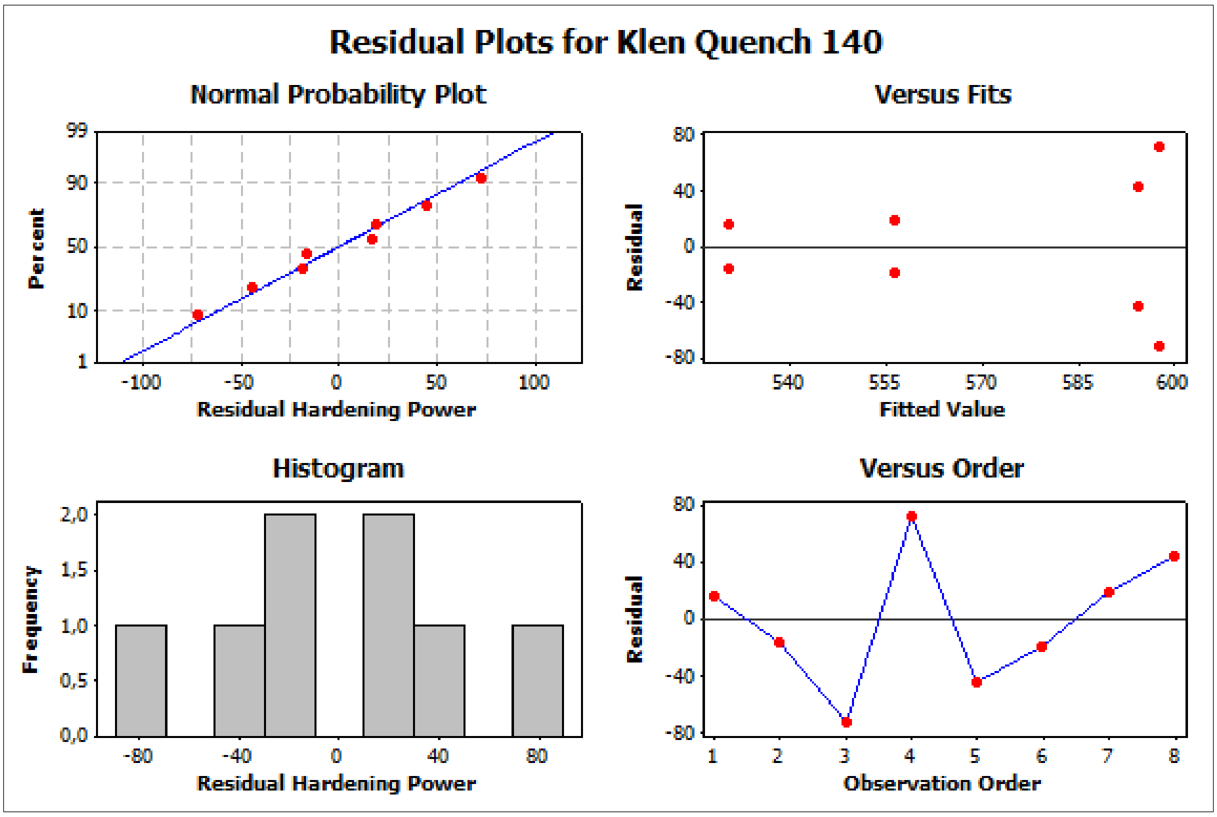

Figure 136 *Residual Plots for Heat Removal Capacity versus temperature and volume for 1 and 10 litres new Klen Quench 140 from Company C at 75 and 95°C without agitation.*

Table 14 *The p-values and R-Sq(adj) for the General Linear Model for Hardening Power versus temperature and volume for 1 and 10 litres new Klen Quench 140 from Company C at 75 and 95°C without agitation.*

Source	p-value
Temperature	0.758
Volume	0.809
Volume*Temperature	0.299
R-Sq(adj) = 0.00%	

In this General Linear Model no parameter were found to be significant. The reason for this could be that there are too few measurements.

14 Appendix E

Results from the Vickers Hardness Testing on 16MnCr5 quenched in QuenchWay 125 B from Company A and 100Cr6 quenched in Klen Quench 140 from Company C.

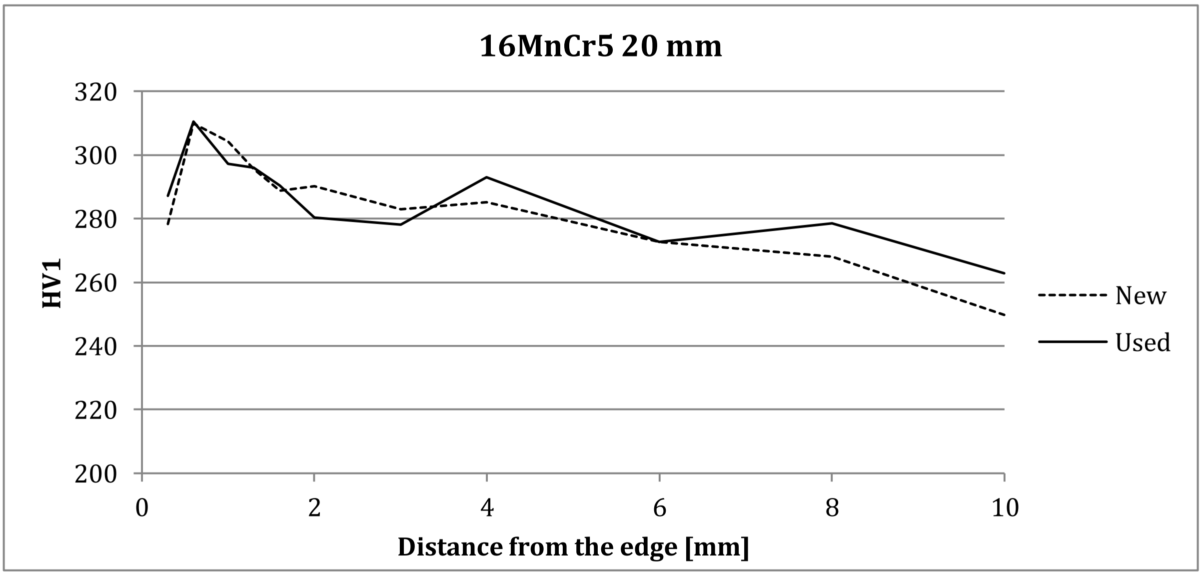

Figure 137 *16MnCr5 hardening in QuenchWay 125 B from Company A with agitation 0,2 m/s, furnace temperature 880°C and oil temperature 130°C*

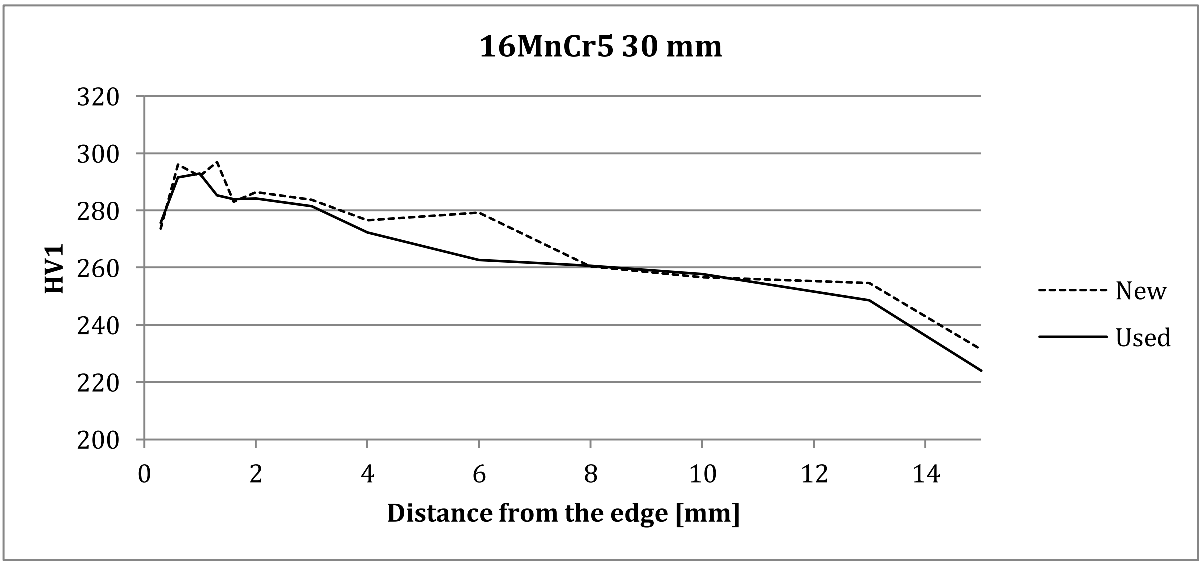

Figure 138 *16MnCr5 hardening in QuenchWay 125 B from Company A with agitation 0,2 m/s, furnace temperature 880°C and oil temperature 130°C*

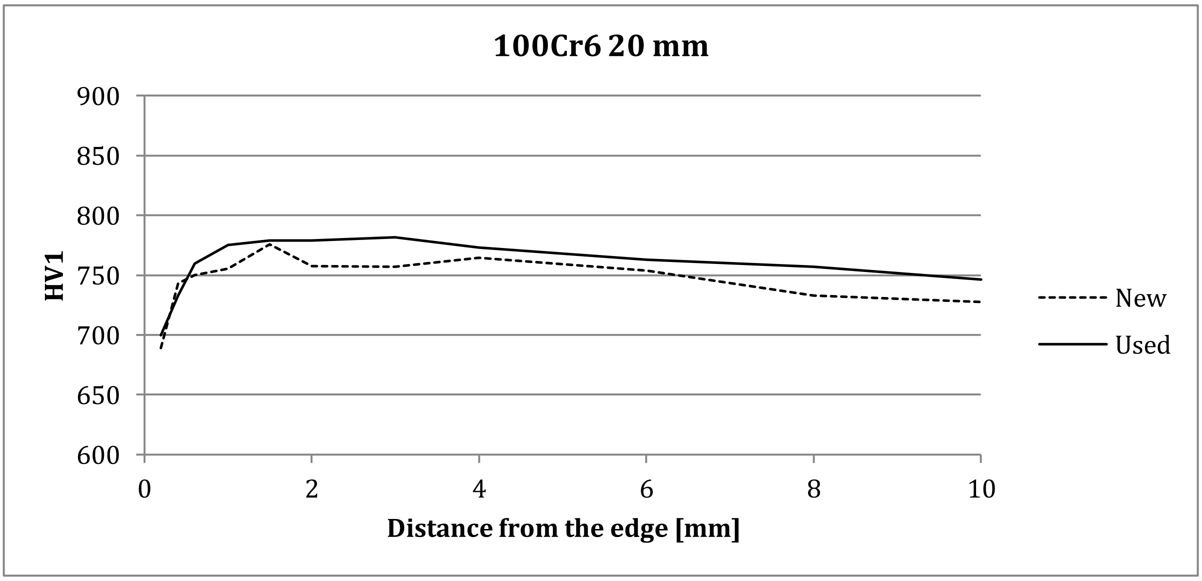

Figure 139 *100Cr6 hardened in Klen Quench 140 from Company C with agitation 0,2 m/s, furnace temperature 860°C and oil temperature 75°C*

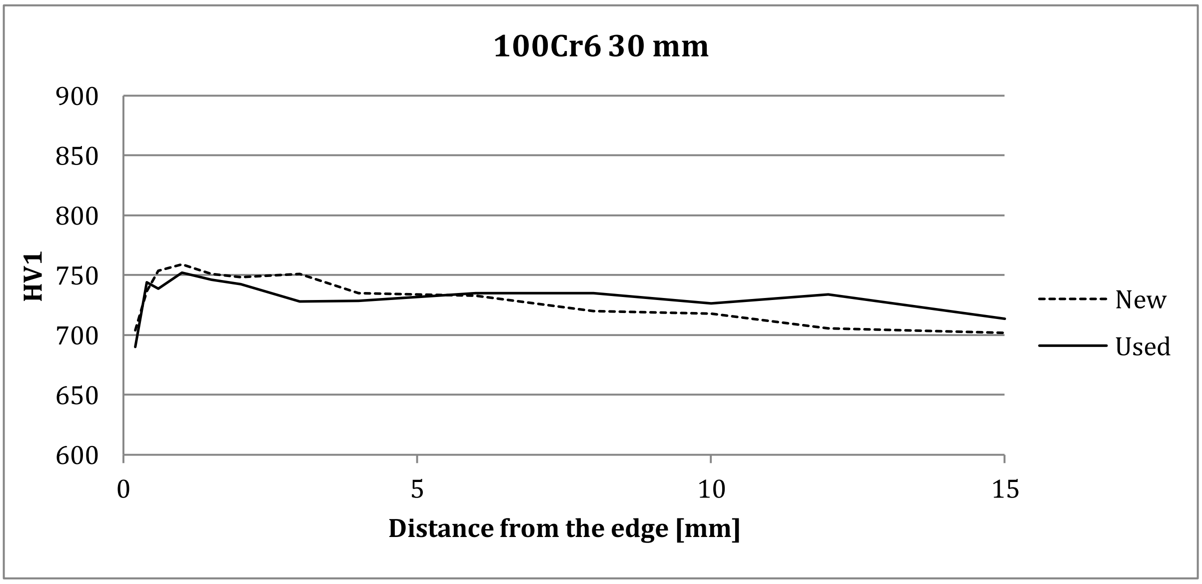

Figure 140*100Cr6 hardened in Klen Quench 140 from Company C with agitation 0,2 m/s, oven temperature 860°C and oil temperature 75°C*