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# RESPONSE SURFACE METHODOLOGY FOR STUDYING THE EFFECT OF OPERATING VARIABLES ON QUENCHING IN OIL MEDIUM

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***Abstract:*** *Quenching is being described as one of the most common heat treatment processes used to impart the desire mechanical properties such as high strength, hardness and near resistance to metal parts using quenchants such as air, water and polymer solution. The quenching process parameters such as time, radial distance and immersion speed played a major role in deciding the heat treatment quality of the steel sample. In this research, response surface methodology was used to study the effect of process parameters on temperature distribution during the quenching process of AISI1020 steel sample. A total of seventeen experimental runs were designed using the three variables adopting Box-Behnken design with full replication technique and mathematical model was developed. Sensitivity analysis was carried out to identify critical parameters. Time was found to be the most influencing parameter on the temperature distribution, followed by immersion speed and the least effect was given by radial distance. The quadratic model developed was evaluated at p-value greater than 95% confidence level, having correlation coefficient R-squared of 0.9997, adjusted R-squared of 0.9993 and predicted R-squared of 0.9953.*

***Keywords:*** *Analysis of Variance (ANOVA), quenchant, austenitization, Response Surface Methodology (RSM), Box-Behnken*

## INTRODUCTION

*Heat treatments can be broadly described as processes or an operation or combination of operations that involves heating and cooling of solid metals to acquire appropriate mechanical properties or for the purpose of obtaining specific properties which could be suited for particular working environments [1,2] (Houghton, 2000; Grum et al., 2001). The heat treatment process include, heating of the steel to a definite temperature; holding (or soaking) at that temperature for a sufficient period of time and cooling at rate in order to change the mechanical properties, the metallurgical structure or the residual stress state.*

*Quenching of steel involves the cooling from the solution treating temperature, typically 845-870°C (1550-1600°F), into the hard structure-martensite [3] (Bates and Totten, 1992) and is typically performed to prevent ferrite or pearlite formation and to facilitate bainite or martensite formation [3] (Bates et al., 1992). Although quenching is the most difficult part in the heat treatment process, as the material properties depends heavily on the cooling rate [4](Buche et al., 2005), it is an integral part of industrial heat-treatment processes for steels and provide means by which mechanical properties of a steel part can be controlled [5](Woodard, 1999). Therefore, quenching operation is one of the most important steps that determine the quality of heat–treated product and the quenching quality is decided by the cooling ability and temperature field distribution of the quenching medium [6](Li Qiang et al., 2003). The quenching medium also known as quenchant includes water, oil, brine, air, molten salts and polymeric materials. The quenchant to be employed in a quenching process depends on the type of steel.*

*Among the quenching medium, oils had an excellent quenching properties as a quenchants, provide moderate cooling rate and therefore result in minimal distortion in the component [7](Ndaliman, 2006). Many components use oil quenching to achieve consistent and repeatable mechanical and metallurgical properties and predictable distortion patterns. The reason oil quenching is so popular is due to its excellent performance results and stability over a broad range of operating conditions. Oil quenching facilitates hardening of steel by controlling heat transfer during quenching, and it enhances wetting of steel during quenching to minimize the formation of undesirable thermal and transformational gradients which may lead to increased distortion and cracking. For many, the choice of oil is the result of an evaluation of a number of factors including: Economics/cost (initial investment, maintenance, upkeep, and life), Performance (cooling rate/quench severity), Minimization of distortion (quench system), Variability (controllable cooling rates) and Environmental concerns (recycling, waste disposal, etc.).[8](Herring, 2010).*

*Oils are generally classified by their ability to transfer heat as fast, medium, or slow "speed" oils. Fast (8-10 seconds) oils are used for low hardenability alloys, carburized and carbonitrided parts, and large cross sections that require high cooling rates to produce maximum properties. Medium (11 – 14 seconds) oils are typically used to quench medium to high hardenability steels. Slow (15-20* *seconds) oils are used where hardenability of a steel is high enough to compensate for the slow cooling aspects of this medium [9](Herring D et. al, 1986). The temperature of the metal surface is reduced to the boiling point (or boiling range) of the quenching liquid. Below this temperature, boiling stops and slow cooling takes place by conduction and convection. The difference in temperature between the boiling point of the liquid and the bath temperature is a major factor influencing the rate of heat transfer in liquid quenching.*

*Response Surface Methodology (RSM), invented by Box and Wilson, is defined as a collection of mathematical and statistical tools or techniques useful for modeling, analyzing and simultaneously solving problems in which a response of interest is influenced by several variables and the objectives is to optimize this response (Giovanni, 1983). Response surface methodology also quantifies the relationship between the controllable input parameters and the obtained response surfaces. It is a well-known up to date approach for constructing approximation models based on physical experimented observations [10](Box et al., 2005). The main advantage of RSM is the reduced number of experimental runs needed to provide sufficient information for statistically acceptable results [11](Montgomery, 2001).*

*Karthikeyan et al. [12] developed mathematical models to optimize the heat treatment conditions for maximum yield strength and ductility of aluminum–silicon carbide particulate composites. The response surface method was used to fit the mathematical models, and the process variables included the volume fraction of SiC, aging temperature, aging time, and solutionizing time. RSM was used for technologic parameter optimization of gas quenching process by Huiping et. al [13]. In this present study, response surface methodology was considered to study the effect of process variables (time, radial distance and immersion speed) on cooling rate of oil quenched process.*

## *METHODOLOGY*

### *Parameter Evaluation of Oil quenched process*

*During the process of oil quenching, the process parameters were classified as the independent parameter and the dependent parameter. The independent parameters during the oil quenching process include time, radial distance and immersion speed while temperature distribution is the dependent parameter. Yao et al. (2003)[14] investigated the transient temperature, structure and internal stress evolution and distribution of oil–quenched centric and eccentric cylindrical tubes by a finite element method. They discovered that at the initial stage of quenching process, the residual axial stresses are tensile at the surface and compressive in the core for both geometries.*

*Therefore, in this research, the temperature distribution is regarded as a variable to be predicted, the range of values for the independent parameters are shown in Table 1. The material of the quenching process is AISI1020 mild steel bar and the quenchant is oil.*

### *Sample Preparation*

*A solid cylindrical mild steel bar (AISI1020) purchased at local steel market was machined at the Fabrication workshop, Department of Mechanical Engineering, Faculty of Engineering and Technology, LAUTECH, Ogbomoso Nigeria to produce a specimen of 100 mm long of 30 mm diameter illustrated in Figure 1. Three 2 mm diameter hole are drilled to a depth of 5mm at 5mm, 15mm and 25mm from outside diameter of the specimen, to accommodate the thermocouples that are used for temperature measurements. Ten samples of the specimen are produced and used for the experiment.*

### *Experimental Set-up*

*The prepared samples of steel probes of length 100mm and diameter 30mm were connected with a chrome/alumel K-type thermocouple via a tight fitting screw to prevent the quenching media from entering the drilled holes during quenching. The thermocouples were connected to a 12 channel temperature recorder model BTM-4208 SD with SD data logger to conduct the data acquisition process of the temperature and time.*

*The complete assembly of the specimens (the specimen and thermocouples) was placed in a temperature controlled furnace Vaster 232 models available at the New Chemical Laboratory, Department of Chemical Engineering, LAUTECH, Ogbomoso Nigeria. Heated and soaked at an austenitized temperature of 850$^{0}$C for one hour to promote complete austenitization of the specimen. The heated specimen was quickly transferred from the furnace into 1000ml quenching medium contained in a vertical tank under static condition and the probe dipped horizontally as practiced in industry via an immersion rig which consists of a one horse power electric motor and a voltage regulator. The speed of the electric motor which represents the speed of the immersion of the heated specimen was monitored with a digital tachometer model DT-2234B. The heating and quenching procedures were repeated twice for immersion speed of 0.1 m/s, 0.35 m/s and 0.6 m/s using mineral oil as the quenchant used.*

*The tensile test samples and other samples prepared for hardness tests and micro-structural analyses were also heated and quenched at immersion speed of 0.1 m/s, 0.35 m/s and 0.6 m/s.*

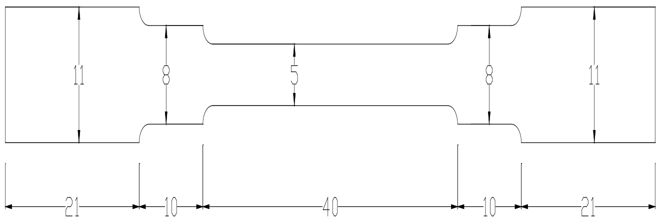

***Figure 1:** Schematic diagram of specimen used for tensile and hardness tests (All dimensions in mm)*

### *Experimental Design for the Response Surface Procedure*

*Response surface methodology has been used to study the optimization of chemical processes and products (Sudesh et al., 2010; Mane et al., 2007; Ven et al., 2002). Response surface methodology was used in this study to investigate the effect of some quenching* parameters for the performance of the quenched steel in heat treatment process. A three factor, Box-Benhnken Design (BBD) model was used to design the experiment. Design-Expert version 8.0.3 was used for the modeling of the identified variables. The factors considered were time, radial distance and immersion speed while the response is temperature distribution. The experimental range of the variables are tabulated was in Table 1. The experimental range was used to design experiment used for the modeling which was tabulated in Table 2.

The quadratic response surface model considering all the linear terms, square terms and linear by linear interactions terms according to Huiping et al., (2008) was described as:

$$Y = \beta_o + \varepsilon \sum_{i=1}^{k} \beta_i x_i + \sum_{i-1}^{k} \beta_{ij} x_i x_j + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \varepsilon \qquad (1)$$

where Y is predicted response used as a dependent variable, $\beta_o$ represents the overall mean, $\beta_i$ represents the linear effect of the input factor $x_i$; $\beta_{ij}$ represents the linear by linear interaction effect between the input factor $x_i$ and $x_j$; $\beta_{ii}$ represents the quadratic effect of the input factor $x_i$ and ε is the random error term.

**Table 1:** Design of factors for temperature distribution

| Factors | Code | Level | | |
|---|---|---|---|---|
| | | Low (-1) | Standard (0) | High (+1) |
| Time (s) | A | 2 | 51 | 100 |
| Radial distance (mm) | B | 5 | 15 | 25 |
| Immersion speed (m/s) | C | 0.1 | 0.35 | 0.6 |

**Table 2:** Box Behnken Design of Experiment Model Range in coded and actual values

| Std | Coded | | | Actual | | |
|---|---|---|---|---|---|---|
| | Time | Radial dist | Immersion speed | Time | Radial dist | Immersion speed |
| 7 | -1 | 0 | 1 | 15 | 0.1 | 56 |
| 3 | -1 | 1 | 0 | 25 | 0.1 | 96.5 |
| 11 | 1 | -1 | 1 | 15 | 0.35 | 71.1 |
| 8 | 1 | 0 | 1 | 25 | 0.35 | 61.9 |
| 10 | 0 | 1 | -1 | 5 | 0.6 | 116.2 |
| 4 | 1 | 1 | 0 | 15 | 0.35 | 71.1 |
| 13 | 0 | 0 | 0 | 5 | 0.35 | 53.2 |
| 1 | -1 | -1 | 0 | 15 | 0.35 | 71.1 |
| 14 | 0 | 0 | 0 | 25 | 0.6 | 120.5 |
| 17 | 0 | 0 | -1 | 5 | 0.35 | 819.9 |
| 5 | -1 | 0 | 1 | 15 | 0.6 | 838.4 |
| 6 | 1 | 1 | 1 | 15 | 0.35 | 71.1 |
| 12 | 0 | 1 | 1 | 15 | 0.6 | 60.2 |
| 9 | 0 | -1 | -1 | 15 | 0.1 | 841 |
| 15 | 0 | 0 | 0 | 25 | 0.35 | 847.1 |
| 16 | 0 | 0 | 0 | 5 | 0.1 | 76.1 |
| 2 | 1 | -1 | 0 | 15 | 0.35 | 71.1 |

### Statistical Data Analysis

Analysis of variance (ANOVA) was used for the analyses of the data obtained from quenching experiment for oil quenching medium. The interactions between the process variables and the responses of different regression models developed for temperature distribution using oil as quenching medium were investigated. The quality of the fit polynomial model was expressed by the coefficient of determination $R^2$, and its statistical significance was checked by the Fisher's F-test in the same in-built statistical program of the Design Expert 8.0.3. Model terms were evaluated by the p-value (probability) with 95% confidence level. Three dimensional surface plots and their respective contour plots were obtained for temperature distribution on the effects of the three factors (time, radial distance and immersion speed).

## RESULT AND DISCUSSION

### Data Analysis

The experimental results, the predicted values and the residuals of data were shown in table 3. A quadratic model was developed from the data showing the relationship between temperature distribution and the input parameters (time, radial distance and immersion speed). The adequacy of the developed model was tested statistically using the analysis of variance (ANOVA) technique and the results of second order response surface model fitting are given in Table 4. The determination coefficient ($R^2$) indicates the goodness of fit for the model. In this case, the value of the determination coefficient ($R^2=0.9997$) indicates that only less than 1% of the total variations are not explained by the model. The value of adjusted determination coefficient (adjusted $R^2=0.9993$) was high, which indicates a high significance of the model. Predicted $R^2$ 0f 0.9953 was also in a good agreement with the adjusted $R^2$. Adequate precision compares the range of predicted values at the design points to the average prediction error. The model adequate precision ratio of 120.65 indicates an adequate signal.

The model equation in terms of actual factors is given as:

$$\text{Temperature}=896.0933-23.1685*A-2.37512*B-143.838*C$$
$$0.00944*A*B+0.138776*A*C+0.150146*A^2$$
$$+0.13925*B^2+276.8*C^2 \qquad (2)$$

where A= Time, B= Radial Distance and C= Immersion Speed.

**Table 3:** Experimental result of oil quenched steel sample

| Run | Variables | | | Temperature Distribution | | |
|---|---|---|---|---|---|---|
| | Time | Radial distance | Immersion speed | Actual | Predicted | Residuals |
| 6 | 100 | 15 | 0.1 | 56.00 | 49.60 | 6.40 |
| 10 | 51 | 25 | 0.1 | 96.50 | 105.71 | -9.21 |
| 17 | 51 | 15 | 0.35 | 71.10 | 71.10 | 0.00 |
| 4 | 100 | 25 | 0.35 | 61.90 | 59.09 | 2.81 |
| 11 | 51 | 5 | 0.6 | 116.20 | 106.99 | 9.21 |
| 13 | 51 | 15 | 0.35 | 71.10 | 71.10 | 0.00 |
| 2 | 100 | 5 | 0.35 | 53.20 | 53.19 | 0.01 |
| 15 | 51 | 15 | 0.35 | 71.10 | 71.10 | 0.00 |
| 12 | 51 | 25 | 0.6 | 120.50 | 114.09 | 6.41 |
| 1 | 2 | 5 | 0.35 | 819.90 | 822.71 | -2.81 |
| 7 | 2 | 15 | 0.6 | 838.40 | 844.80 | -6.40 |
| 14 | 51 | 15 | 0.35 | 71.10 | 71.10 | 0.00 |
| 8 | 100 | 15 | 0.6 | 60.20 | 69.43 | -9.22 |
| 5 | 2 | 15 | 0.1 | 841.00 | 831.78 | 9.22 |
| 3 | 2 | 25 | 0.35 | 847.10 | 847.11 | -0.01 |
| 9 | 51 | 5 | 0.1 | 76.10 | 82.51 | -6.41 |
| 16 | 51 | 15 | 0.35 | 71.10 | 71.10 | 0.00 |

*From the analysis of variance shown in table 4, the Model F-value of 2650.91 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. The p values less than 0.0500 indicate model terms are significant. In this case A, B, C, $A^2$, $B^2$, $C^2$ are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The input parameter which is most significant on the output performance (Temperature) is input parameter A which is Time because it shows the largest F-value of 16331.02 and minimum prob>F value, followed by the Immersion speed and the least effect is seen on Radial distance because of its least F-value of 6.1804. Interactions between the input parameters were not significant having p values >0.05.*

***Table 4:*** *ANOVA for response surface quadratic model of oil quenched steel*

| *Source* | *Sum of Squares* | *df* | *Mean Square* | *F Value* | *p-value Prob> F* | |
|---|---|---|---|---|---|---|
| *Model* | *1772061* | *9* | *196895.6* | *2650.913* | *<0.0001* | *significant* |
| *A* | *1212981* | *1* | *1212981* | *16331.02* | *<0.0001* | *significant* |
| *B* | *459.045* | *1* | *459.045* | *6.180373* | *0.0418* | *significant* |
| *C* | *539.5612* | *1* | *539.5612* | *7.264407* | *0.0309* | *significant* |
| *AB* | *85.5625* | *1* | *85.5625* | *1.151975* | *0.3187* | *Not-significant* |
| *AC* | *11.56* | *1* | *11.56* | *0.155639* | *0.7049* | *Not-significant* |
| *BC* | *64.8025* | *1* | *64.8025* | *0.872471* | *0.3814* | *Not-significant* |
| *A^2* | *547201.1* | *1* | *547201.1* | *7367.266* | *<0.0001* | *significant* |
| *B^2* | *816.4447* | *1* | *816.4447* | *10.99224* | *0.0128* | *significant* |
| *C^2* | *1260.168* | *1* | *1260.168* | *16.96633* | *0.0045* | *significant* |
| *Residual* | *519.9225* | *7* | *74.27464* | | | |
| *Lack of Fit* | *519.9225* | *3* | *173.3075* | | | |
| *Pure Error* | *0* | *4* | *0* | | | |
| *Cor Total* | *1772581* | *16* | | | | |

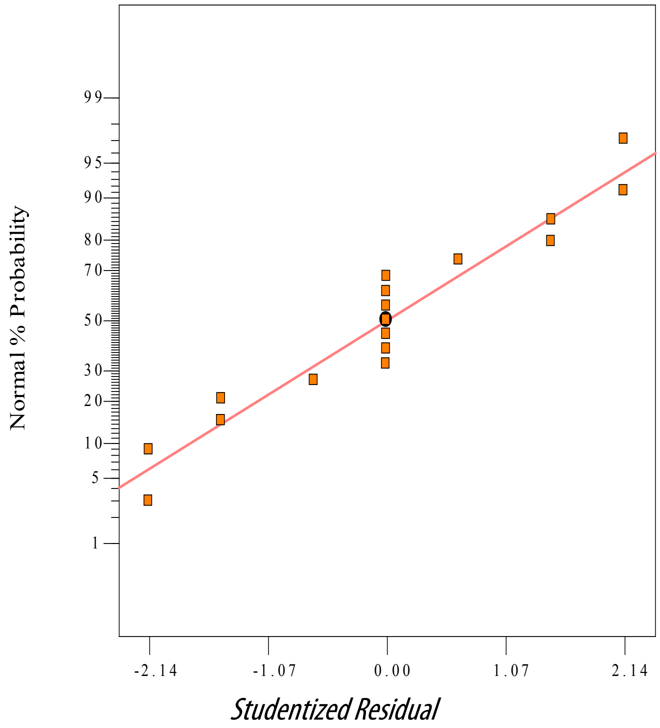

***Figure 2:*** *Normal probability plot of residuals for temperature distribution*

### ***Diagnostic plots of oil quenched steel sample***

*The quality of the model devloped was further tested using different diagnostic plots such as normal probability curve, residuals vs predicted, outliers and predicted against actual plots. The normal probability plot of the residuals for temperature distribution shown in Figure 2 reveal that the residuals are falling on the straight line, which means the errors are distributed normally. All the above consideration indicates an excellent adequacy of the regression model. The residual values were plotted against the individual run indicating minimum difference between the experimental data and the predicted data as shown in Figure 3.*

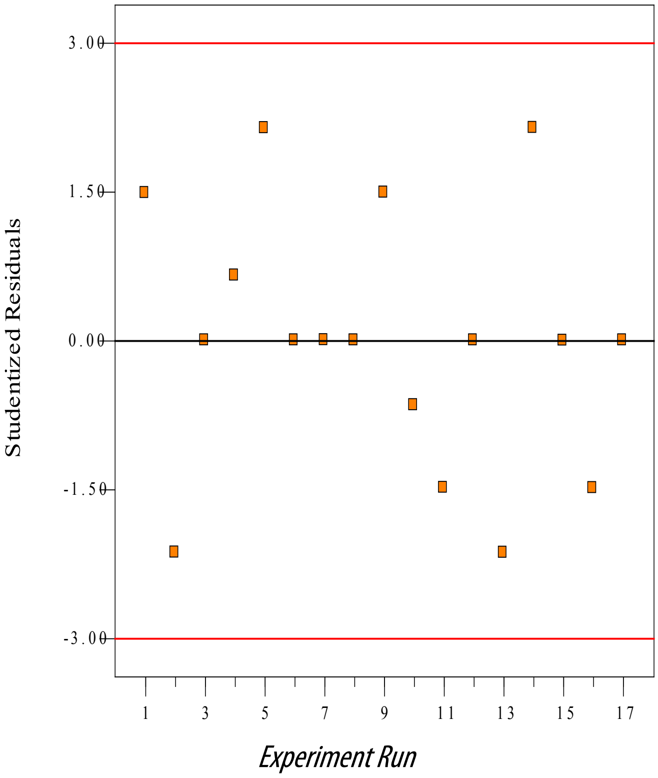

***Figure 3****: Plot of residuals against experimental runs.*

### ***Effect of single factor on temperature distribution***

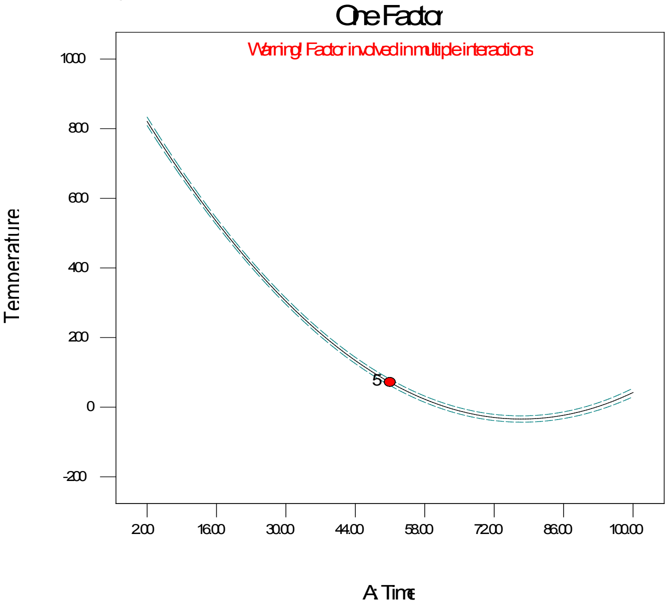

***Figure 4****: Plot of time against the temperature distribution*

*Figure 4 shows the effect of time on the temperature distribution of the quenched steel sample. As time increases from 2 seconds to 100 seconds, temperature distribution reduces.*

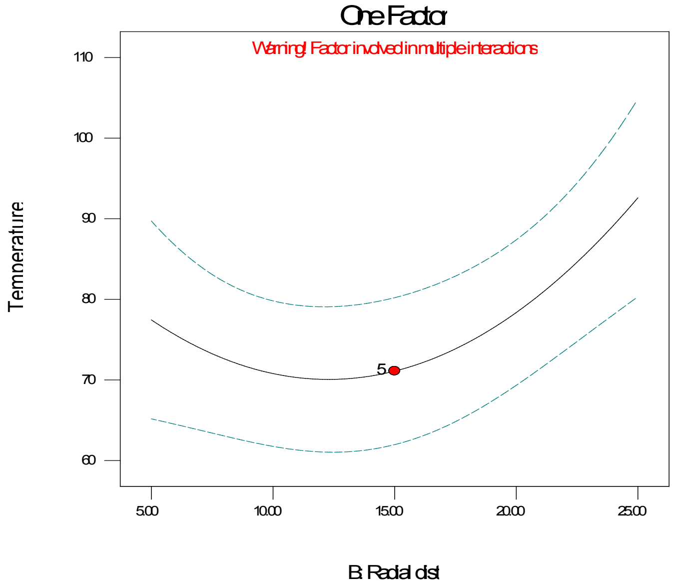

***Figure 5**: Plot of radial distance against the temperature distribution*

*The effect of radial distance on the temperature distribution of steel sample was shown in Figure 5. Temperature distribution decreases from 5mm to 15mm and then increases slightly from 71.5 to 92.6 °C as radial distance increases within the specified range.*

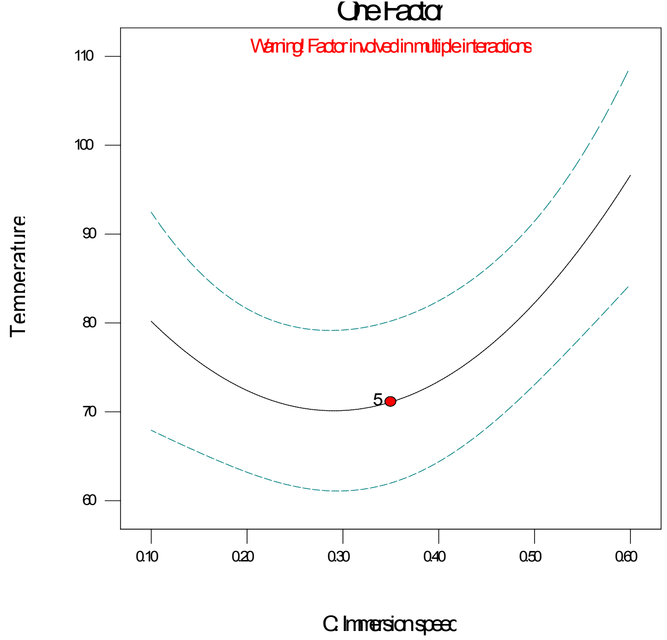

***Figure 6:** Plot of immersion speed against the temperature distribution*

*Figure 6 shows the effect of immersion speed on the temperature distribution of the quenched steel sample. Temperature distribution decreases as immersion speed increases from 0.10 m/s to 0.35m/s but becomes increasing as immersion speed further increases to 0.60m/s.*

## *CONCLUSION*

*This paper has described the use of design of experiments (DOE) for conducting experiments on quenching of steel sample in oil medium. A quadratic model was developed for predicting temperature distribution of steel sample AISI1020 using response surface methodology (RSM). The model developed was validated giving a R-squared value of 0.9997, adjusted R-squared of 0.9993 and predicted R-squared of 0.9953. The model was satisfactory at 99% accuracy. The effects of the factors on the temperature distribution were investigated. Time is the factor that has greater influence on temperature distribution, followed by immersion speed and the least effect was seen on radial distance. The model developed can be used for process behavior prediction for performance measure, for process optimization and for training tools for operators in industrial application.*

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