OPTIMIZATION OF STEEL HARDNESS USING NANOFLUIDS QUENCHANTS

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ABSTRACT

The goal of this study is to specify the optimal factors for the hardening process (tempering temperature, the percentage of nanoparticles, type of base media, nanoparticles type and tempering time) in order to maximize the hardness of medium carbon steel by using Taguchi technique. An (L₁₈) orthogonal array was chosen for the design of the experiment. The optimum process parameters were determined by using signal-to-noise ratio(larger is better) criterion. The important levels of process parameters on hardness were obtained by using analysis of variance which applied with the help of (Minitab17) software to investigate the effect of parameters on the hardness. Percentage of volumetric fractions of nanoparticles with three different levels (0.01, 0.03 and 0.08%) was prepared by dispersing nanoparticles that are (α-Al₂O₃, TiO₂ and CuO) with base fluids (De-ionized water, salt solution, and engine oil). Medium carbon steel specimens were suffered to hardening and tempering heat treatment process. Tempering temperature was (400°C, 550°C) for (30,45 and 60 minutes). Results ended up with a conclusion that tempering temperature (400°C) had the major influence on hardness behavior then type of nanoparticles (TiO₂) followed by time tempering (30min) then base media (salt solution) and finally volume fraction of nanoparticles (0.03%).

KEYWORDS: Nanofluids, quenching, hardness, Taguchi technique.
1. INTRODUCTION
Heat transfer is the driving event on the quenching process, where the specimen is heated to a required temperature and then immersed into the quenching medium. The hot metal immersed to be cooled with different stages. Due to high temperatures, a stable vapor film is formed around the surface of the component. In this stage, heat transfer is very slow because the vapor film acts as an insulator and occurs by radiation through the vapour phase. Then the surface temperature of the metal starts to reduce; simultaneously the vapor film starts to collapse. Now nucleate boiling starts due to the contact of the quenching medium with the metal surface. This effect is characterized by violent bubble formation as the heat is rapidly removed from the metal due to the maximum heat transfer. Here the quenching medium plays an important role to conduct the heat (Baskaran et al., 2016). One of the technical challenges of quenching as a heat treatment process is to select a quenchant medium that could minimize or eliminate these side effects while at the same time provide an interface for heat to be transfer from the work piece to the medium in order to produce the desired properties (Chaves, 2001; Herring, 2010). This study employs nanofluids as quenchants. Nanofluids is the result of dispersion of nanosized materials such as nanoparticles, nanofibers, nanotubes, nanowires, nanorods, nanobubbles or nanosheets in the base fluid like water, oil, acetone, heat transfer fluids, polymer solutions, bio-fluids and etc. Scientist Choi of Argonne Laboratory (USA) successfully prepared nanofluid in (1995) (Mukherjee and Paria, 2013). Nanoparticles are in dimension range of (1-100 nm). Nanoparticles show many different properties than parent material due to increase in surface area to volume ratio (1000 times larger than microparticles). So, nanofluids enhance many thermo-physical properties such as thermal conductivity (Taylor et al., 2013). There are numerous researches on the superior heat transfer properties of nanofluids, especially on the thermal conductivity. (Hwang et al., 2006; Yu et al., 2009; Mintsa et al., 2009) observed an important improvement of the nanofluids’ thermal conductivity compared to conventional coolants. (Park et al., 2004) experimented with copper spheres quenched in nanofluids with alumina nanoparticles at (5–20 %vol.) and sub-cooling at(293–353 °C). Through this experiment, the nanofluids have a low boiling rate as compared with pure water. Furthermore, their investigation showed that the film boiling stage was by-passed to rapid cooling on successive quenching with unwashed spheres. The researchers have concluded that the stable vapour film was prevented due to nanoparticle deposition on the sphere surface. The present study aims to get an optimized effect of quenching media parameters (concentration of nanofluids, type of the nanofluids, tempering temperature, tempering time and type of the base media) on the hardness of medium carbon steel to obtain a maximum of the hardness.
2. EXPERIMENTAL PROCEDURE

2.1. Materials

The following materials were used in the nanofluids synthesis:

Nano titanium dioxide (TiO$_2$) powder, nano aluminum oxide($\alpha$-Al$_2$O$_3$) powder and copper oxide (CuO) nanoparticle (supplied by Zhengzhou Dongyao nano materials Co.LTD.). The properties of these nanoparticles are given on Table 1. Those materials were added to base media (Deionized water, Salt solution (NaCl+water) and Engine oil). Sodium lauryl sulfate as a surfactant was used.

| Nanoparticle material | Average Particle Size (nm) | Purity (%) | Specific surface area (m$^2$/g) | bulk density (g/cm$^3$) | True density (g/cm$^3$) | Crystal form | Color |
|-----------------------|----------------------------|------------|--------------------------------|------------------------|------------------------|--------------|-------|
| $\alpha$-Al$_2$O$_3$  | 50                        | >99.99     | 160.1                          | 0.916                  | 3.91                   | Y            | white |
| TiO$_2$               | 20                        | >99.9      | 220                            | 0.25                   | 3.9                    | Cube         | white |
| CuO                   | 50                        | >99.9      | 120                            | 0.30-0.45              | 6.40                   | Sphere       | black |

2.2. Nanofluid Preparation

In this research, eighteen types of nanofluid are prepared[($\alpha$-Al$_2$O$_3$/ Deionized water), ($\alpha$-Al$_2$O$_3$/ salt solution), ($\alpha$-Al$_2$O$_3$/ engine oil)], [(TiO$_2$/ De ionized water),(TiO$_2$/ salt solution), (TiO$_2$/ engine oil)], [(CuO / De ionized water), (CuO / salt solution), (CuO / engine oil)] with volume fractions of (0.01, 0.03 and 0.08%). In this paper, nanofluid was prepared by two step method where the given nanoparticle is mixed to the base fluid to obtain a suspension. The quantity of nanoparticles required for preparation of nanofluids is calculated using the law of mixture formula. The mass of nanoparticles ($M_{np}$) and base fluid ($M_{bf}$) are measured with the balance of (0.0001 g) an accuracy. The weight percentage ($\phi$) can be calculated by using Eq (1).

$$\phi = \frac{M_{np}/\rho_{np}}{\frac{M_{np}}{\rho_{np}} + M_{bf}}$$

Where:

$\phi$: volume fraction.

$M_{np}$: mass of nanoparticle (g).

$\rho_{np}$: density of the nanoparticle(g/L).

$M_{bf}$: mass of the base fluid (g).
ρ_{bf}: density of the base fluid (g/L). (Hussein et al., 2013)[10]

A mechanical stirrer was used to achieve a homogeneously dispersed solution, as shown in Fig. (1-C). This method was based on (Han and Rhi, 2011; Mahendran et al., 2012) [11] [12]. After preparing the proper mix of the nanoparticles and fluids by a mechanical stirrer, nanoparticles are dispersed in fluids using magnetic stirrer Fig. (1-A). During the process, Sodium Dodecyl Sulphate (SDS) surfactant is added to the solution in proper proportions to ensure the stability of nanofluid. For various purposes, sound energy is used to agitate the particles in a nanofluid. This process is known as sonication. By breaking intermolecular interaction, sonication is also used for speed up the dissolution. Sonication is more useful when the magnetic stirring was not much effective for a given sample. For nanoparticles which were not evenly dispersing in liquids, sonication is most preferable. The sonication process is achieved in two steps were:

A- Initially Sonicate the mixture continuously for (30 min) with sonicator to obtain a uniform dispersion of nanoparticles in fluids, this process is achieved with an ultrasonic mixer (LUC – 410(50 Hz,400W)) that shown in Fig. (1-B).

B-Sonicate the mixture continuously for (90 min) with probe sonicator that shown in Fig. (1-D).

Fig. 1. A-Magnetic stirrer, B- Ultrasonic or bath sonicator C-Electrical blender, D-Ultra sonicator probe sonicator.
2.3. The material of the research specimens

In this research, medium carbon steel has been used as research specimens. The chemical composition analysis of the specimens was carried out at the (Specialized Institute for Inspection and Engineering Qualifying) by x-ray fluorescent. The chemical composition of the carbon steel is given in Table 2.

Table 2. The chemical composition of medium carbon steel specimens.

| Element | C% | Si% | Mn% | P%  | S%  | Cr% | Mo% | Ni% | Al% | Cu% | Fe% |
|---------|----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Composition % | 0.583 | 1.76 | 0.790 | 0.0197 | 0.0035 | 0.0351 | 0.002 | 0.0135 | 0.0264 | 0.033 | Bat |

2.4. Specimen preparations

First of all, for the experiment, is the specimen preparation. The fifty-four steel samples were machined by using (CNC) machine to standard dimensions according to (ASTM E8), then the specimens were grinded and polished.

2.5. Heat Treatment process

Eighteen types of heat treatments were performed, these were quenched and tempered according to the Table 3. Quenching experiment was performed to harden the medium carbon steel. The process involved putting the red hot iron directly into a liquid medium. Firstly, all specimens were heated-up to an austenitizing temperature in an electric furnace (carbolite cwf 1200 muffle furnace). The specimens were held at (900°C) for sufficient time (approximately an hour) to ensure uniformity of temperature throughout the entire volume to achieve a homogeneous structure of austenite. This was followed by the quenching treatment where each group of samples was quenched in different quenching mediums (nanofluids). Then the samples were subjected to tempering process. Tempering process, consists of reheating quenched steel to a suitable temperature below the transformation temperature (400 and 550 °C) with a soaking time were (30, 45 and 60min) and then allowed to cool down gradually.

2.6. Hardness testing

Before hardness tests were performed, all the heat treated specimen surfaces were ground and polished for hardness measurement. Hardness was tested on all samples using (Jesus Miranda Rockwell hardness tester), verified in accordance with (ASTM E18-14a). Rockwell Hardness test was carried out at room temperature to measure the hardness of the medium carbon steel specimens in (C scale). For each sample, four measurements were taken covering the whole surface of the specimen and averaged was taken as final hardness results.
3. **TAGUCHI METHOD**

This is a statistical method also named as robust design method which has its wide applications in most of the fields in recent times. This is a method developed by (Genichi Taguchi) to improve the quality of all the manufactured goods in all the industries (Madhoo and Shilpa, 2017). The researchers introduced a unique concept known as Orthogonal Array which tries to reduce the number of experimentation based on the trials by considering certain control parameters (Thyla et al., 2015). Orthogonal Array provides a minimum number of experimentations and Taguchi’s Signal to Noise ratio serves to give optimum results which are based on the selection of the parameter (Shilpa and Naidu, 2010; Shilpa and Naidu, 2012). The main application of this Taguchi’s method is implemented in the design of experiments (DOE) (Shilpa, M. and Naidu, 2014).

The Signal to Noise ratio can be calculated for three categories as below:

A. LARGER THE BETTER (LTB)

\[ \frac{S}{N} = -10 \log_{10} \left( \frac{1}{n} \sum \frac{1}{Y_i^2} \right) \]

B. SMALLER THE BETTER (STB)

\[ \frac{S}{N} = -10 \log_{10} \left( \sum \frac{Y_i^2}{n} \right) \]

C. NOMINAL THE BEST (NTB)

\[ \frac{S}{N} = 10 \log_{10} \left( \sum \frac{Y_i^2}{S^2} \right) \]  
(Madhoo and Shilpa, 2017) [13]

Where:

Y: results of experiments, observations or quality

N: Number of trials of repetitions.

S: the variance

4. **SELECTION OF CONTROL FACTORS AND LEVELS**

An appropriate orthogonal array for these experiments was selected. Here there are (5) factors with (3) levels hence except tempering temperature with two levels as shown in the Table 3.

For this experimental (L_{18}) orthogonal array is chosen. The (L_{18}) orthogonal array has 18 rows corresponding to the number of tests. Table 4 shows the 18 experiments supported (L_{18}) orthogonal array and their corresponding measured hardness. Replication technique has been adopted to a void inaccuracy as shown in the Table 4.
Table 3. Control factors and their levels.

| Symbol | Control factors       | Levels | Unit |
|--------|----------------------|--------|------|
| A      | Tempering temperature | 400    | 550  | °C   |
| B      | Concentration media  | 0.01%  | 0.03%| 0.08%| ---- |
| C      | base media           | Deionized water | Salt solution | Engine oil | ---- |
| D      | Nano particles type  | αAl₂O₃ | TiO₂ | CuO  | ---- |
| E      | Tempering time       | 30     | 45   | 60   | min  |

5. RESULTS AND DISCUSSIONS

5.1. S/N ratios analysis

The influence of control parameters on hardness was evaluated using (S/N) ratio response analysis. The hardness characteristic selected was (larger is the better type) and the same type of response was used for signal to noise ratio which is given above. The (S/N) ratio response was analyzed using the Equation (2) for all fifty-four tests and presented in Table 4. The plots in Figs. 2 and 3 shows the variation of individual response with the five parameters; tempering temperature, the volume fraction of nanoparticles, type of base media, type of nanoparticles and time tempering separately. The main effect plots are used to determine the optimal design conditions to obtain high hardness.

Table 4. Signal to Noise Ratio for the controlling factors considering Hardness.

| Expt. | Parameters   | Trail (1) | Trail (2) | Trail (3) | S/N Ratio     |
|-------|--------------|-----------|-----------|-----------|---------------|
| A     | B            | C         | D         | E         |               |
| 1     | 400          | 0.01      | Deionized water | α-Al₂O₃ | 30 | 61 | 61 | 60 | 35.658210184 |
| 2     | 400          | 0.01      | Salt Solution | TiO₂    | 45 | 58 | 58 | 58 | 35.268559871 |
| 3     | 400          | 0.01      | Engine oil  | CuO     | 60 | 56 | 57 | 57 | 35.065644549 |
| 4     | 400          | 0.03      | Deionized water | α-Al₂O₃ | 45 | 55 | 55 | 55 | 34.807253790 |
| 5     | 400          | 0.03      | Salt Solution | TiO₂    | 60 | 59 | 58 | 59 | 35.366980613 |
| 6     | 400          | 0.03      | Engine oil  | CuO     | 30 | 57 | 57 | 57 | 35.117497113 |
| 7     | 400          | 0.08      | Deionized water | TiO₂    | 30 | 56 | 58 | 59 | 35.212332444 |
| 8     | 400          | 0.08      | Salt Solution | CuO     | 45 | 58 | 58 | 58 | 35.268559871 |
| 9     | 400          | 0.08      | Engine oil  | α-Al₂O₃ | 60 | 53 | 52 | 54 | 34.482424494 |
| 10    | 550          | 0.01      | Deionized water | CuO     | 60 | 41 | 41 | 41 | 32.255677134 |
| 11    | 550          | 0.01      | Salt Solution | α-Al₂O₃ | 30 | 42 | 44 | 45 | 32.792208535 |
12 550 0.01 Engine oil TiO₂ 45 45 44 47 33.118622012
13 550 0.03 Deionized water TiO₂ 60 47 45 45 33.186538668
14 550 0.03 Salt Solution Cuo 30 45 45 45 33.064250276
15 550 0.03 Engine oil α-Al₂O₃ 45 44 45 44 32.933149227
16 550 0.08 Deionized water Cuo 45 42 42 42 32.464985808
17 550 0.08 Salt Solution α-Al₂O₃ 60 45 45 45 33.064250276
18 550 0.08 Engine oil TiO₂ 30 48 50 46 33.609727610

Table 5. Response table for (S/N) ratio.

|          | Factor A   | Factor B   | Factor C   | Factor D   | Factor E   |
|----------|------------|------------|------------|------------|------------|
| Level 1  | 35.138607  | 34.026487  | 33.93083   | 33.95625   | 34.24237   |
| Level 2  | 32.9432677 | 34.0792783 | 34.13747   | 34.29379   | 33.97686   |
| Level 3  | 34.0170468 | 34.05451   | 33.87277   | 33.90359   |
| Delta    | 2.19533926 | 0.06223153 | 0.206635   | 0.421024   | 0.338785   |
| Rank     | 1          | 5          | 4          | 2          | 3          |

Table 6. Response table of hardness for mean.

|          | Factor A   | Factor B   | Factor C   | Factor D   | Factor E   |
|----------|------------|------------|------------|------------|------------|
| Level 1  | 57.18518519| 50.88888889| 50.33333333| 50.277778  | 52         |
| Level 2  | 44.44444444| 50.94444444| 51.38888889| 52.222222  | 50.44444444|
| Level 3  | 50.61111111| 50.72222222| 51.0555556 | 50         |
| Delta    | 12.74074074| 0.33333333| 0.38888889 | 1.9444444  |
| Rank     | 1          | 5          | 4          | 3          | 2          |
Fig. 2. Main effects plot for (S/N) ratios – Hardness.
Fig. 3. Main effects plot for means—Hardness.
From the response Table 5 and Fig. 2, it is clear that tempering temperature is the most influencing factor followed by nanoparticles type then tempering time, subsequently type of base media and percentage of nanoparticles is located in ranked last. Also, the results show that the effect of the salt solution as base media was higher than deionized water and engine oil, in addition to the (0.03%) volume fraction of nanoparticles and (TiO₂) nanoparticles have higher effects on the hardness.

5.2. Analysis of variance

The Table 7 shows analysis of variance for the hardness value of the medium carbon steel. From Table 7, it is observed that the tempering temperature, percentage of nanoparticles, base media, type of nanoparticles and tempering time affect the hardness of medium carbon steel. The last column of the Table 7 indicates the percentage contribution of each other on the total variation indicating their degree of effect on the result. It can be observed from the (ANOVA) table that the tempering temperature (89.880%) was the most significant parameter on the hardness of medium carbon steel followed by type of nanoparticles (2.234%) then tempering time (1.628%) next base media (0.420%) and the least affected was percentage of volume fraction of nanoparticles (0.0471%). The pooled error associated in the (ANOVA) table was approximately about (5.789%) for hardness. This approach gives the variation of means and variance to absolute values considered in the experiment and not the unit value of the variable.

Table 7. Results of the (ANOVA).

| Source  | DF | SeqSS | AdjMS | F-value | P-Value | Percentage of contribution |
|---------|----|-------|-------|---------|---------|---------------------------|
| A       | 1  | 2191  | 2191  | 683.13  | 0       | 89.88003199               |
| B       | 2  | 1.15  | 0.57  | 0.18    | 0.837   | 0.0471666909             |
| C       | 2  | 10.26 | 5.13  | 1.6     | 0.214   | 0.420810861             |
| D       | 2  | 54.48 | 27.24 | 8.49    | 0.001   | 2.234481061             |
| E       | 2  | 39.7  | 19.85 | 6.19    | 0.004   | 1.62828374             |
| Error   | 44 | 141.2 | 3.21  |         |         | 5.789225437           |
| Lack –of-Fit | 8 | 111.8 | 13.98 | 17.15   |         |                         |
| Pure Error | 36| 29.33 | 0.81  |         |         |                         |
| Total   | 53 | 2438  |       |         | 100     |                         |

5.3. Regression Equation
A Regression model is developed using statistical software (MINITAB17). This model gives the relationship between an independent/predicted variable and a response variable by fitting linear equations to observe data. Regressions equation thus generate correlations between the significant terms obtained from (ANOVA) analysis namely tempering temperature, the percentage of the volume fraction of nanoparticles, base media, type of nanoparticles and tempering time. The regression equations developed for hardness were shown in the Table 8.

Table 8. The regression equations for hardness.

| Quenching media | Type of nano particles | Regression equations |
|-----------------|------------------------|----------------------|
| Deionized water | CuO                    | (HRC) = 92.99 - 0.08494 Tempering temperature - 4.49 % of nanoparticles - 0.0667 tempering time |
| Deionized water | TiO₂                   | (HRC) = 95.27 - 0.08494 Tempering temperature - 4.49 % of nanoparticles - 0.0667 tempering time |
| Deionized water | α-Al₂O₃                | (HRC) = 93.32 - 0.08494 Tempering temperature - 4.49 % of nanoparticles - 0.0667 tempering time |
| Engine oil      | CuO                    | (HRC) = 93.38 - 0.08494 Tempering temperature - 4.49 % of nanoparticles - 0.0667 tempering time |
| Engine oil      | TiO₂                   | (HRC) = 95.65 - 0.08494 Tempering temperature - 4.49 % of nanoparticles - 0.0667 tempering time |
| Engine oil      | α-Al₂O₃                | (HRC) = 93.71 - 0.08494 Tempering temperature - 4.49 % of nanoparticles - 0.0667 tempering time |
| Salt Solution   | CuO                    | (HRC) = 94.04 - 0.08494 Tempering temperature - 4.49 % of nanoparticles - 0.0667 tempering time |
| Salt Solution   | TiO₂                   | (HRC) = 96.32 - 0.08494 Tempering temperature - 4.49 % of nanoparticles - 0.0667 tempering time |
| Salt Solution   | α-Al₂O₃                | (HRC) = 94.38 - 0.08494 Tempering temperature - 4.49 % of nanoparticles - 0.0667 tempering time |

5.4. Model Summary
The summary of the model can be illustrated in Table 9.

Table 9. Model summary.

| S     | R-sq   | R-sq(adj) | R-sq(pred) |
|-------|--------|-----------|------------|
| 1.77579 | 94.05% | 93.15%    | 91.60%     |
The graphs show that the data closely follow the straight lines, denoting a normal distribution. Also, it can be observed from regression equations that the coefficient associated with tempering temperature, Percentage of the volume fraction of nanoparticles, Base media, Type of nanoparticles and tempering time are negative that indicates the hardness of the material decrease with increasing the above parameter.

6. CONCLUSIONS

The approach of (Taguchi’s) robust design method to hardness study led to conclude the following:

A- Taguchi method provides a systematic and efficient methodology for the design and optimization of quenching of nanofluids and tempering heat treatment parameters to maximize hardness with far less effort than would be required for most optimization techniques.

B- From response table for (S/N)ratio with respect to the hardness (LTB) it is clear that tempering temperature is the most significant factor influencing hardness followed by type of nanoparticles then type of nanoparticles next tempering time then type of base media and percentage of volume fraction of the nanoparticles which is the least significant factor.

C- The analysis of variance shows that the percentage contribution of tempering temperature, percentage of volume fraction of nanoparticles, base media, nanoparticles type and tempering time are (89.880%), (2.234%), (1.628%), (0.420%), (0.0471%) respectively.

The d-the pooled error associated with the (ANOVA) analysis was (5.789%) for hardness, and the correlation between the hardness parameters was obtained by multiple linear regression models.
E-The important sequence of optimal conditions for hardness is tempering temperature, type of nanoparticles, tempering time Base media, the percentage of the volume fraction of nanoparticles.

F-The optimal parameters for hardness value are tempering temperature (400°C), (0.03%) volume fraction of nanoparticles, the salt solution as base media, type of nanoparticles (TiO₂) and tempering time (30min).

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