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OPTIMIZATION OF HARDENING PROCESS PARAMETERS USING CHEMICALLY MODIFIED BIOQUENCHANTS VIA TAGUCHI APPROACH

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ABSTRACT

In this research work, Taguchi method was applied to optimize quenching hardness using best combination of hardening process parameters by quenching in chemically modified bioquenchants. High carbon steel samples were hardened by adjusting the process parameters. Several experiments have been conducted based on an orthogonal array L4 with three parameters (austenitizing temperature, austenitizing time and agitation amplitude) at two levels (low and high). Based on the mean response and signal to noise (S/N) ratio, the best optimal parameters setting was arrived for all the bioquenchants used. Confirmation experiments have been carried out to verify the optimized hardness results. The values of the per cent errors for the predicted and experimental results obtained are within the prescribed limit. The bioquenchants used include; epoxidized cottonseed oil (EC), epoxidized-transesterified cottonseed oil (ETC), transesterified cotton seed oil (TC), fresh cotton seed oil (FC), epoxidizedmahogany seed oil (EM), epoxidized-transesterified mahogany seed oil (ETM), transesterified mahogany seed oil (TM) and fresh mahogany seed oil (FM). From the results, maximum hardness values were obtained from all the bioquenchants. This approach is cost-effective, since it reduces the number of experimental trials.

Keywords: Optimization, Taguchi, parameters, hardening process, bioquenchants, chemical modification.

1.0 INTRODUCTION

Heat treaters are encountering an ever-increasing need for practical process design and optimization methods to effectively address quality, cost and production time requirements for thermal treatment of steel parts. Over the last two decades, substantial advances have been made in heat treatment process optimization, now permitting user-friendly and robust means for process engineers, designers, and other heat treatment technical professionals to readily apply powerful statistical techniques to address complex, “real-life” heat treatment challenges (Sims *et al.,* 2017).

Today, many statistical methods are employed by professionals to optimize the process parameters and improve the quality of the components that are manufactured. Statistical design methods such as factorial design, response surface methodology (RSM) and Taguchi methods are now widely used in place of one factor at a time experimental approach. Taguchi techniques have been widely applied for optimization process in material processing (Ghani, 2004; Mandal, 2011).Taguchi design of experiment (DOE) methods incorporates fractional factorial matrixes or orthogonal arrays to minimize the number of experiments required to achieve a given set of performance characteristics (Phillip, 1988). Kumar *et al.,* (2016) investigated heat treatment parameters optimization using Taguchi technique. It was reported from the study that austenitizing temperature is the most significant factor among the hardening process variables. Jie *et al.,* (2014) conducted a study on the effect of quenching parameters on mechanical property of ultra-high strength steel BR1500HS based on Response Surface Methodology (RSM). The finding shows that both austenitizing temperature and soaking time have significant effect on the quenching hardness, tensile strength and per cent elongation. Similarly there was investigation done by Mason and Prevéy (2001) which aimed at optimizing austenite content and hardness in 52100 steel via Taguchi analysis. They concluded that tempering temperature and cold treatment were seen to have the greatest effect on austenite content while austenitizing and tempering temperatures had the greatest influence on the hardness. Determination of appropriate austenitizing temperatures and times for a heat-treating procedure to achieve optimum hardness can appear initially to require extensive, if not prohibitive, experimentation. Fortunately, Taguchi analysis provides an efficient and effective means of achieving these goals (Mason and Prevéy2001).

Limited research had been carried out on the effect of hardening process parameters like austenitizing temperature, austenitizing time and agitation on the quenching hardness of high carbon steel quenched in chemically modified bioquenchants. Therefore, the purpose of this paper is to present an application of Taguchide sign to identify optimum hardness with a particular combination of hardening process parameters.

2.0 EXPERIMENTAL DETAILS

2.1 Materials

The high carbon steel samples used were sourced from trailer leaf spring. The mahogany seed oil was obtained from local market in Numan, Adamawa State, Nigeria. Similarly, the cotton seed oil used was purchased from ABJ Nig. Ltd., Funtua. The mineral oil used in the research work was SAE40.

2.2 Design of Experiment for the Hardening Process

In the event of the hardening process, various operating variables such as austenitizing temperature, soaking time and agitation amplitude were considered and optimized using Taguchi approach. The optimization analysis was run according to the criterion the-larger-the-better .An L4 orthogonal array is chosen for the experimental design based on the operating parameters and their levels. The parameters varied at two levels (Table 1).

Table 1: An L4 orthogonal array for the heat treatment procedure

Austenitizing Temperature (ºC)	Austenitizing time (s)	Agitation Amplitude (mm)
800	30	1.5
800	45	3.0
850	30	3.0
850	45	1.5

The best combination of heat treatment conditions for optimum hardness developed on the quenched samples for each quenching medium obtained from the DOE using MINITAB 16 statistical software are shown in Table 2.

Table 2: Heat treatment conditions for optimum hardness

Quenchant	Austenitizing Temperature (ºC)	Austenitizing time (min)	Agitation Amplitude (mm)
EC	800	30	3
ETC	850	30	1.5
TC	850	30	1.5
FC	800	45	1.5
EM	800	45	1.5
ETM	850	30	3
TM	850	30	3
FM	800	45	3
Mineral oil (SAE40)	800	45	3

2.3 Hardening Process

The best combination of heat treatment conditions for optimum hardness development for each quenching medium is shown in Table 2.These conditions were employed for each quenching medium during the confirmation experiment. Samples were austenitized at the required temperature, soaked and then quickly quenched in the agitated as-received, trans-esterified, epoxidized and epoxidized-trans-esterified cotton and mahogany seed oils and mineral oil. All the quenching media were maintained at room temperature of 27ºC. Laboratory Sieve shaker was used to provide the required agitation during the quenching operation

2.4. Hardness test

The hardness values of the test samples were determined using Vickers hardness machine (MV1-PC model, Serial No.: 07/2012-1329). The test was carried out in accordance with ASTM E18 method. A diamond indenter was used to indent the surface of the test sample by the application of static load of 0.3kgf, which was maintained for fifteen minutes.

Both diagonals of the impression were measured using a lower power graduated microscope. Using the mean value of the diagonal length, the machine gave the hardness value digitally. The procedure was repeated at 2 different points on the test piece and the average values were recorded.

2.5 Microstructural examination

Conventional metallographic preparation procedure was used to prepare samples for microstructural observation. Scanning electron microscope was used to image and study the phases present.

3.0 RESULTS AND DISCUSSION

3.1 Single optimization analysis

The influence of each factor was analysed using response tables. The response tables were obtained using Taguchi method. The mean S/N ratio response in Table 4 and 6 showed that austenitizing temperature is the most powerful parameter that influences the developed hardness of high carbon steel after quenching in ETC, TC and TM. Next to it is agitation amplitude while austenitizing time emerged as the least significant variable affecting the developed hardness. Austenitizing temperature was reported as well by Kumar *et al.,* (2016) to be the most significant factor among the hardening process variables. The likely reason for this trend might be that higher austenitizing temperature causes a raise in hardenability. The improved hardenability, which eventually leads to maximum hardness development after quenching, is strongly connected by the amount of carbon dissolved in the prior austenitic phase. During quenching, however, the undissolved carbides will nucleate pearlite prematurely and act to reduce the hardenability.

In Table 5, the ranking which gives the order of importance of the control factors reveals that hardness developed after quenching in agitation and austenitizing temperature in that order. This could be explained by the fact that maximum hardness would be achieved upon holding the work piece at austenitizing temperature for complete transformation of BCC-ferrite to FCC-austenite to occur and attainment of homogenization. In addition, holding time has considerable impact on the enlargement of austenite grains which in turn has effect on the process of austenite-martensite transformation. Since the pearlite transformation begins at grain boundaries, an increase in the austenite grain size causes a decrease in the critical rate of quenching and hardenability improves (Totten, 2006). Similar results were obtained by Jie*et al.,* (2014).

Figure 1-9 is a plot of the effect of the process parameters on mean S/N ratio of FC, EM and ETM is influenced by austenitizing time.

From Tables 4 and 6, it is evident that agitating EC, FM and SAE40 quenchants contributed majorly for hardness development followed by austenitizing temperature and then soaking time. Agitation plays an important role in the effectiveness of a medium to quench a part. In order to achieve uniform heat transfer throughout the quenching, agitation of a quenching medium is necessary to destabilize the vapour blanket and nucleate boiling.

Table 3: DOE Results from Hardening Process

Bioquenchants	**Expt. No.**	**Factors**			**Response**
		Austenitizing Temperature (ºC)	Austenitizing Time (s)	Agitation Amplitude (mm)	Hardness (HVN)
EC	1	800	30	1.5	582
	2	800	45	3.0	602
	3	850	30	3.0	602
	4	850	45	1.5	560
ETC	1	800	30	1.5	583
	2	800	45	3.0	480
	3	850	30	3.0	635
	4	850	45	1.5	738
TC	1	800	30	1.5	463
	2	800	45	3.0	430
	3	850	30	3.0	476
	4	850	45	1.5	495
FC	1	800	30	1.5	377
	2	800	45	3.0	377
	3	850	30	3.0	326
	4	850	45	1.5	409
EM	1	800	30	1.5	524
	2	800	45	3.0	607
	3	850	30	3.0	385
	4	850	45	1.5	625
ETM	1	800	30	1.5	630
	2	800	45	3.0	523
	3	850	30	3.0	715
	4	850	45	1.5	520
TM	1	800	30	1.5	390
	2	800	45	3.0	419
	3	850	30	3.0	508
	4	850	45	1.5	435
FM	1	800	30	1.5	340
	2	800	45	3.0	415
	3	850	30	3.0	369
	4	850	45	1.5	310
SAE40	1	800	30	1.5	387
	2	800	45	3.0	414
	3	850	30	3.0	400
	4	850	45	1.5	381

Table 4: S/N ratios response table for Hardness of high carbon steel quenched in EC, ETC and TC

	EC				ETC				TC			
Level	1	2	Delta	Rank	1	2	Delta	Rank	1	2	Delta	Rank
Austenitizing Temperature (ºC)	55.45	55.28	0.17	2	54.47	56.71	2.24	1	52.99	53.72	0.73	1
Austenitizing Time (min)	55.45	55.28	0.17	2	55.68	55.49	0.19	3	53.43	53.28	0.15	3
Agitation Amplitude (mm)	55.13	55.59	0.46	1	56.34	54.84	1.50	2	53.60	53.11	0.49	2

Table 5: S/N ratios response table for Hardness of high carbon steel quenched in FC, EM and ETM

	FC				EM				ETM			
Level	1	2	Delta	Rank	1	2	Delta	Rank	1	2	Delta	Rank
Austenitizing Temperature (ºC)	51.53	51.25	0.28	2	55.03	53.81	1.21	3	55.18	55.70	0.52	3
Austenitizing Time (min)	50.90	51.88	0.99	1	53.05	55.79	2.74	1	56.54	54.35	2.19	1
Agitation Amplitude (mm)	51.88	50.90	0.99	1	55.15	53.69	1.47	2	55.15	55.73	0.57	2

Table 6: S/N ratios response table for Hardness of high carbon steel quenched in TM, FM and SAE40

	TM				FM				SAE40			
Level	1	2	Delta	Rank	1	2	Delta	Rank	1	2	Delta	Rank
Austenitizing Temperature (ºC)	52.13	53.44	1.31	1	51.50	50.58	0.91	2	52.05	51.83	0.22	2
Austenitizing Time (min)	52.97	52.61	0.36	3	50.99	51.09	0.11	3	51.90	51.98	0.08	3
Agitation Amplitude (mm)	52.30	53.28	0.99	2	50.23	51.85	1.62	1	51.69	52.19	0.50	1

The results obtained from hardness test gave room for the assessment of the effect of austenitizing temperature, austenitizing time and degree of agitation on the hardness of high carbon steel (Table 3-6).

This agrees with the existing records (Totten, 2006). Furthermore, agitation during quenching produces smaller, more frequent bubbles during the boiling stage, which in turn, creates faster rates of heat transfer throughout the part. Similar observation was reported by Mackenzie and Totten (1989).

Figure 1-9 is a plot of the effect of the process parameters on mean S/N ratio of hardness of High carbon steel quenched in the bioquenchants and SAE40.

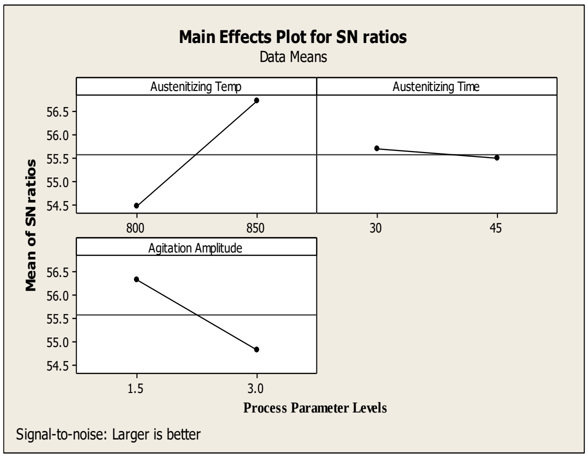

Figure 1: *Impact of process parameters on mean S/N ratio of Hardness of High carbon steel quenched in ETC*

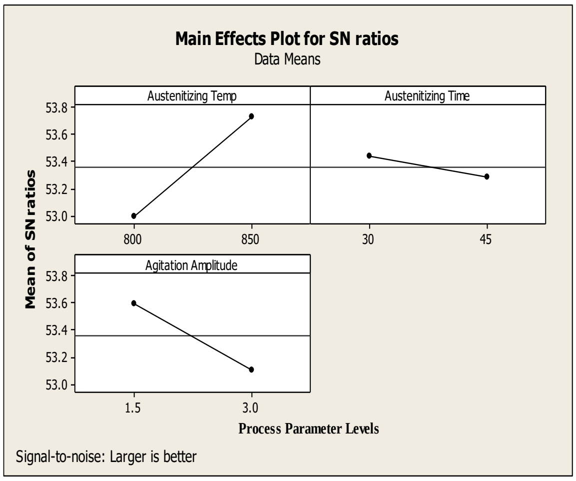

Figure 2: *Impact of process parameters on mean S/N ratio of Hardness of High carbon steel quenched in TC*

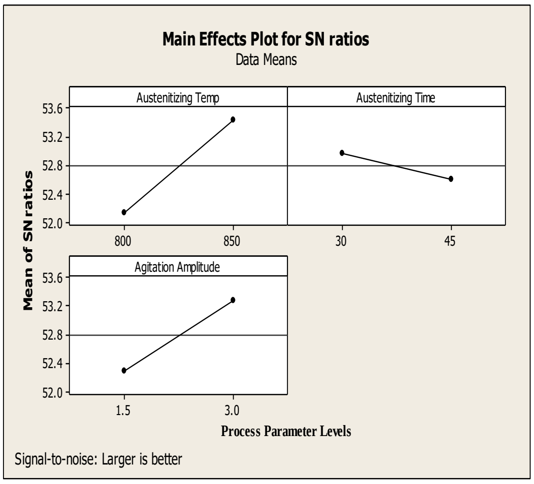

Figure 3: *Impact of process parameters on mean S/N ratio of Hardness of High carbon steel quenched in TM*

The plots in Figure 1-3 permit the appraisal of the influence of hardening process parameters on the hardness. Accordingly, the level of a parameter with the highest S/N ratio gives the optimal level. The optimal process parameter combination for development of maximum hardness in ETC and TC was found to be at factor levels 2 (850 ^{o}C), 1 (30 min) and 1 (1.5 mm) for austenitizing temperature, austenitizing time and agitation amplitude respectively. On the
other hand, process parameters at levels 2 (850 ^{o}C), 1 (30 min) and 2 (3 mm) for austenitizing temperature, austenitizing time and agitation amplitude respectively are observed to produce optimum hardness for quenching in TM. It is noteworthy to mention that, among the hardening process parameters, austenitizing temperature has the greatest influence on the hardness.

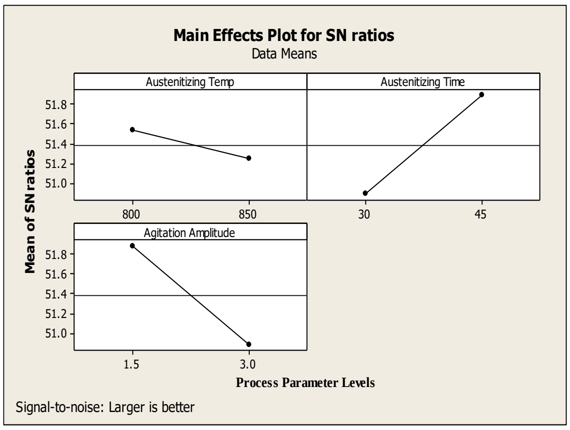

Figure 7: *Impact of process parameters on mean S/N ratio of Hardness of High carbon steel auenched in EC*

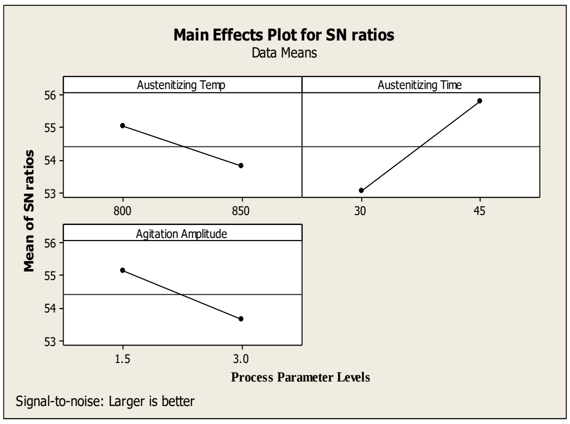

Figure 8: *Impact of process parameters on mean S/N ratio of Hardness of High carbon steel quenched in FM*

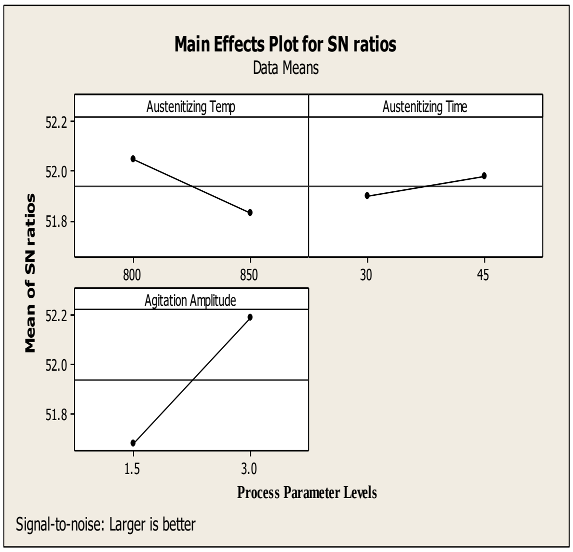

Figure 9: *Impact of process parameters on mean S/N ratio of Hardness of High carbon steel quenched in SAE40*

This is similar to the report of Mason and Prevéy(2001).
According to the S/N response graphs (Figure 7-9), it is evident that quenching in EC, FM and SAE40 give hardness which decreases gradually with increase in austenitizing temperature. Similarly, quenching in FM and SAE40 developed quench hardness which rise mildly with increase in the austenitizing time. In sharp contrast toquenching in EC, a steady decline in quench hardness with increase in soaking time is observed. Furthermore, increase in agitation amplitude while quenching in EC, FM and SAE40 led to marked improvement in the hardness of the work piece. The optimum condition identified was austenitizing temperature of 800 ^{o}C, austenitizing time of 45 min and agitation amplitude of 3 mm. However, it is worth noting that work piece soaked for 30 min (instead of 45 min) and then quenched in EC resulted in maximum hardness development.

3.2. Confirmation Tests

Confirmation experiment was done at optimum level of the method parameters. The predicted S/N ratios and the mean values (predicted hardness values) were calculated using the optimum setting of the method parameters and the model equations. Thus, the predicted mean S/N ratios and the mean values, for quenching hardness in all the bioquenchants and SAE40 are estimated with the help of the following prediction model equations (Equation 1-18);

$$\bar{Y}_{EC} = Y_{EC} + (\overline{A_1} - Y_{EC}) + (\overline{B_1} - Y_{EC}) + (\overline{C_2} - Y_{EC}) \quad (1)$$

$$\bar{Y}_{ETC} = Y_{ETC} + (\overline{A_2} - Y_{ETC}) + (\overline{B_1} - Y_{ETC}) + (\overline{C_1} - Y_{ETC}) \quad (2)$$

$$\bar{Y}_{TC} = Y_{TC} + (\overline{A_2} - Y_{TC}) + (\overline{B_1} - Y_{TC}) + (\overline{C_1} - Y_{TC}) \quad (3)$$

$$\bar{Y}_{FC} = Y_{FC} + (\overline{A_1} - Y_{FC}) + (\overline{B_2} - Y_{FC}) + (\overline{C_1} - Y_{FC}) \quad (4)$$

$$\bar{Y}_{EM} = Y_{EM} + (\overline{A_1} - Y_{EM}) + (\overline{B_2} - Y_{EM}) + (\overline{C_1} - Y_{EM}) \quad (5)$$

$$\bar{Y}_{ETM} = Y_{ETM} + (\overline{A_2} - Y_{ETM}) + (\overline{B_1} - Y_{ETM}) + (\overline{C_2} - Y_{ETM}) \quad (6)$$

$$\bar{Y}_{TM} = Y_{TM} + (\overline{A_2} - Y_{TM}) + (\overline{B_1} - Y_{TM}) + (\overline{C_2} - Y_{TM}) \quad (7)$$

$$\bar{Y}_{FM} = Y_{FM} + (\overline{A_1} - Y_{FM}) + (\overline{B_2} - Y_{FM}) + (\overline{C_2} - Y_{FM}) \quad (8)$$

$$\bar{Y}_{SAE} = Y_{SAE} + (\overline{A_1} - Y_{SAE}) + (\overline{B_2} - Y_{SAE}) + (\overline{C_2} - Y_{SAE}) \quad (9)$$

$$\bar{y}_{EC} = y_{EC} + (\overline{a_1} - y_{EC}) + (\overline{b_1} - y_{EC}) + (\overline{c_2} - y_{EC}) \quad (10)$$

$$\bar{y}_{ETC} = y_{ETC} + (\overline{a_2} - y_{ETC}) + (\overline{b_1} - y_{ETC}) + (\overline{c_1} - y_{ETC}) \quad (11)$$

$$\bar{y}_{TC} = y_{TC} + (\overline{a_2} - y_{TC}) + (\overline{b_1} - y_{TC}) + (\overline{c_1} - y_{TC}) \quad (12)$$

$$\bar{y}_{FC} = y_{FC} + (\overline{a_1} - y_{FC}) + (\overline{b_2} - y_{FC}) + (\overline{c_1} - y_{FC}) \quad (13)$$

$$\bar{y}_{EM} = y_{EM} + (\overline{a_1} - y_{EM}) + (\overline{b_2} - y_{EM}) + (\overline{c_1} - y_{EM}) \quad (14)$$

$$\bar{y}_{ETM} = y_{ETM} + (\overline{a_2} - y_{ETM}) + (\overline{b_1} - y_{ETM}) + (\overline{c_2} - y_{ETM}) \quad (15)$$

$$\bar{y}_{TM} = y_{TM} + (\overline{a_2} - y_{TM}) + (\overline{b_1} - y_{TM}) + (\overline{c_2} - y_{TM}) \quad (16)$$

$$\bar{y}_{FM} = y_{FM} + (\overline{a_1} - y_{FM}) + (\overline{b_2} - y_{FM}) + (\overline{c_2} - y_{FM}) \quad (17)$$

$$\bar{y}_{SAE} = y_{SAE} + (\overline{a_1} - y_{SAE}) + (\overline{b_2} - y_{SAE}) + (\overline{c_2} - y_{SAE}) \quad (18)$$

Where: $\bar{Y}_{EC}$, $\bar{Y}_{ETC}$, $\bar{Y}_{TC}\bar{Y}_{FC}$, $\bar{Y}_{EM}$, $\bar{Y}_{ETM}\bar{Y}_{TM}$, $\bar{Y}_{FM}$, $\bar{Y}_{SAE}$ and $\bar{y}_{EC}$, $\bar{y}_{ETC}$, $\bar{y}_{TC}\bar{y}_{FC}$, $\bar{y}_{EM}$, $\bar{y}_{ETM}$, $\bar{y}_{TM}$, $\bar{y}_{FM}$, $\bar{y}_{SAE}$ represent the predicted S/N ratio and mean value for quenching in EC, ETC, TC, FC, EM, ETM, TM, FM, SAE40 respectively at optimum condition.

$\overline{A_1 A_2}$, $\overline{B_1}$, $\overline{B_2}$, $\overline{C_1}$, $\overline{C_2}$ and $\overline{a_1}$, $\overline{a_2}$, $\overline{b_1}$, $\overline{b_2}$, $\overline{c_1}$, $\overline{c_2}$ are the mean responses of S/N ratio and mean values for factors at designated optimum levels respectively. AiBiCi represent the respective degree/magnitude/amount of austenitizing temperature, austenitizing time and agitation at level i. Note that their corresponding mean responses of S/N ratio and mean values carries bar (for the mean values they are represented as small letters). Y_{EC}, Y_{ETC}, $Y_{TC}Y_{FC}$, Y_{EM}, $Y_{ETM}Y_{TM}$, Y_{FM}, Y_{SAE} and y_{EC}, y_{ETC}, $y_{TC}y_{FC}$, y_{EM}, $y_{ETM}y_{TM}$, y_{FM}, y_{SAE} indicate the average of the S/N ratios and mean values for EC, ETC, TC, FC, EM, ETM, TM, FM, SAE40 respectively.

The predicted hardness values based on the obtained model are illustrated in Table 7. The table compares the predicted values with the experimental results obtained after quenching in all the bioquenchants and SAE40 at the optimized process parameters. The per cent errors of the Taguchi-predicted results with respect to the experimental values as reference are also depicted in the Table.

Table 7: Analysis of Confirmation Experiment for Developed Hardness

Bioquenchants /Oil	**Prediction**		**Experimental**		**Prediction Error (%)**		**Improvement**	
	Mean value (HVN)	S/N ratio (dB)	Mean value (HVN)	S/N ratio (dB)	Mean value error	S/N ratio error	Value	(%)
EC	613	55.76	608	55.68	0.822	0.144	6.00	1.023
ETC	738	57.55	741	57.40	0.405	0.568	3.00	0.493
TC	502	54.04	504	54.05	0.397	0.019	9.00	1.931
FC	418.5	52.51	432	52.71	3.125	0.379	23.00	6.179
EM	685.5	57.13	632	56.01	8.465	2.000	7.00	1.308
ETM	715	57.09	715	57.09	0.000	0.000	-	-
TM	508	54.12	508	54.12	0.000	0.000	-	-
FM	415	52.36	414	52.34	0.242	0.038	-	-
SAE40	414	52.34	415	52.36	0.241	0.038	1.00	0.253

There is improvement in the response as evident in Table 7. However, when ETM, TM and FM were used as quenching medium, there was no improvement in the developed hardness. it is observed that per cent errors based on the S/N ratio values achieved for quenching in TC, ETM, TM, FM and SAE40 are less than 0.1 %, whereas that based on the mean values are below 0.4 %. Similarly, the S/N ratio per cent errors of 0.144, 0.568, 0.379 and 2 % are recorded for the quenching hardness in EC, ETC, FC and EM respectively. Likewise, the mean values per cent errors of developed hardness after quenching in EC, ETC, FC and EM are found to be 0.822, 0.405, 3.125 and 8.465 % respectively

As seen, model results are fairly well fitted with the experimental ones. Since the highest per cent error got (8.465 %) is less than the tolerable range of 10 % (Siddhartha *et al.,* 2011), therefore, it can be concluded that model Equations (1) - (18) are valid and reliable for predicting the quenching hardness in hardening of high carbon steel using EC, ETC, TC, FC, EM, ETM, TM, FM and SAE40 as quenching medium. However, Jie*etal.,* (2014) carried out single objective optimization on hardness of ultra-high strength steel BR1500HS based on response surface methodology (RSM) method. Their findings revelled that peak value of quenching hardness of 52.29 HRC was achieved at austenitizing temperature of 875.4 ^{o}C.

A detailed microstructural study on the quenched samples during optimization was undertaken. As seen in Plates 1-2, the microstructures mostly consist of martensite. Plates 1b and 2b show that the microstructure of the steel samples quenched in ETC and ETM consists of fine martensite. This explains the dramatic improvement in hardness observed by the samples quenched in the ETC and ETM. Notwithstanding, relatively fine martensite structure is observed in samples quenched in EC and EM. Higher amount of retained austenite with coarse martensitic laths is seen in the samples quenched in TC, TM, FC and FM (Plates1[c-d] and 2[c-d]). This accounts for the inferior hardness obtained from samples quenched in the TC, TM, FC and FM. Similar observations were made by Güler*et al.,* (2014).

4.0 CONCLUSION

The experiments carried out demonstrated that, depending on the cooling rate offered by the quenchant, austenitizing temperature, austenitizing time and agitation have significant influence on the hardness developed in the hardening of high carbon steel.

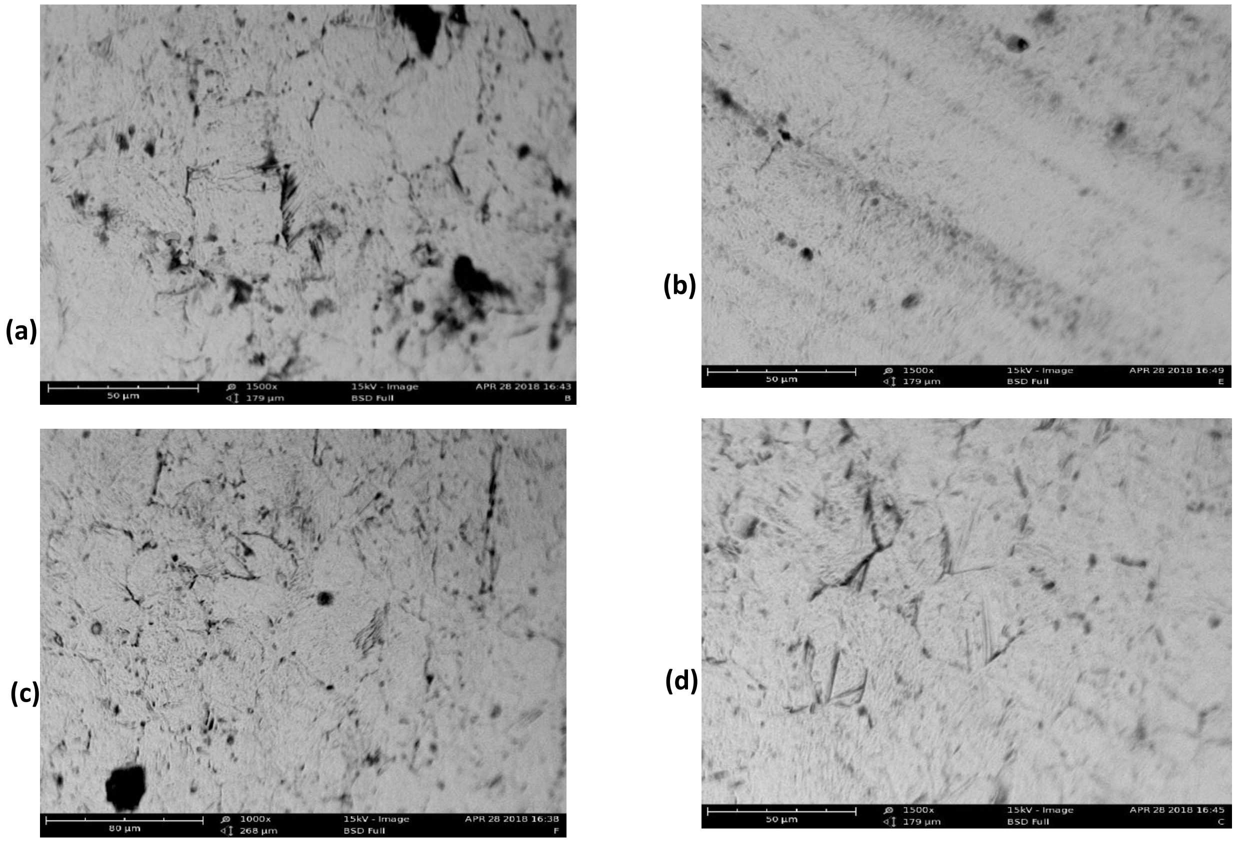

Plate 1: *SEM Image of sample quenched in a) EC, b) ETC, c) TC and d) FC at optimum parameters setting*

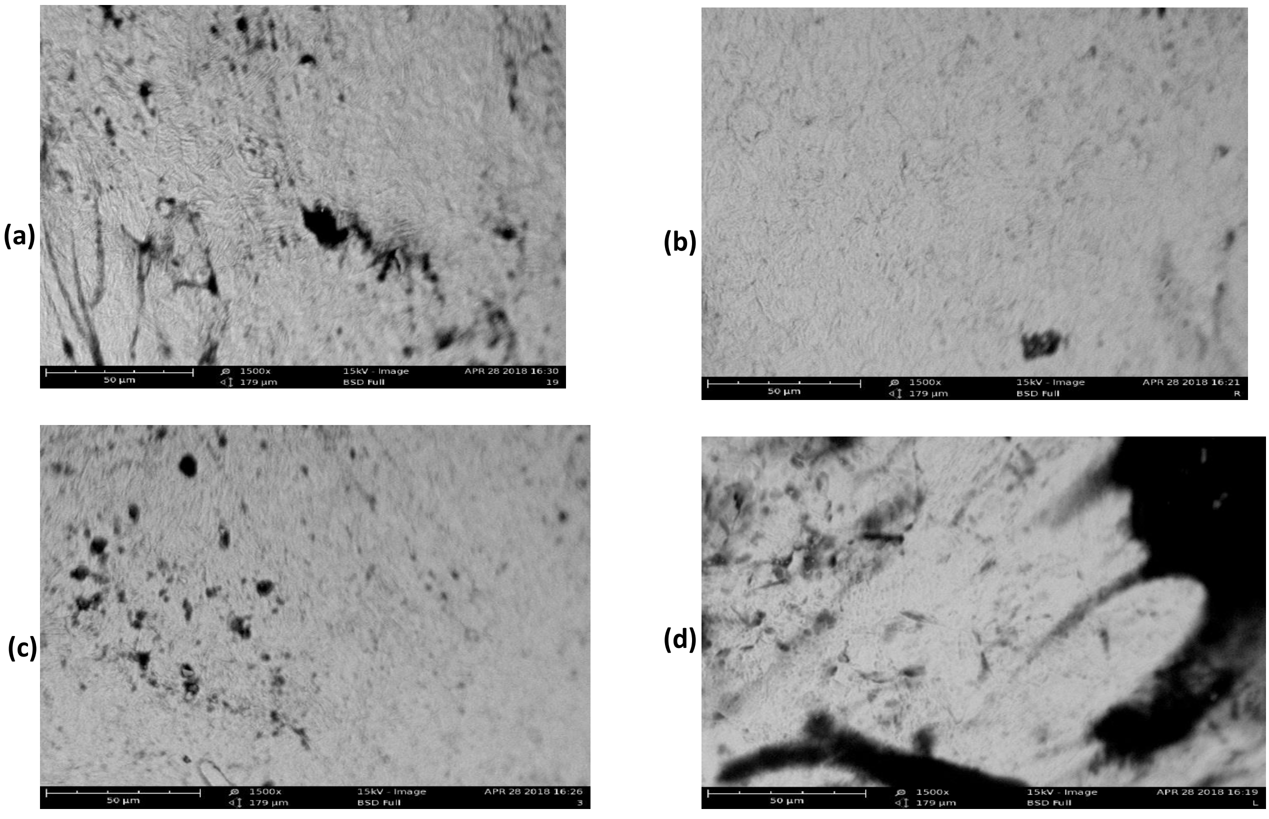

Plate 2: *SEM Image of sample quenched in a) EM, b) ETM, c) TM and d) FM at optimum parameters setting*

The confirmation experiment results show good agreement with the prediction results. Further, maximum hardness of 741 and 715 HVN were obtained by quenching in ETC and ETM respectively in the validation test. Similarly, in the test, optimum hardness of 432 and 414 HVN were recorded for samples quenched in FC and FM while quenching in SAE40 mineral oil yielded steel with peak hardness of 415 HVN.

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