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Heat Treatment Factors Affecting AISI 304 Austenitic Stainless Steels with Wear-resistant in High Temperature and High acid Conditions for using in Agricultural Product Processing

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Abstract

This research was aimed to study factors affecting heat treatment of AISI 304 austenitic stainless steel under high natural acidity and high temperature conditions for using in the agricultural product processing which affect the weight loss after the experiment. This research applied the 2^3 factorial design consisting of 3 independent factors, including temperatures, holding time, and quenching fluids. In each factor, there are two experimental levels: temperature factor levels at 560 °C and 650 °C, the holding time of 20 and 40 minutes and the quenching fluids: cold water and heat-treatment oil. The Pin-on-Disc method was applied to determine the weight loss. The experimental conditions in this study were the temperatures of 70 - 80 °C and natural acidity at pH 3 - 3.5. The study found that all factors statistically significantly affected the weight loss of the stainless steel and the temperature of 650 °C, the holding time of 40 minutes and cooling in water was the most weight loss resistance. Moreover, there was a interaction of the factors, the temperatures and holding time, which affect the weight loss, and they were also significantly statistically. A mathematical model for lost weight estimation from the independent variables was formulated. The results showed that there was a 95% confidence interval, equivalent to ±0.00623 g. of the weight loss value.

Keywords: AISI 304 Stainless Steel, Heat Treatment, Wear Resistant, Factorial Design, Food machinery.

1. INTRODUCTION

Agricultural products are one of important exports of Thailand and an agricultural industry also contributes to economic development in provinces all over the country. Manufacturers add value of agricultural products by using machinery for processing; therefore, the prices of the agricultural goods become higher and their quality is better. Tools and machinery are made of AISI 304 stainless steel for food processing because they do not rust and endure different conditions. In the food industry, vegetables and fruits such as pickled ginger, sour tamarind, passion fruit, and pineapple are involved in food manufacturing and they are high-acid, causing rapid wear and corrosion in the machines. In addition, food processing requires high temperatures which result in rapid wear and tear. He et al. (2018) and Badischa et al. (2009) indicated the high temperatures cause a decrease in wear resistance. When the wear and tear of machinery occurs, food manufacturers have to cease food processing such as printing rollers and sealing packs and this affects food production and wasting both time and money in maintenance. In fact, manufacturing vegetables and fruits such as ginger and tamarinds enable parts of the machine blades have wear and tear. Tukur et al. (2014) concluded that the quenched AISI 304 austenitic stainless steels are sensitized when temperatures are between 470-750°C, resulting in carbide precipitations at grain boundaries and the hardness of the stainless steel increases, especially through air cooling, as shown in the researches of MA et al. (2012) and Khana et al. (2020). The microstructures of the steel were quenched at the temperature of 950 °C after holding time of 15 minutes and there was an increase in quenching cooling rates in air, water and heat-treatment oil. It was found that there was a variation in grain size in the cooling rates. Essoussi et al. (2018) presented a tensile tests were also performed in the present study and it was found that the yield stress of the treated samples tended to decrease. Therefore, the objective of this research was to study suitable factors of wear resistance of AISI 304 austenitic stainless steel under high temperature and high-acid conditions for use in the agricultural product processing. The study also aimed to determine the most appropriate experimental factors of wear resistance and practical applications.

2. MATERIALS AND METHODS

The chemical component of AISI 304 element is 0.080 % C, 18-20 % Cr, 8.0-10.5 % Ni, 2.0 % Mn. The ultimate tensile strength of 505 MPa, yield tensile strength at 215 MPa and elongation at 70 %. Montgomery (2013) suggested that the 2^3 factorial design applied in this study has eight different experimental conditions and testing under each of the conditions is repeated four times (4 replicates) to increase the accuracy of observation values and to reduce experimental errors, as shown in the researches of Boonjubut and Wantang (2014). The order of operations in this experiment with the 2^3 factorial design is random to prevent nuisance variables from confounding with every data value and to enable each value of data independent to each other. The factor levels ranging from the highest to the lowest are shown in Table 1. The selection of the factors and levels which Pre-experimental and literature review. The size of stainless steel sample used 32 pieces in the experiment.

After heat treatment under specified conditions, the specimens will be tested for wear by using a Pin-on-Disc testing machine and immediately measuring the lost weight. Parameters testing and wear evaluation are carried in accordance with the requirements of ASTM International (2014) and ASTM G99-17 (2017). The Pin-on-Disc testing in performing with control factors such as temperatures between 70 - 80 °C, acidity of pickled ginger at pH 3 - 3.5, Petpadap et al. (2018), Kennedy and Lu (2015) and Olofsson et al. (2018) designed a rotating disc speed of 900 rpm., and holding time of 60 minutes and weight of 2,500 g., as shown in Figure 1. The testing machine is used to find the average lost weight under each condition in the 2^3 factorial experiment.

Table 1 Factor levels in the 2^3 factorial design experiments

Factors	Factor Levels		Symbols
	Low (-1)	High (1)	
Solution Temperature (°C)	560	650	A
Holding Time (min)	20	40	B
Quenching fluid	Cold water	Heat-treatment oil	C

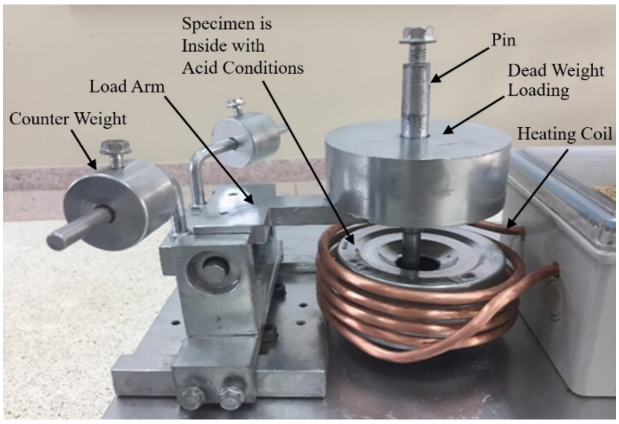


Figure 1 The Pin-on-Disc testing machine

3. RESULTS AND DISCUSSION

3.1 Result

The specimens were immediately measured for lost weight with a 0.01 g. digital scale, after being tested with the Pin-on-Disc. The results obtained from performing the Pin-on-Disc wear test show the mean weight lost during the test under different conditions. In addition, this experiment is run in random order or randomization. The results show the mean lost weight values of each test piece in this experiment using the 2^3 factorial design as shown in Table 2. The heat-treatment oil is low viscosity at 870 kg/m^3 and viscosity 30 mm/s at 40 °C it suitable for quenching of small pieces where drag-out has to be reduced.

Analysis of Variance or ANOVA at a significant level of 5% of hypothesis testing (α = 0.05) is applied in the 2^3 factorial experiment design. The results show that both variables have a linear relationship and the factors significant influence the weight loss as shown in the table.

Table 2 Result summary of the tests

(B) Holding Time (min)	(C) Quenching fluid	(A) Solution Temperature (°C)							
		560	650	560	650	560	650	560	650
20	cold water	0.17 g	0.16 g	0.17 g	0.16 g	0.18 g	0.16 g	0.18 g	0.17 g
	oil	0.18 g	0.17 g	0.18 g	0.16 g	0.18 g	0.17 g	0.19 g	0.17 g
40	cold water	0.17 g	0.14 g	0.17 g	0.14 g	0.17 g	0.15 g	0.16 g	0.16 g
	oil	0.18 g	0.16 g	0.18 g	0.15 g	0.18 g	0.15 g	0.18 g	0.14 g

According to the results of variance analysis, it can be concluded that factors A, B, and C have a significant effect on the weight loss. The P-values are 0.000, 0.000 and 0.004, which are less than a 0.05 significance level or 95% confidence level. The two-factor interaction AB significantly and statistically affects the weight (P-Value < 0.015). The P-Value is greater than 0.05. When considering Table 3, the factors including temperatures, holding time and quenching fluids with P-Value greater than 0.05 have no effect on wear resistance, Jozefa et al. (2020) concluded that the Pareto chart of standardized effects of the factors, the influence of the factors and their interactions can be determined based on this chart, the most significant influence have the factors, temperature A, holding time B, cold water C and interaction between factors of AB as well, as shown in Figure 2. Moreover, considering the coefficient of determination, the R-Square is 84.15% and the adjusted R-Square is 79.53%, which are higher than 75%, indicating that the equation for computing correlation has accuracy and provides a good explanation of wear resistance in this experiment.

Table 3 Analysis of Variance in the 2^3 factorial experiment

Source	DF	Sum of Squares	Mean Squares	F-Value	P-Value
A	1	0.003003	0.003003	82.37	0.000
B	1	0.000903	0.000903	24.77	0.000
C	1	0.000378	0.000378	10.37	0.004
A*B	1	0.000253	0.000253	6.94	0.015
A*C	1	0.000078	0.000078	2.14	0.156
B*C	1	0.000003	0.000003	0.09	0.772
A*B*C	1	0.000028	0.000028	0.77	0.388
Error	24	0.000875	0.000036		
Total	31	0.005522			

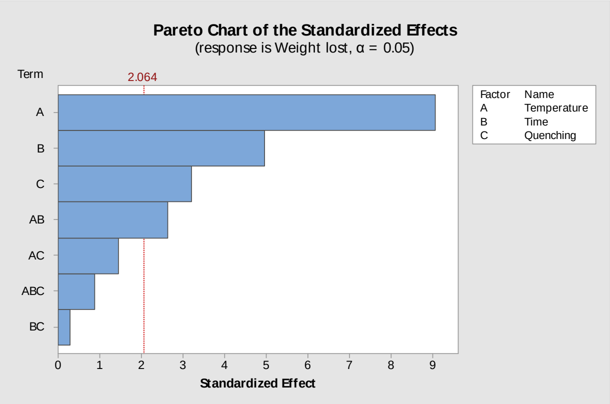


Figure 2 Pareto chart of the standardized effects.

Main Effects Plot in Figure 3 shows the influence of the three main factors. The graph displays between the average lost weight, which also represents wear resistance in different levels of each key factor including temperatures, holding time and quenching fluids. It shows that all factors affect the weight loss. The graph also illustrates that under the conditions of 650 °C temperature, 40 minutes of holding time, and water as a cooling fluid, the weight is lost less than the average lost weight of the specimens.

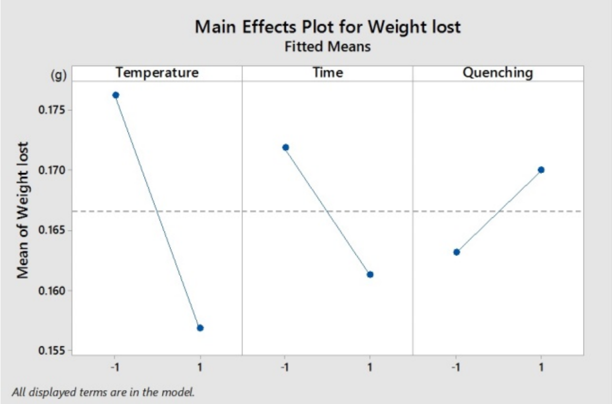


Figure 3 Main effects plot for weight lost

The interaction plot shows the relationship between the average lost weight and factors including temperatures, holding time, and quenching fluids, as shown in Figure 4. The result is that there is linear correlation. According to the plot, there is a linear correlation between the temperatures and holding time which affect the weight loss after heat treatment. In addition, the lost weight after the solution heat treatment is lower at high temperatures and long holding time, compared with higher temperatures and shorter holding time.

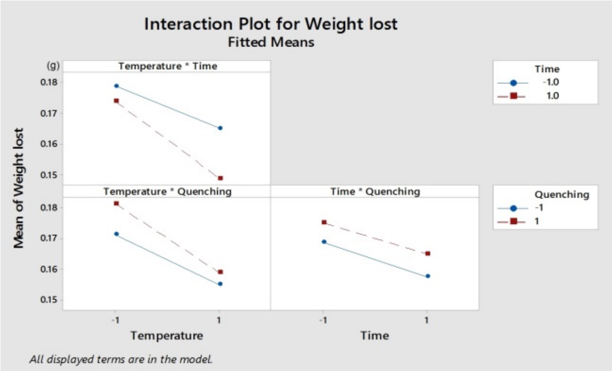


Figure 4 Interaction plot for weight lost

3.2 The Regression Model

In this experiment, a regression equation showing the relationship between the lost weight values and variables in a curve manner by considering only the main factors and cofactors that significantly influence the lost weight values, as follows equation (1).

$$\hat{y} = \hat{\beta}_0 + \hat{\beta}_1 x_1 + \hat{\beta}_2 x_2 + \hat{\beta}_3 x_3 + \hat{\beta}_{12} x_1 x_2 + \hat{\beta}_{13} x_1 x_3 + \hat{\beta}_{23} x_2 x_3 + \hat{\beta}_{123} x_1 x_2 x_3 \quad (1)$$

Where $\hat{y}$ is the fitted values

$\hat{\beta}_0$ is the average weight lost of all data

$\hat{\beta}$ is regression coefficient

x_1 is the temperature (A)

x_2 is the holding time (B)

x_3 is the quenching fluids (C)

From equation (1), a regression equation for estimating the lost weight values of the main factors and cofactors that significantly influence the weight loss and the R^2 is 84.15%, which are high, indicating that the linear regression model provides a good explanation of wear resistance of this experiment, values are as follows:

$$\hat{y} = 0.1993 - 0.000028(A) + 0.00325(B) - 0.0143(C) - 0.000006(AB)$$

3.3 Confidence Intervals

A 95% confidence interval (α = 0.05) is computed from the lost weight values obtained from estimating values in the regression by using the following equation (2).

$$Y \pm t_{\alpha/2,\, N\text{-}a}\sqrt{MSE} \qquad (2)$$

The confidence level at 95% (α = 0.05 and $\alpha/2$ = 0.025) and the Sum of Squares of Error values or MSE are calculated by the division of Sum of Squares of the non-significant factors plus MSE and the degree of freedom. After analyzing the variance of the simulation model, the MSE value is Y±0.00623 g. Therefore, the weight loss values estimated from the model have a confidence interval of 95% or equivalent to ± 0.00623 g. of the estimated values.

3.4 Response optimization

A response optimization plot is a diagram creation of the response optimization for minimum weight loss. The multiple response prediction is temperature factor levels at 650 °C, the holding time of 40 minutes and the cold water quenching, it is the probability of this prediction is about 85% to get the lowest value of 0.1475, as shown in Figure 5. The confidence interval was 0.14127, 0.15373 (95% CI) and prediction interval was 0.13357, 0.16143 (95% PI), when the same experiment was repeated and averaged, the chances of getting the mean response were 95% in this range.

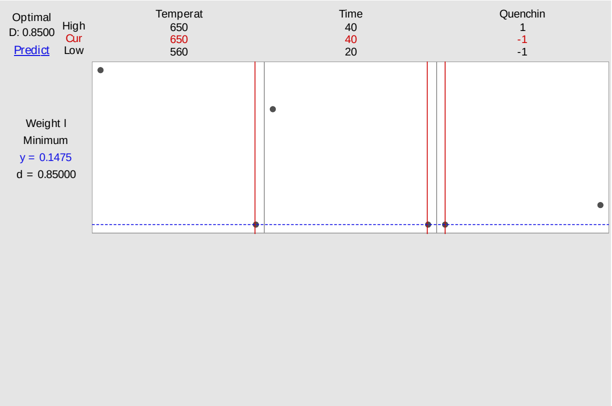


Figure 5 Optimization plot for weight lost

3.5 Contour plot

A contour plot is a diagram creation of the response surface for easy consideration of the relationship between the lost weight value and the level of the two considered factors. The weight loss value is estimated at each level of factors and the diagram shows the trend of lost weight values which are changeable at different levels of factors. In this experiment, there are 3 main factors, but only two factors are correlated to each other. Figure 6 shows the relationship of the weight loss values when the two factors including temperature and holding time change. As shown in the figure, the response surface shows 6 levels of lost weight values.

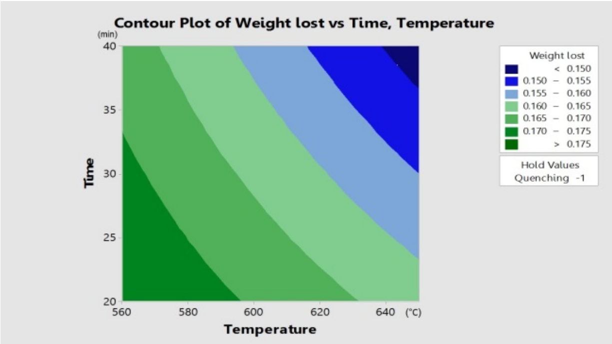


Figure 6 Contour plot of weight lost

3.6 Diagnostic Checking

After the analysis of variance and model is performed, the model adequacy is checked. Moreover, errors or residuals in the experiment are checked whether there is a normal distribution and independence of errors. According to the normal probability plot, as shown in Figure 7(a), it shows a normal distribution of residuals of the results obtained from the experiment conducted with a normal probability method. The data followed the straight line and it is approximated that the remaining values have a normal distribution. Independence of errors and regression mean is checked by examining a scatter diagram of Versus Fits, as shown in Figure 7(b). It is found that there is independence and the remaining expected values are randomly distributed in both positive and negative sides, compared with the lost weight estimates. The histogram plot, as shown in Figure 7(c), illustrates that the distribution of residuals has a bell-shaped curve, indicating a normal distribution of the data. The Versus Order plot in Figure 7(d) is used to check residual variance of the data and examine the relationship between the errors in the experiment. The errors of the data are randomly distributed and their patterns are unclear, indicating that the weight loss values do not depend on the order of trails.

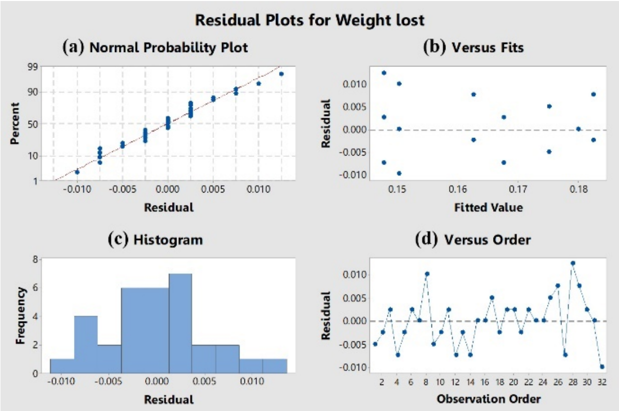
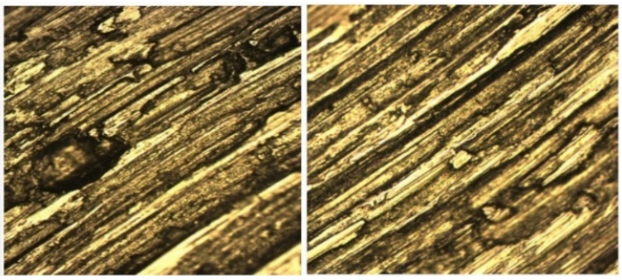


Figure 7 Residual plots for weight lost

3.7 Scratches of the specimens

Each piece of the specimens would be oppressively and then be measured again with a 500x optical microscope for the size of the indentation. After analyzing scratches of the specimens, it was found that the specimens had the highest weight loss at the temperature of 560 °C, with the holding time of 20 minutes, and through heat-treatment oil cooling. In Figure 8(a), the specimens had the low weight loss at the temperature of 650 °C, with the holding time of 40 minutes and through water cooling, as shown in Figure 8(b), consistent with the experimental results.



(a) (b)

Figure 8 Comparison of scratches of test pieces

4. CONCLUSIONS

The results showed that after precipitation hardening, all the main factors in the experiment have a significant effect on weight loss. Almost all the factors, including temperatures, holding time, and quenching fluids significantly influenced the weight loss. In addition, there was also interactions of the two factors and each of them influenced the weight loss significantly. The relationship between the lost weight values and the factors is associated with variations as cofactors. The mathematical model derived from this experimental design was used to estimate the lost weight values with an error not over ±0.00623 g. and with a reliability of 95%. The errors or residuals were found to be normally distributed in accordance with the hypothesis of this experimental design. Moreover, there were random, consistent, and equal distributions at each level of the main factors.

After conducting my research, the results can be summarized as follows.

1. The results indicated that the better factors included the temperature of 650 °C, the holding time of 40 minutes, and cooling in the water. These factors resulted in the lowest weight loss (wear resistance). AISI 304 austenitic stainless steel undergoing the heat treatment process which depends on the appropriate factors can be used to make tools enduring high natural acidity and high temperature conditions.

2. After the physical analysis of the scratches by using an optical microscope, wear conditions were consistent with the experimental results. Different temperatures, holding time, and cooling fluid affected the wear resistance.

3. The result indicated that there was greater wear resistance at high temperature, with long holding time and cooling in water, compared with low temperatures.

The AISI 304 austenitic stainless steel agricultural product processing tools in high natural acidity and high temperature conditions, which causes rapid wear. The optimum factors of heat treatment were able to improve the mechanical properties in the internal structure, increase carbide solubility, increase the durability of stainless steel and reduced weight loss.

Future directions of research will support the processing of agricultural products and food machinery. Because food and agricultural processing machinery are a convenient tool for commercial production, so that the Thai food processing and agricultural sector continues to be recognized as one of the world's leading agricultural suppliers.

5. ACKNOWLEDGMENT

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