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Acidity improvement of refined-bleached used vegetable oils as dielectric liquid using two-level factorial design

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ABSTRACT

Recent studies have shown that modifying the chemical structure of used vegetable oils (UVOs) as an alternative dielectric liquid for oil-immersed transformers has improved these oils' physical, electrical and chemical properties. However, previous researchers have implemented the one-factor-at-a-time method as their experimental design approach. Therefore, they overlooked the possibility that combining mixing process parameters at optimum ratios will yield a better result. Hence, in this study, the two-level (2^k) factorial design is applied to achieve the lowest acidity level of UVOs through chemical refining process namely as refined-bleached used vegetable oils (RBUVOs). The involved process parameters are oil temperature, mixing speed and mixing time. Based on the results of 2^3 factorial design, it is found that oil temperature has the most significant effect on acidity, with a percentage contribution of 35.76%. The result also shows that the best mixing process parameters of RBUVOs were: oil temperature (60 °C), mixing speed (1,000 rpm) and mixing time (30 min). Note that these mixing process parameters produced better RBUVOs with an acidity value of 0.0221 mg KOH/g. A regression model is also developed to predict the acidity of RBUVOs as a function of oil temperature, mixing speed and mixing time.

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1. INTRODUCTION

There are two types of natural ester insulating (NEI) oils, namely, i) virgin vegetable oils (VVOs) and ii) used vegetable oils (UVOs). UVOs have used cooking oils that can be obtained after food is fried in oils. For example, VVOs and UVOs have great potential as transformer insulating oils because they are economical and renewable sources. Other than that, UVOs have shown great potential as fuels, biolubricants, and coolants in oil-immersed power transformers. However, UVOs are not without disadvantages—these oils have low pour points [1], low oxidation stability [2], low impulse breakdown strength [3]–[5], high viscosity [6], higher acidity [7] and its color appearance is indicated poor quality (too dark), which is above its permissible color value (color number) 1.0 [8]. These disadvantages have influenced the decisions made by some energy utilities regarding the use of NEI oil in oil-immersed power transformers. Apart from that,

acidity number is one of the chemical properties of NEI oils that have received much attention from scientists and researchers. According to the IEEE guide for acceptance and maintenance of natural ester fluids in transformers (IEEE C57.147:2008), the maximum permissible limit is 0.06 mg KOH/g [8], [9]. The acidity number is also known as the total acidic content in the transformer insulating oil. In general, the increase in acidity number will accelerating the ageing process of oil and paper [8], [10]. Therefore, various techniques have been used to enhance the properties of NEI oils by the addition of pour point depressants [1], lightning impulse resistant additives [8], [10], antioxidants (oxidation inhibitors) [11], [12], modification of the chemical structure of the oils [6], [13]–[15] as well as by acidity reducers (adsorbents) [16], [17].

Most studies on UVOs are focused on their potential as a biolubricant and biodiesel production [17], [18]. Up to date, there was only one published report that studied the effects of chemical refining (neutralization and bleaching) and treatment (treated with synthetic silicate adsorbent) process) on the UVOs as a potential dielectric fluid for power transformers. Nevertheless, the mixing process parameters (specifically the oil temperature, mixing speed and mixing time) were not optimized specifically for UVOs [8]. Hence, the valuable bioactive compounds might easily degrade during refining due to the high temperature applied [19]. In addition, previous researchers have only implemented the one-factor-at-a-time (OFAT) method as their experimental design approach to determine the maximum level of mixing process parameters that will improve the oil characteristic. Note that the OFAT method requires many tests runs or experiments to estimate the effect that can give better outputs. The large number of test runs required is highly undesirable since it consumes considerable time and cost to execute all experiments [20], [21]. Thus, the mixing process parameters are needed to achieve maximum effect for different types of insulating oils in order to obtain the maximum output of UVOs with high-quality attributes and remove the undesirable component in the crude oil simultaneously.

In this study, the mixing process parameters, such as i) mixing speed used to mix the UVOs with the adsorbent (fuller's earth and synthetic silicate), ii) temperature of the UVOs when they are mixed with the adsorbent (fuller's earth and synthetic silicate), and iii) mixing time when they are mixed with the adsorbent (fuller's earth and synthetic silicate) should be refined using two-level (2^n) factorial design of experiments. This minimizes the total acid number (TAN) of refined-bleached used vegetable oils (RBUVOs). The results obtained from this technique are then verified using analysis of variance (ANOVA). Subsequently, a regression model was developed to predict the acidity number of the RBUVOs as a function of the mixing process parameters (specifically oil temperature, mixing speed and mixing time), and the adequacy of the model is verified using ANOVA.

2. METHOD

2.1. Sample preparation

In this study, UVOs collected from hotels, caterers, and restaurants were chosen for neutralization, bleaching, and treatment. The 1L UVOs were first pre-processed and heated at 110 ℃ for 10 minutes to reduce their water content to approximately 400 ppm. Consequently, heated UVOs were left at room temperature until the current temperature dropped to 60 °C. Next, UVOs were neutralized with sodium hydroxide (NaOH) solution (normality 3.0 N) in a 2L beaker. The mixture was stirred at 500 rpm for 5 min at a temperature of 70 °C using a hot plate magnetic stirrer. The mixed samples were then wrapped with aluminium foil and stored in a dry, cool place for 24 h. After 24 h, the mixed samples were formed into two formed layers known as neutralized UVOs (NUVOs) (top layer) and soap stock (bottom layer). The neutralized oil was washed seven times to remove remnants of NaOH and soap stock completely. Correspondingly, the mixed samples were filtered to remove the soap stock and transferred the NUVOs to another 2L beaker before the bleaching and treatment process. The samples were prepared based on the two-level (2^n) factorial design matrix obtained from the screening process with the addition of Fuller's Earth and synthetic silicate adsorbent. The volume concentrations of both adsorbents were kept fixed at 20 wt% and 15 wt% because this volume ratio was reported to be the optimum volume ratio which significantly improved the acidity number of the UVOs [14]. During the mixing process, new RBUVOs and mixing process parameters was conducted at different setting, as shown in Table 1. After the mixing process, the RBUVOs with mixing process parameters were separated using Whatman filter paper (pore size: 0.2 µm) to remove sludge (i.e., a substance composed of fuller's earth and synthetic silicate adsorbent and polar components present in the RBUVOs). Once the filtration process was complete, the oil sample was poured into a glass bottle and stored at room temperature for 24h prior to the acidity number test.

2.2. Total acid number test

TAN is used to measure the output responses (acidity number) based on all thirteen samples developed by the 2^k factorial design matrix obtained from the screening process. For this work, the acidity

number was measured according to the ASTM D974 standard test method using a compact titrator (model: 848 Titrino Plus, Metrohm AG, and Switzerland). Likewise, the following chemical reagents (potassium hydroxide, isopropyl alcohol, and potassium hydrogen phthalate) were used to determine the TAN of the oil samples. Firstly, 5 g of the oil sample was weighed into a titration vessel, and 20 mL of isopropyl alcohol was added. Next, the solution was titrated, where the concentration of potassium hydroxide in isopropyl alcohol was 0.1 mol/L. The response results were recorded and keyed into Design Expert Software to observe the significant factor and optimum condition for each output response.

2.3. Design of experiments

The 2^k factorial design was used to determine the significance of each mixing process parameter (i.e., oil temperature, mixing speed and mixing time) on the NUVOs. Note that the design of experiments was carried out using Design Expert Software version 10.0.8.0 (Stat-Ease, Inc., Minneapolis, USA). Table 1 shows the level of oil temperature, mixing speed and mixing time variables, whereby low, medium and high are indicated by -1, 0 and +1, respectively. The 2^3 factorial design matrix and five center point used to screen the factors is shown in Table 1, with thirteen test runs. The acidity number tests were carried out using the 2^3 factorial design matrix. The effects of the oil temperature, mixing speed and mixing time (factor 1, factor 2 and factor 3, respectively) on the acidity number of RBUVOs were examined using ANOVA. Following this, the response surface plot was applied to screen the mixing process parameters (i.e., oil temperature, mixing speed and mixing time), which will improve the acidity number of the RBUVOs.

Table 1. Two-level factorial design matrix for three independent variables obtained from Design Expert Software

Experimental trial	Variable code		
no.	A: Oil temperature (°C)	B: Mixing speed (rpm)	C: Mixing time (min)
1	80 (+1)	1,000 (+1)	30 (−1)
2	70 (0)	750 (0)	45 (0)
3	70 (0)	750 (0)	45 (0)
4	70 (0)	750 (0)	45 (0)
5	60 (−1)	500 (−1)	30 (−1)
6	60 (−1)	500 (−1)	60 (+1)
7	60 (−1)	1000 (+1)	60 (+1)
8	70 (0)	750 (0)	45 (0)
9	80 (+)	500 (−1)	60 (+1)
10	60 (0)	1000 (+)	30 (-1)
11	70 (0)	750 (0)	45 (0)
12	80 (+1)	500 (−1)	30 (−1)
13	80 (+1)	1000 (+1)	60 (+1)

2.4. Screening process

This phase involves analyzing all the data from each test run and estimating the optimum points. Based on the results obtained from the screening process, a regression model was developed to predict the acidity number as a function of the oil temperature, mixing speed and mixing time. Here, the acidity number is the response variable, whereas the oil temperature, mixing speed and mixing time are the independent variables. ANOVA was used to determine the statistical significance of the regression model. The regression equation is given by (1):

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2, \quad (1)$$

where y represents the response variable (predicted variable), x_1 and x_2 represent the independent variables (factors), $x_1 x_2$ represent the interaction between factors x_1, x_2, β_1 and β_2 represent the coefficients associated with factors x_1 and x_2, β_{12} represents the coefficient associated with interactions $x_1 x_2$, $\beta_1 x_1$ and $\beta_2 x_2$ represent the effects of factors x_1 and x_2, while β_0 represents the intercept of the regression model. Other than that, regression analysis was carried out based on (1). The sum of squares (SS), mean squares (MS), F-value, p-value, coefficient of determination (R^2), and correlation coefficient (|R|) were determined using ANOVA. Response surface plot was used to determine the best combination level of mixing process parameters of oil temperature, mixing speed and mixing time, maximizing the color reduction (%) of the RBUVOs.

3. RESULTS AND DISCUSSION

3.1. TAN test results

In this study, three factorials namely oil temperature, mixing speed and mixing time were analyzed using two-level factorial design. Single response recorded was a total acid number (TAN) of RBUVOs. The results of the TAN for all thirteen test runs are presented in Table 2.

Table 2. Mean TAN for each oil sample

Experimental trial no.	Variable code A: Oil temperature (°C)	B: Mixing speed (rpm)	C: Mixing time (min)	Mean TAN (mg KOH/g)
1	80 (+1)	1000 (+1)	30 (−1)	0.0458
2	70 (0)	750 (0)	45 (0)	0.0251
3	70 (0)	750 (0)	45 (0)	0.0256
4	70 (0)	750 (0)	45 (0)	0.0258
5	60 (−1)	500 (−1)	30 (−1)	0.0351
6	60 (−1)	500 (−1)	60 (+1)	0.0288
7	60 (−1)	1000 (+1)	60 (+1)	0.0297
8	70 (0)	750 (0)	45 (0)	0.0260
9	80 (+)	500 (−1)	60 (+1)	0.0438
10	60 (0)	1000 (+)	30 (−1)	0.0221
11	70 (0)	750 (0)	45 (0)	0.0280
12	80 (+1)	500 (−1)	30 (−1)	0.0287
13	80 (+1)	1000 (+1)	60 (+1)	0.0516

3.2. Result of the half-normal plot

Figure 1 and Table 3 show the half-normal plot and effect list, respectively, which are obtained from the 2^3 factorial designs of experiments. It can be observed from Figure 1 that factor A (oil temperature), factor B (mixing speed) and factor C (mixing time) are positioned at a distance far away from the straight line. Likewise, interaction AB, interaction ABC, factor C, interaction AC, and factor B are also positioned at a distance far away from the straight line–though the distance is not as marked as that for factor A. This indicates that factors A, B, C, interactions AB, ABC, and AC, are significant model terms. The effect list, which shows the sum of squares (SS) and percentage contribution for all model terms, is shown in Table 3. The results indicate that factor A is the most significant factor, with a percentage contribution of 35.76%, while the SS for this factor is 0.000367205. In contrast, factor B only has a contribution of 1.99%, which clearly shows that this factor has the lowest contribution among all factors. Other than that, the SS for factor B is 2.048×10^{-5}. Finally, factor C, interactions AB, AC and ABC have a contribution of 5.99%, 16.66%, 4.68% and 6.55%, while its SS are 6.1605×10^{-5}, 0.000171125, 4.802×10^{-5}, and 6.728×10^{-5}, respectively. Based on the results, it is evident that factor A (oil temperature) has a significantly higher contribution to the acidity number compared to factors B and C, interactions of AB, AC and ABC. Nonetheless, the contribution of other factors is still considered important.

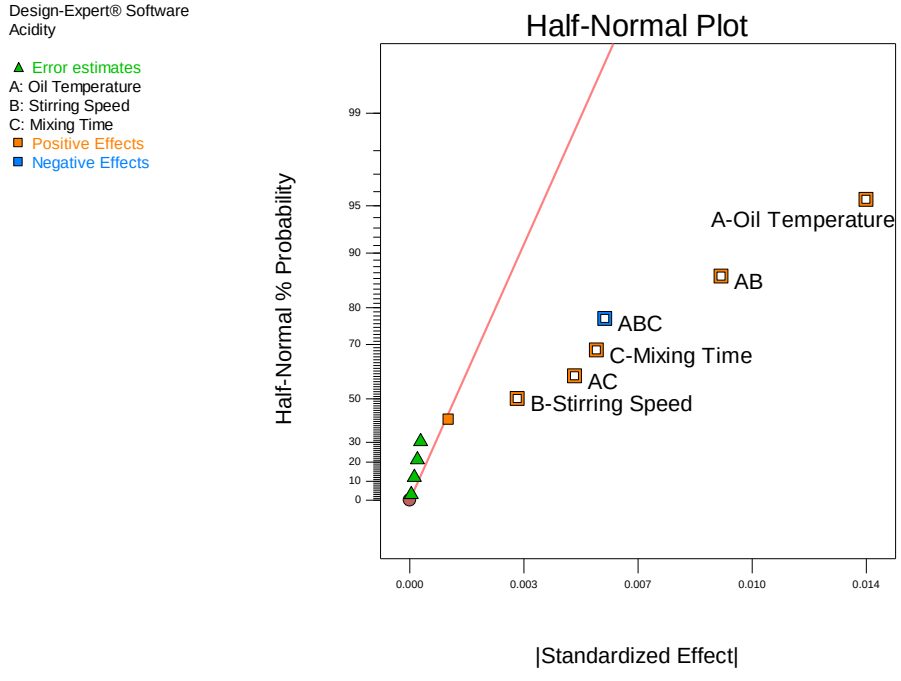


Figure 1. Half-normal plot generated from the 2^3 factorial design

Table 3. Effect list obtained from the 2^3 factorial designs of experiment

Model term	Standardized effects	Sum of squares (SS)	Percentage contribution (%)
A	0.01355	0.000367205	35.79
B	0.0032	2.048×10^{-5}	1.99437
C	0.00555	6.1605×10^{-5}	5.99919
AB	0.00925	0.000171125	16.6644
AC	0.0049	4.802×10^{-5}	4.67626
ABC	-0.0058	6.728×10^{-5}	6.55183

3.3. Results of the ANOVA analysis

The ANOVA analysis is carried out as stated in Table 4. A regression model is developed according to (1), which is shown in (2), indicating the acidity number of RBUVOs as a function of the oil temperature (variable: x_1; unit: ºC), mixing speed (variable: x_2; unit: rpm) and mixing time (variable: x_3; unit: min),

$$y = 0.0357 + 0.0068x_1 + 0.0016x_2 + 0.0028x_3 + 0.0046x_1x_2 + 0.0025x_1x_3 - 0.0029x_1x_2x_3 \quad (2)$$

The mean and standard deviation of the acidity number obtained from the regression equation is found to be 0.0357 and 0.0012 mg KOH/g, respectively. Table 4 shows the SS, degrees of freedom (df), mean squares (MS), F-value, p-value, coefficient of determination (R^2) and correlation coefficient (|R|) for the regression model terms determined using ANOVA. According to Yang *et al.* [22] and Aman *et al.* [23], a p-value less than or equal to 0.05 indicates that the model (or model term) is statistically significant. Apart from that, the results show that the overall regression model is significant since the p-value is 0.0003. Factor A (oil temperature), factor B (mixing speed), factor C (mixing time), interactions AB, AC and ABC, are significant model terms since the p-value is less than 0.05 (<0.0001,0.0153,0.0021,0.0003,0.0034 and 0.0018 respectively). In contrast, the p-value for interaction BC (mixing speed and mixing time) is 0.2179 (which is more than 0.05); therefore, this model term is insignificant.

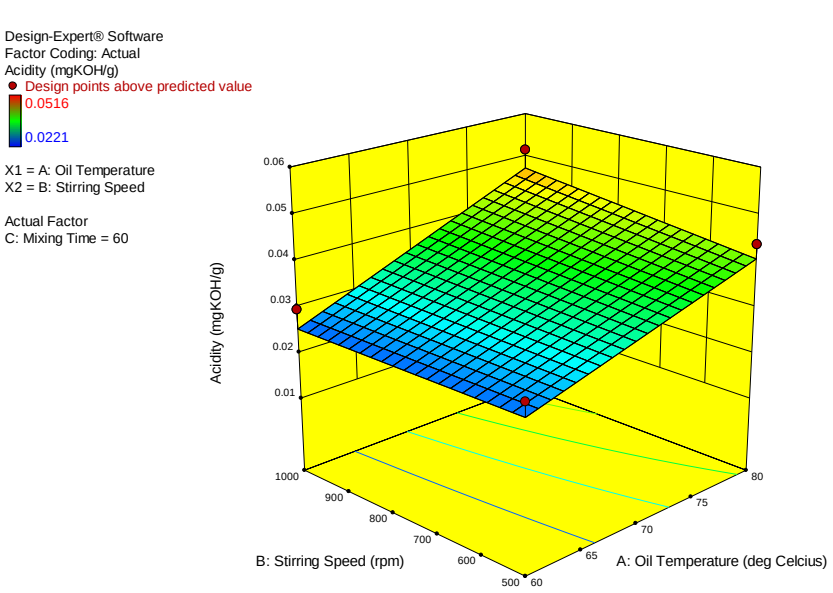
Table 4. ANOVA results for the regression model with factorial response surface fitting

Source	SS	df	MS	F-value	p-value	R^2
Overall model	0.0007	7	0.0001	85.06	0.0003	0.9933
A-Oil Temperature	0.0004	1	0.0004	296.13	<0.0001	
B-Mixing speed	0.0000	1	0.0000	16.52	0.0153	
C-Mixing Time	0.0001	1	0.0001	49.68	0.0021	
AB	0.0002	1	0.0002	138.00	0.0003	
AC	0.0000	1	0.0000	38.73	0.0034	
BC	2.645×10^{-6}	1	$2.645\text{x }10^{-6}$	2.13	0.2179	
ABC	0.0001	1	0.0001		0.0018	
Residual	7.605×10^{-6}	5	1.521×10^{-6}			
Lack of fit	2.645×10^{-6}	1	2.645×10^{-6}	2.133	0.2179	
Pure error	4.96×10^{-6}	4	1.24×10^{-6}			
Total correlation	0.0010	12				

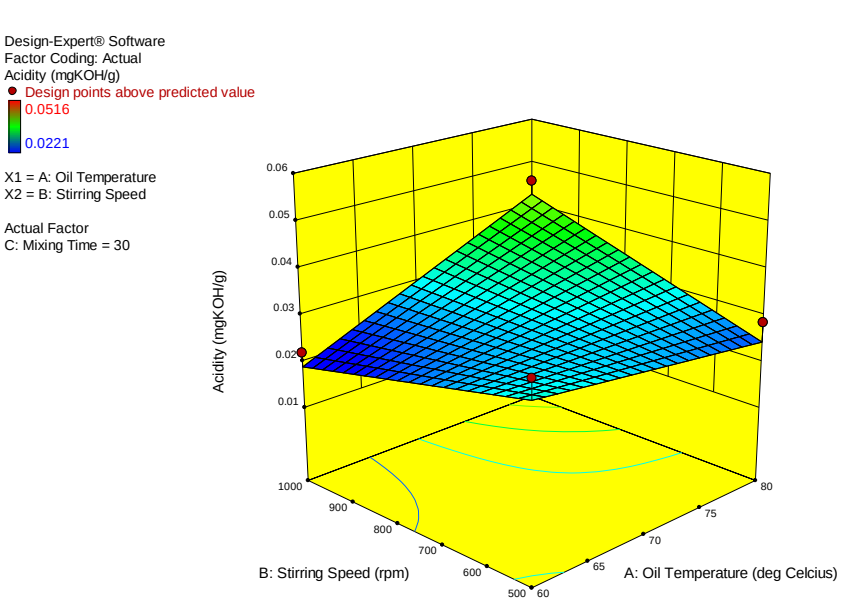
Based on the ANOVA results, it can be inferred that interaction BC (mixing speed and mixing time) will not have a pronounced effect on the acidity number if it is used alone as a mixing process parameter in the RBUVOs. In other words, interaction BC (mixing speed and mixing time) needs to be combined with factor A (oil temperature) to enhance the acidity number of the RBUVOs by a significant margin. It can also be deduced that the regression model developed in this study is adequate since the coefficient of determination (R^2) value is 0.9933, which indicates the model explains 99.33% of the total variation of the acidity number due to the variation of the independent variables (i.e., oil temperature, mixing speed and mixing time). In addition, the p-value of the overall regression model is 0.0003 (less than 0.05), indicating that the model is significant. On the other hand, the correlation coefficient (|R|) shows a correlation between the observed and predicted acidity number. In general, if |R| is closer to 1, this indicates a strong correlation between the observed and predicted values. Therefore, the |R| value of the regression model is found to be 0.9933, showing a strong correlation between the predicted acidity number and those obtained from experiments.

Figure 2 is the response surface plot which shows the variation of the acidity number of the RBUVOs when the mixing process parameters (i.e., oil temperature, mixing speed and mixing time) are varied. The beauty of this plot is that the interaction between the factors influencing the acidity number of the RBUVOs can be visualized and interpreted easily. It can be seen from Figure 2 show that the best combination of mixing process parameters to achieve the lowest acidity number (0.0221 mg KOH/g) of RBUVOs are oil temperature of 60 ℃, mixing speed of 1,000 rpm, and a mixing time of 30 min.

In general, the interaction between oil temperature and mixing speed (interaction AB) had the greatest effect on acidity number of RBUVOs. Based on the response surface plot shown in Figure 2(a), at the highest level of mixing time (60 min), it was observed that the acidity number reached highest level (0.0516 mg KOH/g) with oil temperature (80 ℃) and mixing speed (1000 rpm). However, the acidity number reached lowest level (0.0221 mg KOH/g) with mixing time (30 min), oil temperature (60 ℃), and mixing speed (1000 rpm), as shown in Figure 2(b). This is because higher oil temperature resulted in more reaction between chemical material and water to form more substance (hydrolysis) in RBUVOs, increasing acidity. According to [24], [25], the higher oil temperature has a more pronounced effect on natural ester hydrolysis that will increase acidity.



(a)



(b)

Figure 2. 3D response surface plot; to identify (a) highest and (b) lowest acidity values from mixing process parameters

4. CONCLUSION

In this study, it is proven that the two-level (2^n) factorial design of experiments is a useful technique to determine the best combination level of mixing process parameters of oil temperature, mixing speed and mixing time which will enhance the acidity number of RBUVOs. The main advantage of the 2^n factorial design of experiments approach is that the factors which will have a significant effect on the acidity number of the NUVOs can be determined from fewer test runs, as indicated by the percentage contribution of each factor. This considerably reduces time and cost, which is typically an issue with conventional experimental techniques. Apart from that, the results obtained from the 2^3 factorial designs reveal that oil temperature has the most pronounced effect on the acidity number of RBUVOs, with a percentage contribution of 35.79%. Furthermore, the response surface plot generated reveals that the best combination level of mixing process parameters of oil temperature, mixing speed and mixing speed, which will yield the lowest acidity number, are 60 ℃, 1,000 rpm, and 30 min, respectively. A regression model is also developed in this study, and it is found that the model is adequate to predict the lowest acidity number of the RBUVOs as a function of oil temperature, mixing speed and mixing time setting, whereby the coefficient of determination (R^2) and p-value of the model are 0.9933 and 0.0003, respectively.

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