Optimization of Progressive Freezing for Residual Oil Recovery from a Palm Oil–Water Mixture (POME Model)

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ABSTRACT: Oil and grease remain the dominant contaminants in the palm oil mill effluent (POME) despite the conventional treatment of POME. The removal of residual oil from palm oil–water mixture (POME model) using the progressive freezing process was investigated. An optimization technique called response surface methodology (RSM) with the design of rotatable central composite design was applied to figure out the optimum experimental variables generated by Design–Expert software (version 6.0.4. Stat-Ease, trial version). Besides, RSM also helps to investigate the interactive effects among the independent variables compared to one factor at a time. The variables involved are coolant temperature, $X_A$ (4–12 °C), freezing time, $X_B$ (20–60 min), and circulation flow, $X_C$ (200–600 rpm). The statistical analysis showed that a two-factor interaction model was developed using the obtained experimental data with a coefficient of determination ($R^2$) value of 0.9582. From the RSM-generated model, the optimum conditions for extraction of oil from the POME model were a coolant temperature of 6 °C in 50 min freezing time with a circulation flowrate of 500 rpm. The validation of the model showed that the predicted oil yield and experimental oil yield were 92.56 and 93.20%, respectively.

1. INTRODUCTION

Palm oil has gradually developed as an essential in the agriculture sector, specifically for Malaysia and Indonesia. As illustrated in Figure 1, both countries had concurred 32 and 54% of world palm oil production in 2019.

Figure 1. World palm oil production in 2019.

54% of palm oil production, respectively. Palm kernel oil and crude palm oil (CPO) are the primary outputs of the palm oil mill procedure. Besides, palm oil contributes 42 million tons to the global oil and grease production annually. Despite that, the rising global need for palm oil is projected to intensify the production of palm oil mill effluent (POME), a byproduct of the palm oil sector. POME refers to an extremely contaminating waste manufactured from the fresh fruit bunch (FFB) process in the palm oil mill that is inevitable. POME is a massive amount of liquid waste, which possesses an offensive odor, is organic in the environment, and is nontoxic. Previous studies reported that approximately 30% of water consumption during the palm oil extraction process is converted into steam, while the remaining 50% is translated into POME. The high value of degradable organic matter inside raw POME might be because of the presence of unrecovered palm oil inside it.

In another perspective, high volume of residual CPO streamed into the POME pond will lower the oil extraction rate (OER) of the palm mill because of the inadequacy of the machines in treating FFB. In every ton of POME manufactured by the FFB processing system, 0.7% by weight of residual oil was identified, or specifically, 1 ton of FFB produced 0.2 ton of CPO (main product) production and 0.75 ton of POME (byproduct). To our best knowledge, the remaining 0.05 ton could include the other main product (palm kernel) and byproducts (empty fruit bunch, palm kernel shell, and shredded fiber). As the demand for palm oil has globally increased from year to year, expanding the OER is now a vital need. In parallel, the palm oil business is also aspiring for POME pollution decline to cultivate a greener appearance of
the organization and to establish sustainability. The phenomenon is contributed by many palm oil mills, particularly in Malaysia, who have permitted the ponding process for effluent treatment. Nonetheless, it is a challenge for POME treatment in Malaysia to discover a different technology to be used for the treatment. The available treatments such as evaporation, adsorption, and the reverse osmosis method were found to have less efficiency because they are not profitable, and the process involved is complex.

In the surge to look for appropriate POME treatment strategies, recovering the residual oil from POME could offer a good alternative. For instances, POME obtained from the oil palm crushing mills in Thailand consists of elevated chemical oxygen demand (COD) and oil in the range of 45,000 and 6000 mg/L, respectively. After the oil recovery process, more than 70% of COD was significantly decreased, and 78% of the oil was efficiently recovered from the POME. Therefore, the recovery of oil from POME has shown a positive or huge reduction in the COD value of the POME and intensifies the rate of residual oil recovery.

Presently, numerous separation techniques can be employed to extract oil from POME, which encompass evaporation, coagulation, adsorption method, and reverse osmosis. These methods were applied to mitigate the environmental loading from oily waste and to reduce the processing outlay. However, several of the traditional oil recovery methods possess disadvantages, which include demanding a large extent of energy and complex process. For instance, the evaporation process would require at least one kg of steam to disperse one kg of water content. On the other hand, the physicochemical treatments such as coagulation and adsorption require a high amount of chemicals, which later required a proper disposal method, or else, it will again pollute the environment. Therefore, further investigation of innovative technology is vital for recovering the residual oil from POME to achieve the demand for green palm oil production.

An innovative oil recovery process is introduced in this study, namely, progressive freezing (PF), which has never been explored, mainly in Malaysia. The initiative is vital to discover a new option that can substitute the present techniques by designing a more appropriate and competent method. PF is a technique used to concentrate a solution by freezing or solidify one liquid component into pure solid and successively dividing the part of the frozen solid from the concentrated liquid. The ultimate advantage of PF is that it can maintain the thermally sensitive materials in the concentrate because of the involvement of low process temperatures and low-energy conditions compared to evaporation. Therefore, PF had been widely applied for various industrial applications, for instance, application of PF in concentrating model liquid food,17 widely applied for various industrial applications, for instance, osmosis method were found to have less efficiency because they are not profitable, and the process involved is complex.

In this study, a novel application of the PF method has been introduced for residual oil recovery from POME. The process is believed to extract a high quantity of oil from the effluent because of the difference in freezing or melting points of oil and water. In this research, the performance of the PF process signified by the percentage of oil recovery at different operating conditions such as coolant temperature, freezing time, and circulation flowrate was evaluated.

As observed in a previous study, response surface methodology (RSM) through central composite design (CCD) was found to provide the most effective experimental design and optimization as well as evaluate the best effects of process variables involved in the palm oil recovery process. RSM uses a regression model to study the significance of the research through the obtained experimental data. One factor at a time (OFAT) is the most popular conventional design of experiment (DOE) used in optimizing the multivariate system. However, the conventional approach does not include the study of combined effect of dependent variables and it requires more experimental runs to obtain the optimum conditions, which are considered unreliable as compared to CCD. Based on the past research as well, optimization of PF variables for oil recovery from POME was found to be minimal; thus, this research focuses on the investigation of the combined effects of the coolant temperature, freezing time, and circulation flowrate toward the percentage of oil recovery as the response variable. The process variables were optimized using rotatable central composite design (RCCD) along with the RSM method. A model was developed to determine the optimum PF conditions, where the maximum percentage of oil recovery was achieved from the model palm oil–water mixture (POME model).

2. CONCLUSIONS

Palm oil–water mixture (POME model) residual oil removal by PF was investigated using RSM based on a RCCD. A 2FI model was proposed to correlate the batch experiment variables for oil recovery from POME. It was found that the model was able to predict the experimental data to high accuracy with $R^2$ of 0.982. The interactions among the coolant temperature, $X_A$, and freezing time, $X_B$, was found to have the most significant effect on oil recovery from POME. PF process optimization was studied, and the computed values by the models were found closer to the experimental values. Optimum conditions to maximize the residual oil recovery were obtained at a coolant temperature of 6 ºC, freezing time of 50 min, and circulation flowrate at 500 rpm with an absolute error of 0.69%. The validation of the model showed predicted oil yield, and experimental oil yield was 92.56 and 93.20%, respectively.

3. MATERIALS AND METHODS

3.1. Materials. The sample used for this experiment was the palm oil–water mixture model, which consists of a mixture of 6000 mg of CPO per liter of water. This value was chosen based on literature and equipment limitation. Palm oil was gathered from the bulk storage tank (BST) of FELCRA Nasaruddin Belia Berhad, Bota Kanan, Perak. The sample was then stored at low temperature to avoid any decomposition, oxidation, and changes to the free fatty acid content. Because the POME model was made, as shown in Figure 2, no pretreatment was needed. Few equipment or substances were used in this experiment in order to ensure the success of this study. The primary solution to conduct the experiment was ethylene glycol because it has a wide range of low temperature values, which is generally applied in the process of heat transfer. Instead, a 50% volume ratio (v/v) of ethylene glycol and water were mixed and utilized as the PF coolant. The composition of the coolant was employed to facilitate the supercooling of the solution and to avoid slushy form (viscous) of the ethylene glycol–water solution.

3.2. Experimental Setup. The choices of equipment were highly crucial in extracting the residual oil from the POME
model. This was to ensure the achievement and to produce the best output from the experiment. Thus, the arrangement of the apparatus was developed from the freeze-crystallization basic idea, which contains a peristaltic pump, refrigerant system, circulated freeze-crystallizer, and oil and grease analyzer, as well as pH meter. Circulated freeze-crystallizer is the primary equipment of this system, which functions to allow solidification to take place, while the cooling jacket provides a place for the circulating coolant around the crystallizer body. At this time, heat was transferred from the solution inside the crystallizer body to the coolant inside the cooling jacket. Thus, the temperature could be controlled at the desired reading with the help of the coolant. The body of the crystallizer is generally built in a cylindrical shape. This is because the cylindrical shape as shown in Figure 3 provides a smooth surface, large surface area, and can minimize the friction between the samples. In order to ensure a good mixture of the solution produced, a peristaltic pump was used to circulate the sample taken. Last but not the least, an oil and grease analyzer and pH meter were used at the end of the experiment to measure the value before and after purification. Figure 3 shows the experimental setup for PF by using a circulating freeze-crystallizer (CFC).

3.3. Experimental Procedure. The experimental procedure began with the preparation of the palm oil–water sample, which had been stored beforehand. The coolant mixture was prepared at 50% ethylene glycol and 50% distilled water, which was then manipulated at the desired value for coolant temperature. The CFC was connected to the refrigerant system, peristaltic pump, and storage tank. As the desired coolant temperature was reached, 2.5 L of the sample was poured into the storage tank and the pump flowed the sample into the cylindrical freeze-crystallizer to let the solidification process happen. The coolant temperature from the refrigerant system was then manipulated at various speeds.

Next, the process was stopped, the crystallizer was taken out from the system, and the liquid mixture was transferred into a sample bottle for characterization purposes. The sample was then measured using a TOG analyzer. This was to study the performance of PF in recovering residues in POME. For accurate results, the experiment was repeated thrice. Finally, the experiment was repeated with different parameters, which were coolant temperature, circulation flowrate and freezing time. The oil percentage removal was calculated, as shown in eq 1.

\[
Y(\%) = \frac{Y_0 - Y_1}{Y_0} \times 100
\]

where \(Y\) is oil recovery (\%), \(Y_0\) is the initial oil and grease value (mg L\(^{-1}\)), and \(Y_1\) is the final oil and grease value (mg L\(^{-1}\)).

3.4. Design of Experiment. In this study, the optimization process for progressive freeze concentration of POME solution to recover the residual oil was conducted by using RCCD, which is the best DOE that applies RSM. CCD is the experimental design which had been introduced by Box and Wilson in 1951. CCD is one of the best designs applied in RSM. However, this design involves selection of the right type of CCD. The type of CCD includes spherical (SCCD), rotatable (RCCD), orthogonal (OCCD), and face-centered (FCCD) central composite design. In this study, the RCCD approach has been employed to determine the interaction between the process variables and the process response. According to the past research, the common type of CCD used is either face-centered or rotatable. One of the reasons of choosing rotatable CCD is because of the ability to execute extreme analysis in DOE compared to face-centered CCD. Hence, the studied range would be much wider and reliable to observe the trends. The regression and graphical analysis of the data obtained for the optimization process were carried out through Design–Expert 6.0.4 software (Stat-Ease Inc., Minneapolis, USA). There are four major steps involved in RSM as an optimization method.

First, the independent variables of major effect on the system and the dependent variables were selected through screening studies followed by the restriction of the experimental region. The levels for each parameter were also defined in this step. Second, the type of experimental design was chosen to carry out the experimental run according to the designated value of the set of factors or variables. Next, the obtained experimental data were tabulated and analyzed statistically through second-order polynomial model fitting. The model’s fitness was evaluated before the optimum values for each studied variable were obtained. Figure 4 shows the process block diagram of conducting RSM.
The coolant temperature \((X_A, °C)\), freezing time \((X_B, \text{min})\), and circulation flow \((X_C, \text{rpm})\) were chosen as the independent variables. The design used \(2^n\) factorial runs with \(2n\) axial runs and replications of center points \((n_c)\), (Myers, 1971). Therefore, eight factorial points, six axial points, and three replicates at the central points were used for three variables in the experiments. Thus, the total number of runs \((N)\) required for the three independent variables is 17, as shown in eq 2.

\[
N = 2^n + 2n + n_c = 2^3 + (2 \times 3) + 3 = 17
\]

The next step in optimization through RSM is to conduct the data analysis and model suitability check in representing the real relationship by using analysis of variance (ANOVA). ANOVA has been used to determine the interaction between the independent variables and the response variables. For instance, it has been used to determine the quality of the fitted polynomial model through the coefficient of determination \((R^2)\), and the statistical significance was validated by conducting an F-test.

### 4. RESULTS AND DISCUSSION

#### 4.1. Model Fitting

In this study, three independent variables were investigated, and their levels are shown in Table 1. The designed experiments were conducted according to the randomized scheme, and the result is tabulated in Table 2. As mentioned before, Design–Expert software was used as a tool to study the regression analysis of experimental data and interpret them by using ANOVA analysis. The percentage of oil recovery \((Y)\) was taken as the response variable of the designed experiments.

#### 4.2. Regression Model Equation and ANOVA

In determining the best model to be used for the response, the proposed model should be considered a favorable beginning for the model fitting. The proposed model by the software is the 2FI model, which was not aliased and adequately significant to represent the oil percentage recovery. The selection can be proved through the relevant summary reports shown in Tables 3 and 4, in which F-test and R² values were observed.

To fit a good model, a test for significance of the regression model and individual model coefficients with lack of fit test was performed. The sequential models involved are mean, linear, two-factor interactions (2FI), quadratic, and cubic models. Usually, the significant factors were ranked based on the F-value and p-value with a 95% confidence level. According to the fitted model data shown in Table 3, it is clearly shown that the 2FI model is significant because it fits the conditions where the p-values are less than 0.05. Meanwhile, the lack of fit test is considered good if the model is not significant (p-value > 0.1). Based on Table 4, the only computed p-value that exceeds 0.1 is the model 2FI with 0.1041; thus, it is considered insignificant. Therefore, the 2FI model has been chosen as the best model to be used for model fitting. The final empirical model in terms of the coded factor for residual oil recovery \((Y, \%)\) is shown in eq 3

\[
Y, \% = 87.03 + 0.59X_A + 0.59X_B + 0.96X_C - 3.5X_AX_B - 2.4X_AX_C - 1.35X_BX_C
\]
Table 3. Sequential Model Sum of Squares

| source    | sum of squares | DF  | mean square | F value | prob > F | remarks |
|-----------|----------------|-----|-------------|---------|----------|---------|
| mean      | $1.128 \times 10^5$ | 1   | $1.128 \times 10^5$ | 1.01    | 0.4239   |         |
| linear    | 37.40          | 3   | 12.47       | <0.0001 | significant |
| 2FI       | 128.20         | 3   | 42.73       | 47.29   | <0.0001 | significant |
| quadratic | 0.94           | 3   | 0.31        | 0.25    | 0.8585  |         |
| cubic     | 6.03           | 3   | 2.01        | 15.45   | 0.0614  |         |
| residual  | 0.26           | 2   | 0.13        |         |         |         |
| total     | $1.13 \times 10^5$ | 15  | 7533.04     |         |         |         |

Table 4. Lack of Fit Test

| source    | sum of squares | DF  | mean square | F value | p-value | remarks |
|-----------|----------------|-----|-------------|---------|---------|---------|
| linear    | 135.17         | 9   | 15.02       | 115.53  | 0.0086  | insignificant |
| 2FI       | 6.97           | 6   | 1.16        | 8.93    | 0.1041  | insignificant |
| quadratic | 6.03           | 3   | 2.01        | 15.45   | 0.0614  |         |
| cubic     | 0.000          | 0   | 0           |         |         |         |
| pure error| 0.26           | 2   | 0.13        |         |         |         |

Table 5. ANOVA for the Response Surface 2FI Model for Oil Recovery from the POME Model

| source    | sum of squares | DF  | mean square | F value | prob > F | remarks |
|-----------|----------------|-----|-------------|---------|----------|---------|
| model     | 165.60         | 6   | 27.60       | 30.55   | <0.0001  | significant |
| $X_A$     | 2.66           | 1   | 2.66        | 2.95    | 0.1244  |         |
| $X_B$     | 3.83           | 1   | 3.83        | 4.24    | 0.0735  |         |
| $X_C$     | 10.06          | 1   | 10.06       | 11.13   | 0.0103  | significant |
| $X_A X_B$ | 69.45          | 1   | 69.45       | 76.86   | <0.0001 | significant |
| $X_A X_C$ | 32.70          | 1   | 32.70       | 36.18   | 0.0003  | significant |
| $X_B X_C$ | 10.24          | 1   | 10.24       | 11.33   | 0.0098  | significant |
| residual  | 7.23           | 8   | 0.90        |         |         |         |
| lack of fit | 6.97         | 6   | 1.16        | 8.93    | 0.1041  | insignificant |
| pure error | 0.26          | 2   | 0.13        |         |         |         |
| cor total | 172.83         | 14  |             |         |         |         |
| std. dev  | 0.95           | 3   | 0.95        | 22.966  |         |         |
| mean      | 86.73          | 3   | 27.11       |         |         |         |
| R-squared | 0.9582         | 20  | 0.9582      |         |         |         |

10.41% chance that a “lack of fit F-value” occurs because of noise.

Figure 5 shows the actual and predicted percentage of residual oil recovery. It was found that the coefficient of determination, $R^2$, and adjusted $R^2$ were 0.9582 and 0.9268, respectively, as shown in Table 5. The value is considered acceptable because it is very close to unity. The value of $R^2$ describes the closeness of the selected model to the experimental data points. Meanwhile, the adjusted $R^2$ measures the amount of variation about the mean explained by the model. The predicted $R^2$ value of 0.7092 is not as close to the adjusted $R^2$ value. The finding might indicate a significant block effect or potential problem with the model or laboratory data. 31 Issues to be addressed are model reduction, response transformation, experimental run repetition, and outliers. Table 5 shows an adequate precision greater than 4 (22.966), which portrays adequate model discrimination.

4.3. Effect of Independent Variables. As mentioned earlier, RCCD by RSM was used to study the individual and interaction effects of the three independent variables on residual oil recovery from POME model solution. The three-dimensional response surface plots for the results achieved are shown in Figures 6, 7 and 8 where each shows the combined effect of the experimental parameters. Based on ANOVA analysis, circulation flowrate ($X_C$) was found to have the main impact on the residual oil recovery from the POME model solution in comparison to the other variables. The finding is illustrated by the high F value of 11.13 for the circulation flowrate. Meanwhile, the coolant temperature and freezing time have been found to not have a significant positive impact on the oil recovery to high p-values (>0.05).

However, all the interactions between the variables were found to be significant on the oil recovery. The interaction between the coolant temperature and freezing time ($X_A X_B$) demonstrated the major effect on the response variable as compared to the other interactions ($X_A X_C$ and $X_B X_C$) provided by the high F value of 76.86. 32 Figure 6 shows the change of the percentage of residual oil recovery concerning coolant temperature and freezing time which is depicted at a fixed circulation flow speed of 500 rpm. In this experiment, the coolant temperature and freezing time were varied from 4 to 12 °C and 20 to 60 min, respectively. It was observed that with the increment of freezing time to 50 min and coolant temperature at 6 °C, the percentage of oil recovery reached the maximum at 92.56%. Therefore, at a lower coolant temperature and long period of freezing time, the amount of residual oil recovered was higher than that at a higher temperature. As indicated in Figure 6, the increase in oil recovery caused by the increase of freezing time at a constant coolant temperature was greater than the increase of coolant temperature at a constant freezing time. Theoretically, a lower temperature leads to a decrease in the oil and grease value in
the liquid phase. Coolant temperature is highly related to the freezing rate, thus making it an important parameter in the PF. In another study by Yahya et al. (2015), decreasing the coolant temperature from 29 to 24 °C enhanced the quality of iodine value (IV) of olein and lowering the effective partition constant ($K$) resulted in more soluble solids to remain in the concentrate. This leads to a higher purity of ice solids. The interaction between the temperature and time was the most effective parameter in this study.

**Figure 5.** Predicted vs actual values plot for oil recovery (%).

**Figure 6.** 3D surface plot of combined effect of coolant temperature and freezing time at a circulation flowrate of 500 rpm.

**Figure 7.** 3D surface plot of a combined effect of coolant temperature and circulation flowrate at a constant freezing time of 50 min.

**Figure 7** shows the surface plot for the oil recovery as a function of coolant temperature and circulation flowrate ($X_1 X_2$) at a constant freezing time of 50 min. The residual oil shows a slowly decreasing trend with coolant temperature, whereas it increases gradually with the increase of flow speed. In this PF process, the peristaltic pump had been used to ensure a proper flow and mixing inside the crystallizer. Thus, the circulation flowrate plays a critical role to increase oil removal from the solution. As can be observed, when the flow speed was less than 300 rpm, the amount of oil extracted was low (73%). The result may be because the POME model
solutions do not mix well, which later can decrease the production of ice in the PF process. On the other hand, the higher speed of the pump causes higher residual oil to be obtained through the PF process. The result is consistent with the previous report by Ojeda et al. (2017), where the study used a sequence of increasing stirring speed of 0, 500, 800, and 2100 rpm for progressive freeze concentration of sucrose solution and concluded that the best value of the concentration index was obtained at a higher stirring speed (800 rpm). This result is explained by the increasing mass transfer rate of solutes from the ice front to the liquid fraction because of the fluid motion. However, the highest speed (2100 rpm) does not substantially enhance the final concentration acquired, which as a result of higher heat produced at high agitation and the increases in viscosity affects the energy balance in the ice formation. In this study, speed is the most influential factor to be controlled in order to get a higher percentage of oil recovery.

Figure 8 shows the combined effect of freezing time ($X_0$) and circulation flowrate ($X_C$) on residual oil recovery at a constant coolant temperature of 6°C. From the plot, it can be seen that the interaction between freezing time and pump speed has insignificant effects on oil recovery from the POME model, represented by the low value of the $F$ value (11.33) as compared to the other interactions. The 3D surface plot reveals that the oil yield increased, reaching a maximum of 92.56% at the highest value of freezing time (50 min) and circulation flowrate (500 rpm). In other words, the percentage of oil recovery increased with the increase in the freezing time and pump speed. In the PF process, the longer freezing time could provide higher concentration efficiency. By thoroughly controlling the duration per run of the PF process, the highest purity of solids could be obtained. If concentration of the

Figure 8. 3D surface plot of combined effect of freezing time and circulation flowrate at a constant coolant temperature of 6°C.

Figure 9. Optimal region on the coolant temperature and freezing time for maximization of oil recovery from POME.
solute in the concentrate is considered, the longer duration could produce higher solute concentration in the concentrate, in which higher efficiency is achieved.38 Moreover, Amran et al. (2018) reported the same results where the highest solute recovery (96%) with the lowest effective partition constant (K) was obtained at 50 min circulation time.39 A thicker layer of solids was obtained through longer the freezing time.

4.4. Selection of Optimal Levels and Estimation of the Optimum Response Characteristic. The main objective of this experimental study is to optimize the separation conditions in order to get oil recovery from POME. Optimization of the PF parameter was carried out using a numerical optimization method. Figures 9 and 10 show the response surface and contour plot obtained at the optimum PF condition, represented by the maximum oil recovery. The optimum operating conditions for residual oil recovery from the POME model by the PF process were observed at the coolant temperature of 6 °C, freezing time of 50 min, and circulation flowrate of 500 rpm. The optimum conditions obtained in this study aligned with the past studies, where the lower coolant temperature, higher freezing time, and circulation flowrate are favorable in the PF process.40,41 The predicted and experimental values for the residual oil recovery from the POME model were obtained as 92.56 and 93.2% at optimum conditions, respectively. The results achieved are relevant to another optimization study of enzymatic sludge palm oil recovery reported by Norhayati (2013), where 81.95% residual oil was recovered with an R-squared value of 0.852.21 A comparison between predicted and experimental results has been made to check the accuracy of the proposed optimum

| parameters          | optimum conditions |
|---------------------|--------------------|
| coolant temperature | 6 °C               |
| freezing time (min) | 50                 |
| circulation flowrate (rpm) | 500           |
| oil recovery (%)    | predicted: 92.56   |
|                     | experimental: 93.20|

Table 6. Experimental and Predicted Value of Oil Recovery, Y at Optimum Operating Conditions

Figure 10. Contour plot of coolant temperature and freezing time on oil recovery at optimum conditions.
conditions and the developed model. The results indicate that the error was less than 0.69%, and it was concluded that the results obtained agree quite well with the predicted ones. The detail of optimization is shown in Table 6.

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**ABBREVIATIONS2-COL**
- ANOVA: analysis of variance
- BST: bulk storage tank
- CFC: circulated freeze-crystallizer
- CPO: crude palm oil
- OFAT: one factor at a time
- POME: palm oil mill effluent
- PF: progressive freezing
- RCCD: rotatable central composite design
- RSM: response surface methodology

**SYMBOLS2-COL**
- $X_A$: [°C] coolant temperature
- $X_B$: [minutes] freezing time
- $X_C$: [rpm] circulation flowrate
- $Y$: [%] oil recovery
- $Y_0$: [mg L$^{-1}$] initial oil and grease value
- $Y_1$: [mg L$^{-1}$] final oil and grease value

**SUBSCRIPT AND SUPERSCRPT2-COL**
- 2FI: 2-factor interaction
- $2^n$: factorial runs
- $2n$: axial runs
- $n_C$: replication of center points

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