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Cryogenic Freezing Process Optimization based on Desirability Function on the Path of Steepest Ascent

R. Uporn, and P. Luangpaiboon

***Abstract*—**This paper presents a comparative study of statistical methods for the multi-response surface optimization of a cryogenic freezing process. Taguchi design and analysis and steepest ascent methods based on the desirability function were conducted to ascertain the influential factors of a cryogenic freezing process and their optimal levels. The more preferable levels of the set point, exhaust fan speed, retention time and flow direction are set at -90°C, 20 Hz, 18 minutes and Counter Current, respectively. The overall desirability level is 0.7044.

***Keywords*—**Cryogenic Freezing Process, Taguchi Design and Analysis, Response Surface Method, Steepest Ascent Method and Desirability Function Approach.

I. Introduction

THE Frozen food industry is prevalent today and has variable types of freezing technology. One type of freezing that was popular and high productivity is Cryogenic Freezing by Liquid Nitrogen. It provides the high cooling performance and high freezing quality of frozen products. The major problem of users or operators in the freezing process are to determine the influential factors and their optimal levels for freezing the process and to determine the relationship of each factor to control and configure the proper condition of the freezer to the highest quality. Therefore, the aim of this research is to study and determine the effective factors and their optimal levels of the freezing process. In this case study the cryogenic freezing system uses the freezer model QS Freezer with the fresh pork to serve as the representative of meat product group.

In the frozen food processing, the quality that the users or customers need are the losing weight after freezing process with minimal weight loss, texture and quality of the product as close to fresh as possible. Therefore, this research chooses to study with the weight loss after freezing and thawing processes (percentage of freezing loss and percentage of drip loss thawing) and the product texture via the shear force value with texture analyzer (cutting force). The statistical design and analysis of experiments is applied as a tool to analyze and compare the experimental results. This tool consists of Taguchi orthogonal design and analysis (TDA) and the steepest ascent method (SAM) based on the desirability function approach (DFA). The DFA is used because there are various responses under a consideration. It is necessary to convert the value of multiple responses to single response for comparing the results of the current operating condition with the new designed condition.

To maintain the quality and extend the shelf life of food products is one of the greatest challenges because the perishable is the loss of the quality and value of food products including nutritional value. Therefore, the chilling and freezing processes are popular for the food preservation. This research focuses to study on a freezing process to reduce the temperature of the food cool down to -18°C or lower. That temperature level is the point of the water inside product changed to ice crystals with no biochemical reaction. This will maintain the quality of food tissue. There are four classified types of the freezer according to the principle of refrigeration. They consist of air freezing, contract plate freezing, liquid-immersion freezing or direct immersion freezing and cryogenic freezing.

Freezing is a process which a product changes its physical state from liquid to solid and reduces its temperature to one of storage or below -18°C. During the process three stages can differentiate with the pre-freezing stage. It is the time period between the moment at which a product at an ambient temperature is subjected to a freezing process and the moment at which the water starts to crystallize. This stage is relatively short and the amount of heat removed is small. Freezing stage is the period during which the temperature is almost constant, because the heat being extracted is causing the main part of the water to change phase into ice. The longer stage and the enthalpy change is the highest and a reduction to the storage temperature is the period of time during which the temperature is reduced from the temperature at which most of the "freezable" water has been converted to ice, to the intended final temperature. The enthalpy change is very small during this stage. During the phase change of water to ice, ice crystals were growth inside food cell. The shape and size of ice crystal depend on freezing rate.

It is a generally accepted postulation that the quality and texture of frozen food is enhanced by minimizing the size of

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ice crystals formed during the freezing process. The faster the product goes through this curve, especially at the freezing stage the smaller the ice crystals and therefore the quality of the frozen product is increased. The water contained in food products is found in the tissues between the cells (extra cellular fluid) and inside the cells (intercellular fluid) [1]. These fluids have different concentrations, the concentration of salts being higher inside the cells. The cell membrane acts as an osmotic barrier maintaining this difference of concentration.

When the cold is applied to a product, the first fraction to become ice will be the extra cellular fluid as it has a warmer freezing point than the fluid inside the cells. If the freezing rate is low it will provoke a migration of the water from inside the cells which will start freezing on the surface of the extra cellular crystals already formed. As the cells loose the water, the remaining solution becomes more and more concentrated and their volumes shrink causing the wall cells to collapse. The final frozen product is formed by large and irregularly shaped ice crystals. When this slow frozen product is thawed, part of the intracellular fluid that has crystallized outside the cells will drop out taking away with it part of the flavor and nutritional constituents (drip loss).

A high freezing rate will have an opposite effect in the crystallization. The water between cells will start freezing very fast, and besides, a big number of small crystals will form inside the cells, the water migration from the cells being very small. As the size of the crystals is small and their shape regular, there is no pressure to break the cell membranes. The losses of cellular fluids in the thawing process are almost non-existent if the freezing rate has been high enough (it will also depend slightly on the product texture). Cryogenic freezing is the highest freezing rate as the ultra rapid freezing rate, which can decrease the temperature of the frozen products to fast cause of the properties of cryogen substance that used has low boiling point, one of the cryogen substances that have been popular and is used in this research is Liquid Nitrogen (LIN).

Liquid nitrogen is a product of the separation of the air and produce oxygen as a byproduct of the production, which is stored in a tank at a pressure of 2 kilograms per square centimeter at an absolute pressure liquid nitrogen to a temperature of-196°C, which is to be used for the benefit of frozen food and other industries. There are both continuous and discontinuous for the freezer by used the liquid nitrogen system. For the continuous, frozen foods will moved into the tunnel of freezer, along the belt contact with cool air, direct contact with sprayed liquid nitrogen. Rate frozen by this method has been very fast. In this case study on the cryogenic freezer by liquid nitrogen the model of QS Freezer, which is the continuous tunnel freezer, can be set some process factors. Those are the set point, the speed of conveyor drive motor for controlling the retention time and the exhaust fan motor used to control the air flow direction of freezer. Moreover, there are two types of flow direction which consist of co-current (product and cold air flow as the same direction) and counter current (product move along tunnel convert with cold air flow direction). Fig. 1 showed the QS Freezer and its working principle.

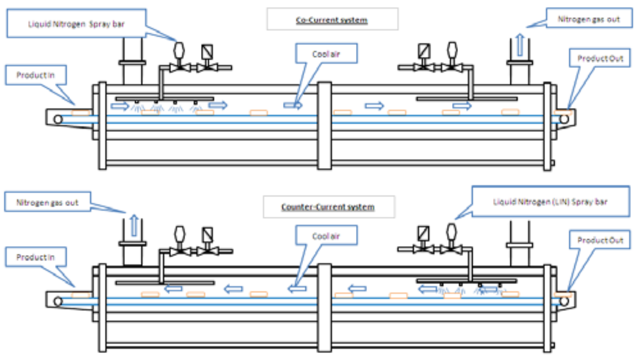

Fig. 1 The principle of QS Freezer

II. Related Methods

A. Completely Randomized Design (CRD) or One Way-ANOVA

This is an experimental design for single factor experiment. Its design varies to determine the degree to which the different factor levels (a) affect the mean of the response significantly or not. The hypothesis of CRD is shown as:

$$H_0 : \mu_1 = \mu_2 = \cdots \mu_a \quad (1)$$
$$H_0 : \mu_i \neq \mu_j \text{ , at least 1 pair.}$$

Linear statistical model of the CRD can be shown as below.

$$y_{ij} = \mu + \tau_i + \varepsilon_{ij} \begin{cases} i = 1,2, \dots, a \\ j = 1,2, \dots, n \end{cases} \quad (2)$$

where y_{ij} is the j^{th}observation of the i^{th} treatment, μ is the population mean, τ_i is the effect of the i^{th}treatment and ε_{ij} is the ij^{th}random error or residual. One way-ANOVA is the upper one tail test that will reject the null hypothesis when $F_0 > F_{\alpha,a-1,N-a}$ The ANOVA is shown in Table I.

TABLE I
ANOVA Table for the CRD

Source of Variation	Sum of Squares	dfdf	Mean Squares	F_0
Between Treatments	$SS_{treatment}$	a-1	$MS_{treatment} = \frac{SS_{treatment}}{a-1}$	$F_0 = \frac{MS_{treatment}}{MS_E}$
Within Treatments	SS_E	N-a	$MS_E = \frac{SS_E}{N-a}$	
Total	SS_T	N-1		

B. Desirability Function Approach

Desirability Function is used to consider the responses and serve to turn a multiple response optimization problem into a single response one, which depends on the number of independent variables. The value of satisfaction for the desirability or $d_i(\hat{y}_i)$ will be between 0 and 1. When $d_i(\hat{y}_i)$ is equal to zero it means that the response is outside the scope of recognition but if d is increased, the satisfaction will increase as well. When $d_i(\hat{y}_i)$ is equal to one it is completely satisfied. In this research there are two types of transformation for predicted responses that are the nominal-the-best (3) and smaller-the-best types (4).

Smaller the Best

$$d_i(y_i) = \begin{cases} 1 & ;\ if\ \hat{y}_i \le y_i^{MIN} \\ \left(\frac{y_i^{MAX} - \hat{y}_i}{y_i^{MAX} - y_i^{MIN}}\right)^{x_i} & ;\text{if}\ \ y_i^{MIN} \le \hat{y}_i \le y_i^{MAX} \\ 0 & ;\ if\ \hat{y}_i \ge y_i^{MAX} \end{cases} \quad (3)$$

Target the Best or Nominal the Best

$$d_i(\hat{y}_i) = \begin{cases} 0 & ,\ \text{if}\ \ \hat{y}_i \le y_i^{min}\ \text{or}\ \hat{y}_i \ge y_i^{max}, \\ \left[\frac{\hat{y}_i - y_i^{min}}{T_i^{min} - y_i^{min}}\right]^{P1_i} & ,\ \text{if}\ \ y_i^{min} < \hat{y}_i \le T_i^{min}, \\ \left[\frac{y_i^{max} - \hat{y}_i}{y_i^{max} - T_i^{max}}\right]^{P2_i} & ,\ \text{if}\ \ T_i^{max} < \hat{y}_i \le y_i^{max}, \\ 1 & ,\ \text{if}\ \ T_i^{min} < \hat{y}_i \le T_i^{max} \end{cases} \quad (4)$$

where $d_i(\hat{y}_i)$ is the desirability function of $\hat{y}_i$, varies from 0-1. y_i^{min} and y_i^{max} are the lower and upper bounds, respectively. T_i^{min} and T_i^{max} are, respectively, the lower and upper target of the response. In order to aggregate the individual desirability the composite desirability of D will be calculated by (5)

$$D = \sqrt[n]{d_1(\hat{y}_1) + d_2(\hat{y}_2) + \cdots + d_n(\hat{y}_n)} \quad (5)$$

where D is a composite desirability, d_i is the individual desirability and n is number of desirability functions [2].

C. Taguchi Design and Analysis on the DF (TDADFA)

Taguchi's method is an efficient tool to help for improving the quality. It is done by selecting the appropriate factors with orthogonal array to reduce the number of experiments and suit with the condition. The use of standard orthogonal array depends on the level and the number of factors. L is used as the symbol along with the number which indicates the number of experiments allowed for an implementation [3-4]. In this case study, the significant factors are chosen via the preliminary study based on the CRD analysis. All four significant factors of the set point, the exhaust fan speed, the retention time and the flow direction are used to determine the relationship with all three responses. They consist of a percentage of the freezing loss (Y_1), a percentage of drip loss thawing (Y_2) and the cutting shear force value (Y_3) including the possible controlled ranges (Δ) (Table II).

TABLE II
DESCRIPTION OF FACTORS AND THEIR LEVELS

Factor	Unit	Symbol	Feasible level	Δ	Current level
Set Point	°C	X_1	-60 to -100	5	-80
Exhaust Fan Speed	Hz	X_2	20 to 40	5	30
Retention Time	Minute	X_3	5 to 25	3	15
Direction Flow	Newton (N)	X_4	Co/Counter Current		Co-Current

In this case, there are four factors and two levels and the interesting array with some interactions will be created with the experiments of $L_8(2^7)$ orthogonal array (Table III).

TABLE III
$L_8(2^7)$ ORTHOGONAL ARRAY

Experimental run	Factors						
	X_1	X_2	X_1X_2	X_3	X_1X_3	X_2X_3	X_4
1	1	1	1	1	1	1	1
2	1	1	1	2	2	2	2
3	1	2	2	1	1	2	2
4	1	2	2	2	2	1	1
5	2	1	2	1	2	1	2
6	2	1	2	2	1	2	1
7	2	2	1	1	2	2	1
8	2	2	1	2	1	1	2

D. Steepest Ascent Method (SAMDFA)

Response surface method is useful for modeling and analyzing the industrial problems. A response of interest is affected by several factors and the objective is to optimize this response. If there are two factors of x_1 and x_2 to maximize the yield (y) of a process is determined via the following relationship.

$$y = f(x_1, x_2) + \varepsilon \quad (6)$$

where the function of f is unknown. Assume that the first-order model is an adequate approximation to the true surface in a small region of the x's. The steepest ascent method (SAM) is a procedure for moving sequentially along the path of steepest ascent, that is, the direction of maximal increased response, based on the firth order model,

$$\hat{y} = \hat{\beta}_0 + \sum_{i=1}^{k} \hat{\beta}_i x_i \quad (7)$$

The first-order response surface, that is, the contour of $\hat{y}$, is a series of parallel line such as that shown in Fig. 2. The direction of steepest ascent is the direction in which $\hat{y}$ increases most rapidly. The steps along the path are proportional to the regression coefficients of $\{\hat{\beta}_i\}$. The second order is later applied to approximate the true relationship between y and the independent variables by the polynomial model as followed [5-6].

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i<j} \beta_{ij} x_i x_j + \varepsilon \quad (8)$$

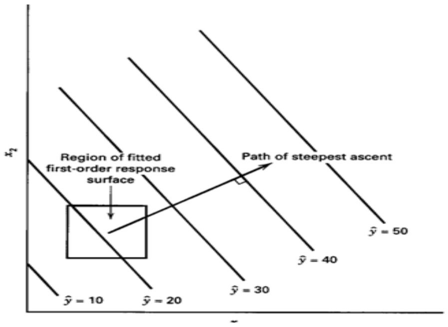

Fig. 2 First-order response surface improvement via the path of steepest ascent

III. Quality Measurement of Frozen Food

The ultra-structure of pork changes during freezing. Frozen storage and thawing affect the pork appearance and water-holding capacity. Therefore, there is a need to prepare and measure the product samples and the details are followed. This study used the fresh pork from BETAGRO Company, a premium brand HYMEAT. The animals were reared intensively and fed with concentrate until they reached a commercial slaughter weight. The carcasses, pigs between 22 and 24 weeks of age, were slaughtered, that had been stored for 24 hours up to the maximum of 48 hours at 4 ^{o}C after slaughter. Each of pork loin was cut into each 5cm x 10cm x 2cm cutlets, giving a sample weight of approximately 80-100 grams.

Use cloth to absorb excess water on sample surface to ensure that the no weight loss from dripping of excess water. After that each sample was weighted and recorded as an initial weight before freezing with 10 samples for each freezing treatment and an additional one for checking the temperature. The thermocouple was then put at the center of sacrificed sample to capture core temperature of food sample. After that, pork samples were put into the QS Freezer until the core temperature reached -20^{o}C or lower. Prepare plastic bags to vacuum pack sample after they were frozen and weighted an empty plastic bag.

A. Percentage of freezing loss

Take out the frozen samples at the freezer out-feed and immediately vacuum pack in weighted plastic bag to minimize condensation on sample surface which will affect to final sample weight. Record weight of the packed frozen sample and calculate the freezing weight loss. Then, keep the frozen sample in freezing cabinet at -18^{o}C for 24 hr before measuring drip loss.

$$\%Freezing\ Loss = \frac{(initial\ weight\ of\ pork - weight\ of\ frozen\ pork\ in\ pack - plastic\ bag\ weight) \text{x } 100}{initial\ weight\ of\ pork} \quad (9)$$

B. Percentage of drip loss thawing

After the predetermined storage period at least 24 hours, take frozen sample out and unpack from plastic back. Arrange on tray, the meat samples were thawed in a thermostatically controlled room at 4^{o}C for 10 hours in order to ensure that the temperature of all pork samples had reached 4^{o}C. Calculate the thawing drip loss.

$$\%Drip\ Loss\ Thawing = \frac{(weight\ of\ frozen\ pork - weight\ of\ thawed\ pork\ ) \text{x } 100}{weight\ of\ frozen\ pork} \quad (10)$$

C. Cutting Shear Force resistance Analysis

Prepare pork sample for cutting test with 10 pieces of raw pork and thaw the sample pork in each treatment, cut longitudinally to the muscle fibers, 5 pieces of 1cm x 1cm x3cm for each pork sample. Measure the sheared cut perpendicular to muscle fiber force of the sample by Texture analyzer, brand INSTRON model Bluehill2, by blade set with knife with 25 kilograms load cell, speed of blade cut was 2 mm per second. Record the cutting force value in Newton unit.

IV. Experimental Results

A. TDA Results

For Taguchi design and analysis low and high levels were selected based on the current operating condition (Table IV). After coding the natural levels to transformed levels the factors and some interaction were put in the orthogonal array (Table V).

TABLE IV
Selected Factors and Their Levels

Factor	Symbol	Level	
		1 (Low)	2 (High)
Set Point	X_1	-80	-100
Exhaust Fan Speed	X_2	20	30
Retention Time	X_3	15	18
Direction Flow	X_4	Co-Current	Counter Current

TABLE V
$L_8(2^7)$ Orthogonal Array for the TDA

Run#	X_1	X_2	X_3	X_4
1	-80	20	15	Co-Current
2	-80	20	18	Counter Current
3	-80	30	15	Counter Current
4	-80	30	18	Co-Current
5	-100	20	15	Counter Current
6	-100	20	18	Co-Current
7	-100	30	15	Co-Current
8	-100	30	18	Counter Current

All the experiments were run and analyzed the responses categorized by their types (Table VI).Percentage of freezing loss and drip loss thawing need to reach the minimum whereas the cutting force needs to close to with raw pork, in this case equal to 19.26 N. The experimental results of Taguchi method for each response are shown as in Table VII.

TABLE VI
RESPONSES AND THEIR TARGETS

Response	Symbol	Target	Type of target
Freezing Loss	y_1	minimum	smaller the better
Thawing Drip Loss	y_2	minimum	smaller the better
Cutting Force	y_3	close to raw product	nominal the best

TABLE VII
TDA EXPERIMENTAL RESULTS

Run#	X_1	X_2	X_3	X_4	y_1	y_2	y_3
1	-80	20	15	Co-Current	1.0943	4.8310	21.64
2	-80	20	18	Counter Current	1.2835	4.4236	36.00
3	-80	30	15	Counter Current	1.0885	3.9239	26.79
4	-80	30	18	Co-Current	0.9925	5.3469	18.29
5	-100	20	15	Counter Current	0.9835	4.4618	29.07
6	-100	20	18	Co-Current	0.5648	6.2423	21.54
7	-100	30	15	Co-Current	0.5109	6.8163	19.04
8	-100	30	18	Counter Current	1.0101	3.7462	24.92

Based on the results of all the responses by the TDA, the influential factors and interaction effects on the response of percentage of freezing loss (Y_1) were X_1, interaction between X_2X_3 and X4 The level of each factor should be set as X_1 at level 2 (-100°C), X_2 at level 2 (30Hz) and X_3 at level 2 (18 minute) and X_4 at level 1 (Co-Current). Influential factor on the response of percentage of drip loss thawing (Y_2) was X_4 and should be set at level 2 (Counter Current), other remaining factor can be set at the current level. Influential factors and interaction effects on the response of cutting force (Y_3) were factor X_4 and X_2. The process settings for X_4 should be at the level 1 (Co-Current), X_2 at level 2 (30Hz).

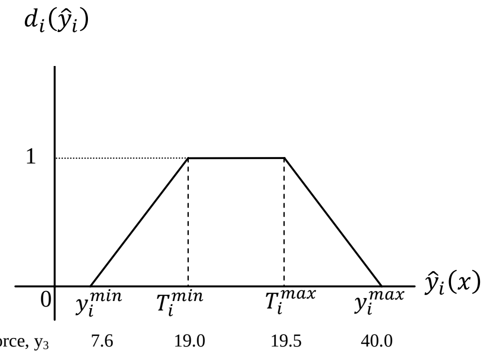

Fig. 4 Desirability transformations for Y_3

TABLE IX
ORTHOGONAL ARRAY AND DESIRABILITY FUNCTION LEVELS

Run#	X_1	X_2	X_1X_2	X_3	X_1X_3	X_2X_3	X_4	d_1	d_2	d_3	D
1	1	1	1	1	1	1	1	0.3381	0.5476	0.8958	0.5494
2	1	1	1	2	2	2	2	0.1804	0.6084	0.1952	0.2778
3	1	2	2	1	1	2	2	0.3429	0.6830	0.6446	0.5325
4	1	2	2	2	2	1	1	0.4229	0.4706	0.9296	0.5698
5	2	1	2	1	2	1	2	0.4304	0.6027	0.5332	0.5172
6	2	1	2	2	1	2	1	0.7794	0.3370	0.9005	0.6184
7	2	2	1	1	2	2	1	0.8242	0.2513	1.0000	0.5917
8	2	2	1	2	1	1	2	0.4082	0.7095	0.7248	0.5943

In addition, it can be analyzed the individual desirability ($d_i(\hat{y}_i)$) and composite desirability (D) of the response to specify the treatment with the highest desirability by a use of the specification and target for each response (Table VIII). For Y_1 and Y_2, the one-side transformations given by (3) were used (Fig. 3) and Y_3, the two-side transformations given by (4) was used (Fig. 4).The desirability values are shown in Table IX.

TABLE VIII
SPECIFICATION AND TARGET FOR DESIRABILITY FUNCTION

Response	Spec	Target	Type
% Freezing Loss, Y_1	0.3 - 1.5		Smaller the best
% Thawing Drip Loss , Y_2	1.8 - 8.5		Smaller the best
Cutting Force , Y_3	7.6 - 40	19.0 -19.5	Nominal the best

For the TDA with the composite desirability, the effective factors and their interaction effects with the highest desirability level were X_4, X_1, X_1X_3, X_2, X_1X_2 and X_2X_3. The level of the factor X_1 should be set at level 2 (-100°C), X_2 at Level 2 (30Hz), X_3 at level 2 (18 minute) and X_4 at level 1 (Co-Current).

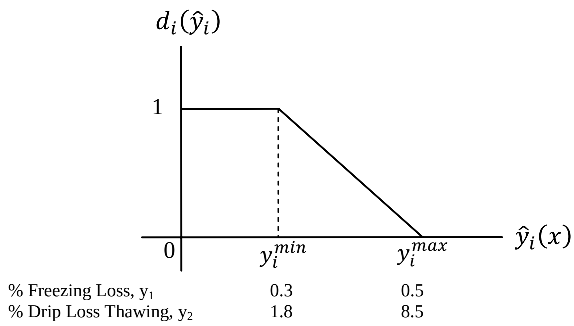

Fig. 3 Desirability transformations for Y_1 and Y_2

B. SAM Results

The values of composite desirability (D) were calculated from experimental runs of the orthogonal array. The linear regression was analyzed to form to relationship of D and all influential factors. The regression equation was given below.

$$D = 0.537 + 0.0980X_1 - 0.102X_4$$

TABLE X
REGRESSION ANALYSIS BASED ON DESIRABILITY FUNCTION

Predictor	Coef	SE Coef	T	P
Constant	0.5327	0.1404	3.83	0.012
X_1	0.09802	0.06444	1.52	0.189
X_4	-0.10189	0.06444	-1.58	0.172

S= 0.0911252 R-Sq = 49.1% R-Sq(adj) = 28.7%

Analysis of Variance

Source	DF	SS	MS	F	P-Value
Regression	2	0.039978	0.019989	2.41	0.185
Residual Error	5	0.041519	0.008304		
Total	7	0.081497			

Source	DF	Seq SS
X_1	1	0.019215
X_4	1	0.020763

The path of the steepest ascent is then generated via the equation below.

$$x_i^T = x_i + a\left(\frac{\hat{\beta}_i}{\sqrt{\hat{\beta}_1^{\ 2}+\cdots+\hat{\beta}_k^{\ 2}}}\right) \quad (11)$$

$$x_1^T = x_1 + a\frac{(0.0980)}{0.14145} \ or \ x_1 + a(0.69)$$
$$x_4^T = x_4 + a(0.72)$$

TABLE XI
DESIRABILITY LEVEL FORM THE STEEPEST ASCENT METHOD

Run#	Design Point	X_1	X_4	D
1	Original	-100	Co-Current	0.6184
2	1st	-95	Counter Current	0.6727
3	2nd	-90	Counter Current	0.7044

V. CONCLUSION

This research focuses on the cryogenic freezing process optimization by designed experiments and response surface methods. The mechanism and characteristics of cryogenic freezing process are very complicated and conducted to investigate. In addition, the completely randomized design was applied to a preliminary study, the analysis of variance was performed to determine the optimal combination of process factors which consist of set point, exhaust fan speed, retention time and direction flow. The desirability function approach of the nominal-the-best and smaller-the-better was used to compromise the multiple responses of freezing loss, thawing drip loss and cutting force into single response called the overall desirability. Firstly, the Taguchi design and analysis based on the natural and coded variables were developed from the statistically significant parameters. Secondly, the multi regression model in forms of the path of steepest ascent moved the region of experimental region toward the design point with the maximal D level. The experimental results showed that there is a significant D increase to the level of 0.7044. The optimal condition of process variables are set point at -90°C, exhaust fan speed at 20 Hz, retention time at 18 minute and direction flow at Counter Current mode**.**

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