



## MATHEMATICAL MODEL STUDY TO OPTIMIZE THE FREEZE DRYING PROCESS FOR PRODUCTION OF DRIED YOGURT

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### ABSTRACT

The aim of this study was to build mathematical models for optimizing a technological process producing a freeze-dried yogurt product with good quality based on solving multi-objective optimization problems. The application of Utopia Point Method for the optimization process determined the optimal freeze-drying conditions including drying temperature of 36.6°C, drying pressure of 0.023 mmHg and drying time of 35.6 hours. The optimal drying process resulted in the freeze-dried yogurt product with a moisture content of 0.963%, a crispiness of 15.953 mN and 69.291% of viable beneficial microorganisms were preserved. In addition to the good quality criteria of the dried product, the drying process also consumed only 19.94 kWh of electrical energy to produce 1 kg of product, which suggests the high production applicability of the developed freeze-drying process.

## 1. Introduction

Yogurt is usually produced as semisolid food products, which derived from processed animal milks under appropriate fermentation conditions with the involvement of *Streptococcus salivarius* ssp *thermophilus* and *Lactobacillus delbrueckii* ssp *bulgaricus* bacteria (Bamforth C. W. and Cook D. J., 2019). The combination of nutritional components and live microorganisms forms the basis for a highly nutritious food that can easily complement a healthy dietary regimen (German J. B., 2014). Yogurt possesses a diverse and balanced chemical composition, including carbohydrates, proteins, lipids, minerals, and various vitamins. Lactose constitutes approximately 98% of the carbohydrates and 54% of the total solids in non-fat yogurt, along with small amounts of galactose, glucose, and oligosaccharides (Yildiz F., 2010). The protein content of yogurt is improved compared to raw milk, making yogurt a rich source of biologically active and plentiful

protein, providing all essential amino acids and containing growth factors and precursors for bioactive peptides (Yildiz F., 2010). The fat content of yogurt primarily consists of triglycerides, accounting for about 98%, with the remainder comprising phospholipids, cholesterol, and  $\beta$ -carotene. Traditional whole-milk yogurt contains 3 to 4 g of lipids per 100 g, of which 65% are saturated fatty acids. The remaining portion consists of 31% monounsaturated fatty acids and 4% polyunsaturated fatty acids (Marette A., Picard-Deland É., and Fernandez M., 2017). The mineral composition of yogurt and dairy products includes both major elements (Ca, Mg, Na, K, P, and Cl) and trace elements (Fe, Cu, Zn, and Se) (Marette A., Picard-Deland É., and Fernandez M., 2017). Both fat-soluble and water-soluble vitamins are present in milk and yogurt. Full cream yogurt may contain significant amounts of vitamin A, B-complex vitamins, and vitamin D (Marette A., Picard-

Deland É., and Fernandez M., 2017). Additionally, yogurt is a rich source of vitamin B12 (Karmi O., Zayed A., Baraghehi S., Qadi M., and Ghanem R., 2011).

Freeze-drying is a process used to remove water from products by sublimation. The freeze-drying process involves three stages: 1) freezing the raw material, 2) primary drying, and 3) secondary drying (G.Wilhelm. Oetjen and Peter. Haseley., 2004; Bhushani A. and Anandharamakrishnan C., 2017; Dzung N.T, Chuyen H.V, Linh V.T.K, and et al., 2022). Freeze-drying is the most complex method of water removal and finds application primarily in the production of high-value food products. Currently, freeze-drying technology is being used to produce various food products such as instant coffee, tea, meat, herbs, and high quality fruits and vegetables (Dzung N.T, Chuyen H.V, Linh V.T.K, and et al., 2022; Anandharamakrishnan C., 2017). Freeze drying help preserving high quality of food products that are challenging to achieve with other drying methods. Another outstanding feature of this drying method is the structural stability of the product, preventing the collapse of the solid matrix after drying. As a result, a porous, non-caking product is obtained, facilitating rapid rehydration when water is added to the substance thereafter (Athanasios I. Liapis and Roberto Bruttini, 2020). Freeze drying can prevent the denaturation of whey proteins and the Maillard reaction between lactose and protein in milk. In the dairy industry, freeze-drying is mainly employed to preserve original strains of cultures and probiotic microorganisms for use as functional ingredients (Anandharamakrishnan C., 2017). The biological values of freeze-dried bio products endow them with robust survivability, providing an advantage in developing functional dairy components (Fellows P., 2000; Dzung N.T, Chuyen H.V, Linh V.T.K, and et al., 2022).

## 2. Materials and methods

### 2.1. Raw material

Yogurt was prepared using ingredients include full cream milk powder (210g), sugar (37g), starter culture (3g) and water (750g).

Prior to fermentation, yogurt samples were standardized to achieve a consistent dry matter content of 25%.

### 2.2. Equipment

The main equipment used in this study is the DS-12 Freeze Drying System, which was designed, and fabricated by the research team of Associate Professor Dr. Dzung N.T from the Department of Chemical and Food Technology, Ho Chi Minh City University of Technology and Education, Vietnam.



**Figure 2.** The DS-12 Freeze Drying System

### 2.3. Methods

#### 2.3.1. Determination of factors affecting the freeze-drying process

In this study, the factors influencing the yogurt freeze-drying process including the drying temperature ( $Z_1$ , °C), the drying pressure ( $Z_2$ , mmHg), and the drying time ( $Z_3$ , hours) were investigated. These parameters were measured and controlled using temperature sensors, pressure gauges, and time counters integrated within the DS-12 freeze-drying system.

#### 2.3.2. Determination of output responses

- Energy consumption: The energy consumption per unit mass ( $Y_1$ , kWh/kg dried product) was calculated using a wattmeter (Dzung N.T and Phuong V.D., 2016). The formula for calculating the energy consumption is as follows:

$$Y_1 = \frac{P \cdot \tau}{G} = \frac{(U \cdot I \cdot \cos \varphi) \cdot \tau}{G}, \text{ (kWh/kg)} \quad (1)$$

Where: U represents the Voltmeter reading (V); I represents the Ammeter reading (A);  $\tau$  stands for time in seconds (h);  $\cos \varphi$  denotes the power factor; P indicates the value reading on the Wattmeter (kW), G represents the weight of the material.

- Moisture content of the product: The moisture content of the product ( $Y_2$ ) was determined using the convective drying method in a drying oven, as described in AOAC – 927.05. Accurately weighed 5g of finely ground sample was placed in a clean, dry, pre-weighed aluminum dish. The sample was then subjected to the drying cabinet at a temperature of 105 °C until a constant mass was achieved (AOAC International, 2000). The moisture content of the product was determined using the following formula:

$$Y_2 = 100 - \frac{G_i}{G_e} (100 - W_i), \text{ (%) } \quad (2)$$

Where:  $G_i$  represents the initial mass of yogurt before drying (g);  $G_e$  represents the mass of yogurt after drying (g);  $W_i$  represents the initial moisture content of yogurt (%).

- Texture analysis of the product: The texture of freeze-dried yogurt ( $Y_3$ ) was measured based on the structural deformation obtained using the Brookfield CT3 Texture Analyzer equipped with a TA-SBS cylindrical probe. For all measurements, the sample thickness is set to 14 mm. The following parameters are configured: single sample test, probe speed of 1 mm/s, and target distance of 25 mm. The crispiness of a sample was defined as the maximum pressing force (mN) to cause the structural deformation of the sample. The lower pressing force represents better crispiness.

- Preservation of microorganisms: The preservation of microorganisms was determined by measuring the remaining proportion of lactic acid bacteria after each freeze-drying experiment. The total number of lactic acid bacteria in both yogurt samples (before and after drying) was determined using the method

described in the ISO 15214:1998 and following the description by TCVN 7906:2008. The total number of lactic acid bacteria was determined using the following formula:

$$L = \frac{\sum C}{V \cdot \left( n_1 + \frac{1}{10} n_2 \right) d} \quad (3)$$

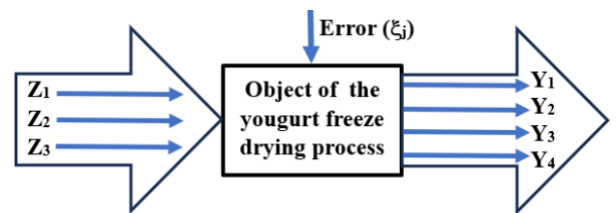
Where:  $\sum C$  represents the total count of viable lactic acid bacteria counted on all plates with at least one plate containing a minimum of 15 lactic acid bacteria colonies; V denotes the volume of the diluted sample plated on each plate, measured in milliliters;  $n_1$  is the number of plates retained in the first dilution step;  $n_2$  is the number of plates retained in the second dilution step; d is the dilution factor corresponding to the plates retained in the first dilution step.

The survival rate of microorganisms, expressed as the percentage of viable lactic acid bacteria ( $Y_4$  %), is determined using the following formula:

$$Y_4 = \frac{L_i}{L_e} \times 100, \text{ (%) } \quad (3)$$

Where:  $L_i$  represents the total number of lactic acid bacteria in 1g of yogurt before freeze drying;  $L_e$  represents the total number of lactic acid bacteria in the corresponding amount of 1g of yogurt before drying.

### 2.3.3. Experimental Design Method



**Figure 1.** Black Box model for experimental design method

The objective functions of the freeze-dried yogurt product in this study are  $Y_1$ ,  $Y_2$ ,  $Y_3$ , and  $Y_4$ , which are closely related to the technological factors  $Z_1$ ,  $Z_2$ , and  $Z_3$ .

A second-order orthogonal experimental design model was constructed with  $k = 3$ . The

variables  $x_1, x_2, x_3$  represent the coded variables of  $Z_1, Z_2,$  and  $Z_3,$  respectively. The experimental mathematical model designed as a second-order orthogonal matrix is described as the following equation:

$$Y_j = b_0 + \sum_{i=1}^k b_i x_i + \sum_{u \neq i; u=1}^k b_{iu} x_u x_i + \sum_{i=1}^k b_{ii} (x_i^2 - \lambda) \quad (5)$$

With  $i, u = 1 \div k; k = 3; j = 1 \div 4$

These variables  $x_1, x_2, x_3$  were coded by variables of  $Z_1, Z_2, Z_3$  presented as follow:

$$x_i = \frac{Z_i - Z_i^0}{\Delta Z_i}; Z_i = x_i \Delta Z_i + Z_i^0 \quad (6)$$

Where:

$$Z_i^0 = (Z_i^{\max} + Z_i^{\min})/2; \Delta Z_i = (Z_i^{\max} - Z_i^{\min})/2; \quad (7)$$

$$Z_i^{\min} \leq Z_i \leq Z_i^{\max}; i = 1 \text{ to } 3 \quad (8)$$

The experimental design model consists of a number of experiments:

$$N = n_k + n_* + n_0 = 2^k + 2k + n_0 = 18 \quad (9)$$

Where:  $k = 3; n_0 = 4$

$\alpha$  value is calculated as:

$$\alpha = \sqrt{\sqrt{N} 2^{(k-2)} - 2^{(k-1)}} = \sqrt{\sqrt{18} 2^{(3-2)} - 2^{(3-1)}} = 1.414 \quad (10)$$

Conditions for obtaining an orthogonal matrix:

$$\lambda = \frac{1}{N} (2^k + 2\alpha^2) = \frac{1}{18} (2^3 + 2 \cdot \sqrt{2}^2) = \frac{2}{3} \quad (11)$$

### 2.3.4. Optimization method

- Single-objective optimization problem: Considering the yogurt freeze drying process as the technological object, the objective functions of interest are  $Y_j = f_j(Z) = f_j(x)$ , which depend on the technological factors  $Z_1, Z_2,$  and  $Z_3$  those were coded as  $x_1, x_2$  and  $x_3$ . These factors form a vector of influencing variables, also known as the variable vector  $Z = \{Z_i\} = (Z_1, Z_2, Z_3)$ , corresponding to  $x = \{x_i\} = (x_1, x_2, x_3)$ , where  $i = 1 \div 3$ . These variables vary within the defined domain  $\Omega_x$ , and the values of the objective function  $f_j(x)$  constitute the value domain  $\Omega_f$

(Dzung N.T and Hai D.T. H., 2016). Hence, the single-objective optimization problem can be established as follows:

Find the optimal solution  $x^{jopt} = (x_1^{jopt}, x_2^{jopt}, x_3^{jopt}) \in \Omega_x$ :

$$\begin{cases} Y_j = f_{j\min} (x_1^{jopt}, x_2^{jopt}, x_3^{jopt}) = \text{Min} \{f_j (x_1, x_2, x_3)\} \\ j = 1 \div 4; \\ \forall x \in \Omega_x = \{-1.414 \leq x_1, x_2, x_3 \leq 1.414\}; \end{cases} \quad (12)$$

- Multi-objective optimization problem: For the yogurt freeze drying process as the technological object, the technological factors  $Z = (Z_1, Z_2, Z_3) \in \Omega_z$ , those were coded as  $x = (x_1, x_2, x_3)$  simultaneously influence multiple objective functions:  $f_1(x), f_2(x), f_3(x), f_4(x)$ . Therefore, it is necessary to concurrently investigate the objective functions  $f_j(x)$  within the same variable space  $\Omega_x$ , varying within the domain  $\Omega_x$ . Hence, a multi-objective optimization problem arises (Dzung N.T and Hai D.T.H., 2016). In the case all single-objective optimization problems seek to find the minimal, the multi-objective optimization problem can be established as follows:

Find the optimal solution  $x^{opt} = (x_1^{opt}, x_2^{opt}, x_3^{opt}) \in \Omega_x$ :

$$\begin{cases} Y_j = f_{j\min} (x_1^{opt}, x_2^{opt}, x_3^{opt}) = \text{Min} \{f_j (x_1, x_2, x_3)\} \\ j = 1 \div 4; \\ \forall x \in \Omega_x = \{-1.414 \leq x_1, x_2, x_3 \leq 1.414\}; \end{cases} \quad (13)$$

The multi-objective optimization problems were solved using the Utopia Point Method:

In the situation that a common solution existing when solving single-objective optimization problems (12) or the solutions to all single-objective optimization problems coincide, which means when  $Y_1 = f_1(x_1, x_2, x_3), Y_2 = f_2(x_1, x_2, x_3)$  and  $Y_3 = f_3(x_1, x_2, x_3); Y_4 = f_4(x_1, x_2, x_3)$  reach the minimum values ( $Y_{1\min}, Y_{2\min}, Y_{3\min}, Y_{4\min}$ ), all optimal solutions  $(x_1^{jopt},$

$x_2^{jopt}, x_3^{jopt} \equiv (x_1^{opt}, x_2^{opt}, x_3^{opt})$  were achieved with all  $j = 1 \div 4$ . Therefore, the utopia optimal method is existent, and  $(x_1^{jopt}, x_2^{jopt}, x_3^{jopt}) \equiv (x_1^{opt}, x_2^{opt}, Z_3^{opt})$  is referred to as the utopia optimal solution of the utopia optimal method. This solution also serves as the solution for the multi-objective optimization problem (13). In this case  $Y^{UT} = (Y_{1min}, Y_{2min}, Y_{3min}, Y_{4min})$  is called utopia point.

In cases where solving single-objective optimization problems (12) doesn't result a common solution, meaning a utopia solution and a utopia optimal method don't exist, the task now changes to solving the multi-objective problem (13) to search for a set of compromise solutions called optimal solutions  $(x_1^{opt}, x_2^{opt}, x_3^{opt})$  which satisfy all the objective functions  $Y_j$  ( $j = 1 \div 4$ ) simultaneously converge to their minimum values.

To find the optimal solution set  $(x_1^{opt}, x_2^{opt}, x_3^{opt})$ , this study employed the utopia point method with the combination norm  $S(x)$ .

Although a utopia solution does not exist, a utopia point still exists as  $Y^{UT} = (Y_{1min}, Y_{2min}, Y_{3min}, Y_{4min})$ . As a result, the combination norm  $S(x)$  is established as follows:

$$S(x) = \sqrt{\sum_{j=1}^m (Y_j - Y_{jmin})^2} \quad (14)$$

With all  $x = (x_1, x_2, x_3) \in \Omega_x$ . Thus, the multi-objective optimization problem is restated as follows: Find  $x^{opt} = (x_1^{opt}, x_2^{opt}, x_3^{opt}) \in \Omega_x$  to satisfy the following requirements:

$$\begin{cases} S_{min} = S(x_1^{opt}, x_2^{opt}, x_3^{opt}) = \text{Min} \left\{ \sqrt{\sum_{j=1}^m (Y_j - Y_{jmin})^2} \right\} \\ \forall x \in \Omega_x = \{-1.414 \leq x_1, x_2, x_3 \leq 1.414\} \end{cases} \quad (15)$$

Solving problem (15) will yield the solution  $x^{opt} = (x_1^{opt}, x_2^{opt}, x_3^{opt}) \in \Omega_x$ . In that case:  $Y_j^S = f_j(x_1^{opt}, x_2^{opt}, x_3^{opt})$ , with  $m = 4$ .

### 3. Results and discussions

#### 3.1. Determination of chemical composition of the raw material

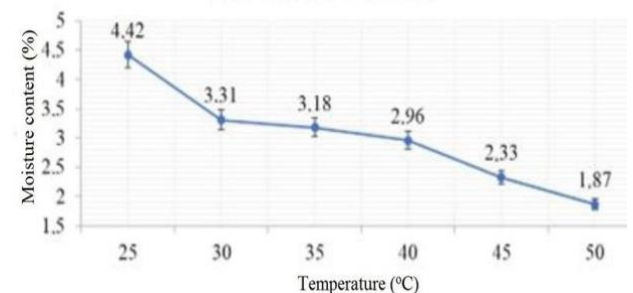
**Table 1.** Chemical composition of yogurt material

N <sub>o</sub>	Composition	Percentage (%)
1	Water	83.0 ± 2.0
2	Protein	2.9 ± 0.1
3	Carbohydrate	10.2 ± 0.2
4	Lipid	3.5 ± 0.1
5	Mineral	0.2 ± 0.0

As shown in Table 1, the water content in yogurt was 83%. Therefore, the requirement to reduce water activity to the desired moisture content would be significant. Low water activity helps inhibit the growth of most bacteria, yeasts, and molds, as well as oxidative reactions and enzymatic activities. Moreover, the removal of water from the product facilitates preservation and transportation (Mawilai P., Chaloeichitratham N., and Pornchaloempong P., 2019; Sogi D. S., Siddiq M., and Dolan K. D., 2015).

#### 3.2. Determination of the appropriate ranges of the input technological factors

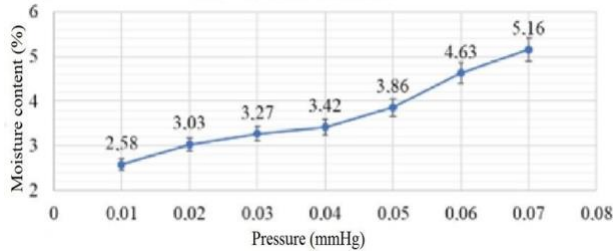
The single-factor experiments were conducted to determine the suitable ranges of temperature, pressure, and time to be used in the optimization model. The results are illustrated in Fig. 2, 3 and 4.



**Figure 2.** Relationship between product's moisture content and drying environment temperature

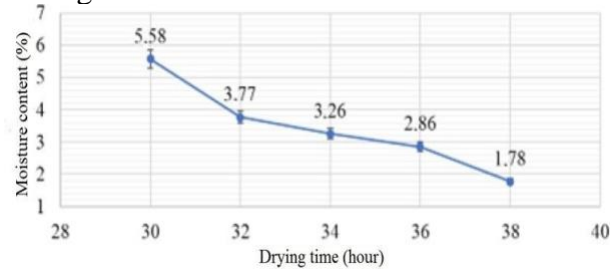


Based on the graph in Figure 2, it is observed that when the drying environment temperature is below 30°C, the product moisture is high. Choosing such a low temperature for drying would prolong the drying time and increase energy costs. Therefore, a reasonable temperature range for drying is between 30°C and 40°C. Within this range, the product moisture remains stable and meets the structural requirements of the final product.



**Figure 3.** Relationship between product’s moisture content and drying environment pressure

Referring to the graph in Figure 3, the data shows that the drying environment pressure has an impact on the product moisture after drying. As the drying environment pressure increased, the product moisture also increased. Thus, it is necessary to select an appropriate pressure range to save energy costs and minimize product losses during the drying process. Accordingly, the chosen pressure range is from 0.02 to 0.04 mmHg.



**Figure 4.** Relationship between product’s moisture content and drying time

Based on the data in Figure 4, it is observed that within the time range from 32 to 36 hours, the product moisture exhibits minimal variation, remaining stable and meeting the required specifications. Therefore, we can select a drying time between 32 and 36 hours for experimental purposes.

### 3.3. Construction of experimental models describing the yogurt freeze drying process

After conducting single-factor experiments to identify appropriate experimental ranges for each technological factor, a central composite design model was constructed and presented in Table 2.

**Table 2.** Data for the levels of influencing factors

Input factors		Z <sub>1</sub> (°C)	Z <sub>2</sub> (mmHg)	Z <sub>3</sub> (h)
Coded experimental levels	-α	32.17	0.016	31.17
	-1	33	0.02	32
	0	35	0.03	34
	+1	37	0.04	36
	+α	37.83	0.044	36.83
Variance range ΔZ <sub>i</sub>		2	0.01	2

From Table 2, a design of a second-order orthogonal experimental matrix was proceeded with a total of 18 experiments based on the combinations of Z<sub>1</sub>, Z<sub>2</sub>, Z<sub>3</sub> (Experimental variables) coded as x<sub>1</sub>, x<sub>2</sub>, x<sub>3</sub> (Coded variables) in Table 3.

The experiments for freeze drying of yogurt were carried out at the different combinations of Z<sub>1</sub>, Z<sub>2</sub> and Z<sub>3</sub> as shown in Table 3. After each experiment, the products are collected and subjected to analysis for determining the values of Y<sub>1</sub> (kWh/kg), Y<sub>2</sub> (%), Y<sub>3</sub> (mN) and Y<sub>4</sub> (%). The results of the objective functions (Y<sub>1</sub>, Y<sub>2</sub>, Y<sub>3</sub> and Y<sub>4</sub>) were recorded and presented in Table 3a and Table 3b.

**Table 3a.** Results for the objective functions in the experimental model

N <sub>o</sub>	Experimental variables			Coded variables			
	Z <sub>1</sub> °C	Z <sub>2</sub> mmHg	Z <sub>3</sub> h	x <sub>1</sub>	x <sub>2</sub>	x <sub>3</sub>	
2 <sup>k</sup>	1	37	0.04	36	1	1	1
	2	33	0.04	36	-1	1	1
	3	37	0.02	36	1	-1	1
	4	33	0.02	36	-1	-1	1
	5	37	0.04	32	1	1	-1
	6	33	0.04	32	-1	1	-1
	7	37	0.02	32	1	-1	-1
	8	33	0.02	32	-1	-1	-1

2k	9	37.8	0.03	34	1.414	0	0
	10	32.2	0.03	34	-1.414	0	0
	11	35	0.044	34	0	1.414	0
	12	35	0.156	34	0	-1.414	0
	13	35	0.03	36.8	0	0	1.414
	14	35	0.03	31.2	0	0	-1.414
n <sub>0</sub>	15	35	0.03	34	0	0	0
	16	35	0.03	34	0	0	0
	17	35	0.03	34	0	0	0
	18	35	0.03	34	0	0	0

**Table 3b.** Results for the objective functions in the experimental model

N <sub>0</sub>		Output responses			
N		Y <sub>1</sub>	Y <sub>2</sub>	Y <sub>3</sub>	Y <sub>4</sub>
		(kWh/kg)	(%)	(mN)	(%)
2k	1	19.64	1.28	27.73	69.34
	2	19.04	1.53	31.49	63.78
	3	20.44	1.26	24.48	77.21
	4	20.04	2.21	30.61	68.66
	5	16.03	2.80	44.95	65.07
	6	15.9	3.51	51.04	67.25
	7	15.92	2.36	35.77	71.41
	8	15.73	2.80	50.11	65.9
2k	9	17.96	0.94	18.31	72.58
	10	16.96	3.50	39.31	66.4
	11	16.76	2.85	41.66	69.38
	12	19.19	0.84	28.13	74.01
	13	22.99	0.86	13.05	72.67
	14	14.94	3.81	41.99	69.07
n <sub>0</sub>	15	17.51	1.65	24.69	65.18
	16	17.62	1.33	21.92	68.56
	17	18.04	1.42	22.83	65.96
	18	17.66	1.21	25.96	74.8

After processing the experimental data, calculating coefficients ( $b_i$ ,  $b_{ui}$  and  $b_{ii}$ ) in the regression equation (5), testing the significance of the regression equation coefficients using the Student's t-test, and checking the compatibility of the regression equation with the experimental results using the Fisher test, we obtained the following regression equations describing the low-temperature vacuum drying process of yogurt material:

- Regression equation describing energy cost:

$$Y_1 = 17.81 + 0.23x_1 - 0.413x_2 + 2.25x_3 - 0.26x_2x_3 - 0.307x_1^2 + 0.45x_3^2 \quad (16)$$

- Regression equation describing product moisture:

$$Y_2 = 1.550 - 0.497x_1 + 0.278x_2 - 0.780x_3 - 0.226x_2x_3 + 0.315x_1^2 + 0.373x_3^2 \quad (17)$$

- Regression equation describing product crispness:

$$Y_3 = 20.042 - 5.001x_1 + 2.781x_2 - 9.04x_3 + 3.586x_1^2 + 6.643x_2^2 + 2.937x_3^2 \quad (18)$$

- Regression equation describing preservation of microorganisms

$$Y_4 = 69.291 \quad (19)$$

The results of the mathematical models indicate that the experimental regression equations  $Y_1$  (kWh/kg),  $Y_2$  (%),  $Y_3$  (mN) and  $Y_4$  (%) describing the energy cost per 1 kg of the product, product moisture content, crispness and the survival rate of microorganisms in the dried yogurt product, respectively, consistently align with experimental data through testing using Fisher's standard. The mathematical models for  $Y_1$ ,  $Y_2$  and  $Y_3$  depended on the temperature of drying environment  $x_1$  ( $Z_1$ , °C), pressure of drying environment  $x_2$  ( $Z_2$ , mmHg) and drying time  $x_3$  ( $Z_3$ , h). Meanwhile, the  $Y_4$  objective function, which describes the beneficial microorganism survival rate for gut health, did not significantly depend on any investigated factors ( $Y_4 = 69.291\% = \text{const.}$ ). Ideally,  $Y_4$  should be 100% because in a low-pressure and low-temperature environment, microorganisms can theoretically survive. However, this loss occurred due to the fact that during the sublimation process, microorganism cells were carried away by steam.

To provide evidence for this, after completing the drying process, an analysis performing with the condensed water sample in the freeze-condensation equipment of the melting system revealed the presence of microorganism cells. This demonstrates that microorganism cells were carried away with the sublimating water vapor.

### 3.4. Solving the optimization problems to determine the technological conditions

#### 3.4.1. Solving the single-objective optimization problem

The experimental results have demonstrated that the objective function  $Y_4$  is independent of  $x_1$  ( $Z_1$ , °C),  $x_2$  ( $Z_2$ , mmHg) and  $x_3$  ( $Z_3$ , h). As a result, it can be excluded from the objective function space or the value domain of the objective function. Consequently, only three objectives  $Y_1$ ,  $Y_2$ ,  $Y_3$  remain in the multi-objective optimization problem (13). However, to solve the multi-objective optimization problem (13) and find the optimal drying technological conditions, the first step is to determine whether the utopia method and utopia solution are existing. Therefore, the single-objective optimization problem (12) needs to be solved.

**Table 4.** Optimal coded variable values and predicted values for the objective functions,  $j = 1 \div 3$

Optimal values	$Y_{1min}$	$Y_{2min}$	$Y_{3min}$
$x_1^{jopt}$	-1.414	0.789	0.697
$x_2^{jopt}$	1.414	-1.414	-0.209
$x_3^{jopt}$	-1.414	0.617	1.414
$Y_{jmin}$	14.54	0.82	14.17

The single-objective optimization problem (12) was solved using the Add – in – Solver function in Microsoft Excel 2022. The results of the single-objective optimization are presented in Table 4.

The optimal values for each single-objective optimization problem ( $Y_{1min}$ ,  $Y_{2min}$ ,  $Y_{3min}$ ) are as follows:

The optimal value for energy cost ( $Y_{1min}$ ) is as follows:  $Y_{1min} = 14.54$  kWh/kg with the corresponding technological conditions: Temperature of drying chamber  $x_1^{1opt} = -1.414$  (coded value), which is equivalent to 32.17°C; Pressure of drying chamber  $x_2^{1opt} = 1.414$  (coded value), which is equivalent to 0.044 mmHg; Drying time  $x_3^{1opt} = -1.414$  (coded value), which is equivalent to 31.17 hours.

The optimal value for product moisture ( $Y_{2min}$ ) is as follows:  $Y_{2min} = 0.82\%$  with the

corresponding technological conditions: Temperature of drying chamber  $x_1^{2opt} = 0.79$  (coded value), which is equivalent to 36.58°C; Pressure of drying chamber  $x_2^{2opt} = -1.414$  (coded value), which is equivalent to 0.0156 mmHg; Drying time  $x_3^{2opt} = 0.62$  (coded value), which is equivalent to 35.26 hours.

The optimal value for product crispiness ( $Y_{3min}$ ) is as follows:  $Y_{3min} = 14.17$  mN with the corresponding technological conditions: Temperature of drying chamber  $x_1^{3opt} = 0.70$  (coded value), which is equivalent to 36.4°C; Pressure of drying chamber  $x_2^{3opt} = -0.21$  (coded value), which is equivalent to 0.028 mmHg; Drying time  $x_3^{3opt} = 1.414$  (coded value), which is equivalent to 36.8 hours.

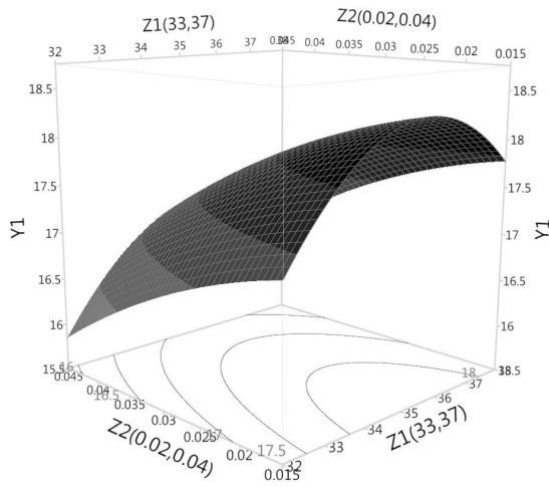
Thus, the single-objective optimization problems do not have a common solution for the entire system ( $x_1^{iopt}, x_2^{iopt}, x_3^{iopt}$ )  $\neq$  ( $x_1^{kopt}, x_2^{kopt}, x_3^{kopt}$ ) with  $i, k = 1 \div 3$  and  $i \neq k$  to simultaneously satisfy the minimum values of all three objectives  $Y_j$  ( $j = 1 \div 3$ ).

Table 4 shows that the single-objective optimization problems (12) do not have common solutions so there are no utopia optimal methods and no utopia solutions existing.

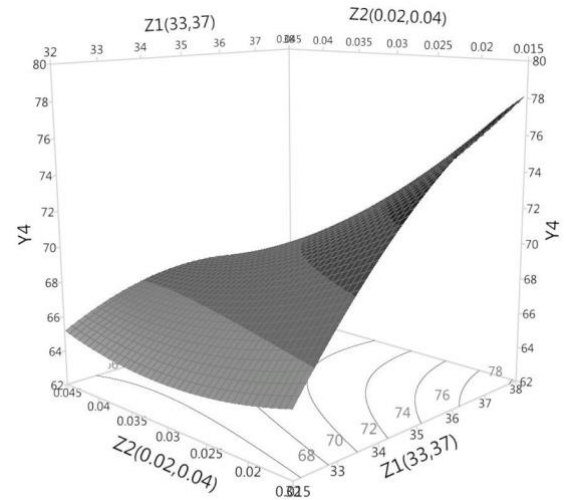
Figure 5 “a), b), c), d)” illustrates the interactive effects of input technological factors on the output objectives including energy consumption, product moisture content, crispiness, and the survival rate of microorganisms in the dried products.

The 3D graphs indicate that energy consumption increased as the drying environment pressure was reduced. Additionally, raising the drying temperature also demanded higher energy input to achieve the desired product moisture content. Therefore, selecting an appropriate combination of pressure and drying temperature plays a crucial role in minimizing the energy requirements for the drying process, thereby contributing to cost reduction in product manufacturing.

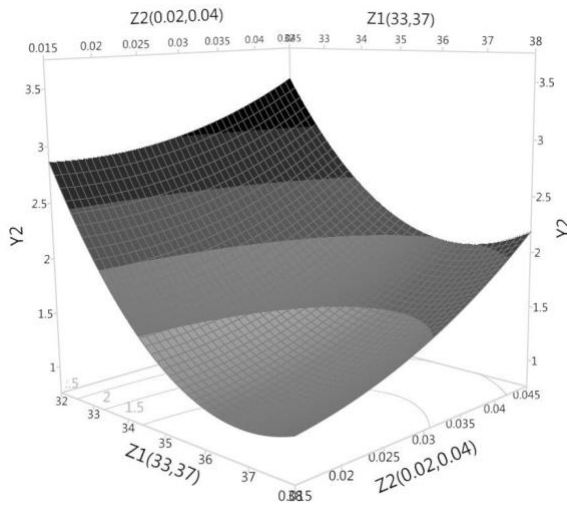




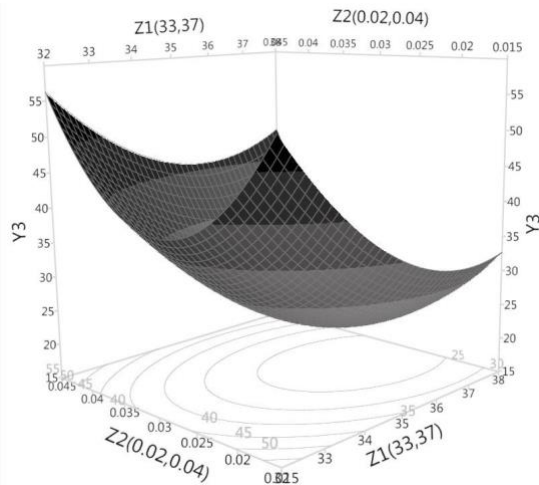
a)  $Y_1 = f_1(Z_1, Z_2, Z_3 = 34.00)$ , regression equation (16)



d)  $Y_4 = f_4(Z_1, Z_2, Z_3 = 34.00)$ , regression equation (19)



b)  $Y_2 = f_2(Z_1, Z_2, Z_3 = 34.00)$ , regression equation (17)



c)  $Y_3 = f_3(Z_1, Z_2, Z_3 = 34.00)$ , regression equation (18)

**Figure 5.** 3D plots presenting the interactive effects of input technological factors on the output objectives.

$Z_1$ : temperature of drying chamber;  $Z_2$ : pressure of drying chamber;  $Z_3$ : drying time

$Y_1$ : energy consumption (kWh/kg);  $Y_2$ : moisture content (%);  $Y_3$ : crispiness (mN);  $Y_4$ : microbial survival rate(%).

### 3.4.2. The multi-objective optimization problem

Because single-objective optimization problems do not share a common solution so the utopian solution does not exist to satisfy all single-objective optimization problems. As a result, the selection of a solution to achieve the optimal value of one objective often leads to the deterioration of the other objectives, which is a common and inherent issue in the field of engineering. The major purpose of this study is to find a compromise solution that satisfies all objectives and meets the technological requirements. Hence, at this point, the research problem has evolved into a multi-objective optimization problem (Dzung N.T, Chuyen H.V, Linh V.T.K, and et al., 2022).

Although utopia optimal solutions do not exist, there is still the presence of a utopia point  $Y^{UT} = (Y_{1min}, Y_{2min}, Y_{3min}) = (14.54, 0.82, 14.17)$ . This serves as the basis for establishing the composite standard  $S(x)$  (14). The multi-objective optimization problem with the composite standard  $S(x)$  (15) was solved using the Add – in – Solver function in Microsoft

Excel 2022. The results of the multi-objective optimization problem are presented in Table 5.

**Table 5.** Coded values, actual values after multi-objective optimization, and predicted results for the optimal sample.

Coded values			Paréto experimental values	
$x_1^{opt}$	$x_2^{opt}$	$x_3^{opt}$	j	$Y_{jmin}^S$
0.8113	-0.0681	0.8034	1	19.935
			2	0.963
			3	15.953
			4	69.291

After conducting the experiments and solving the multi-objective optimization problem using utopian point method, the optimal technological parameters for the production of freeze-dried yogurt were found as follows:

- Temperature of drying chamber  $x_1^{opt} = 0.8113$ , which is equivalent to  $Z_1^{opt} = 36.60^\circ\text{C}$
- Pressure of drying chamber  $x_2^{opt} = -0.0681$ , which is equivalent to  $Z_2^{opt} = 0.023 \text{ mmHg}$
- Time of drying process  $x_3^{opt} = 0.8034$ , which is equivalent to  $Z_3^{opt} = 35.6 \text{ hours}$

The predicted values for the corresponding objective functions are as follows:

- Energy cost  $Y_{1min}^S = 19.935 \text{ kWh/kg}$
- Product moisture content  $Y_{2min}^S = 0.963\%$
- Crispiness  $Y_{3min}^S = 15.953 \text{ mN}$

These optimized parameters and predicted results will contribute to the efficient production of dried yogurt with the desired characteristics.

### 3.5. Validation of the predicted values

To validate whether the optimal technological conditions in Table 5, derived from the multi-objective optimization problem

(15), are suitable for practical use and production, an experiments for freeze drying of yogurt were conducted under the optimal conditions ( $Z_1^{opt} = 36.60^\circ\text{C}$ ;  $Z_2^{opt} = 0.023 \text{ mmHg}$ ;  $Z_3^{opt} = 35.6 \text{ hours}$ ) using a freeze drying system (DS-12). The results of the drying process are presented in Table 6.

**Table 6.** Predicted results, actual results, and the differences.

Outputs	$Y_1$	$Y_2$	$Y_3$
Unit	kWh/kg	%	mN
Experimental results, $Y_j^E$	21.15	0.874	17.23
Predicted results, $Y_{jmin}^S$	19.935	0.963	15.953
Difference (%)	5.75	10.18	7.41

The calculated optimal energy cost during the drying process ( $Y_{1min}^S$ ) was 19.935 kWh/kg, which is 5.75% lower than the actual experimental result of 21.15 kWh/kg. Regarding the product's moisture content ( $Y_{2min}^S$ ), the optimized calculation yielded 0.963%, which is 10.18% higher than the actual experimental result of 0.874%. For the crispiness of the dried yogurt ( $Y_{3min}^S$ ), the optimal calculation resulted in 15.953mN, which is 7.41% lower than the actual experimental result of 17.23mN.



**Figure 6.** Freeze drying yogurt product of the optimal technological conditions.

Based on the experimental verification of the optimal technological conditions, it can be concluded that while there are some deviations between the predicted values in Table 5 and the experimental results, these discrepancies are at the acceptable levels. Therefore, the optimal technological conditions in Table 5 can be applied in practical yogurt freeze drying processes. In fact, these optimized conditions have been successfully implemented in some dried yogurt production companies in Vietnam. Freeze drying yogurt product of the optimal technological conditions can be seen in Figure 6.

### 3.6. Evaluation of product quality

According to the report by Duan et al. (2016), the energy consumption during the freeze-drying process is significant, especially for high-value materials with high moisture content (Gallardo-Rivera C. *et al.*, 2021). Therefore, it can be concluded that improving the freeze-drying method by setting up optimization problems to find the process parameters will enhance heat transfer efficiency, reduce drying time, and consequently lower energy consumption during the freeze-drying process. The experimental results show that the energy consumption of 21.15 kWh/kg is an effective energy cost within the realistic combination condition of  $Y_1$  less than 26.04 kWh/kg. As per the optimal parameters for the freeze-dried yogurt, the crispiness value is 17.23 mN, which is in accordance with the set condition of 15.953 mN. Thus, the crispiness of the freeze-dried yogurt in the optimal sample fully meets the structural requirements. Several reports have shown that the preservation of lactic acid bacteria in yogurt through freeze-drying is highly effective, with survival rates reaching up to 88.23% as reported by Gallardo-Rivera et al. (2021), and up to 87.2% as reported by Lim Y., Hong S., Shin, et al. (2015). In this study, the survival rate of lactic acid bacteria reached 69.29%, which, although not as expected, was achieved without using additional components to protect the bacteria, only using plain yogurt. The results also demonstrate the potential for application and improvement in

preserving live microbial resources through freeze-drying methods.

### 4. Conclusions

This study has successfully addressed the issues regarding the chemical composition analysis of yogurt, established optimization problems, and consequently determined the optimal freeze-drying regime for yogurt. Additionally, a complete and feasible technological process for production has been proposed. The optimal freeze-drying regime was determined to be at an ambient drying temperature of 36.6°C, an environmental pressure of 0.023 mmHg, and a drying time of 35.6 hours.

This optimized drying regime resulted in a dried yogurt product with a moisture content of 0.874%, a crispiness of 17.23 mN, and the capability to retain 69.29% of the initial population of lactic acid bacteria. Furthermore, it has been found to consume 21.15 kWh per 1 kg of the product. These findings demonstrate the successful application of the proposed freeze-drying process for yogurt production. The results of this research provide valuable insights into optimizing the yogurt drying process and offer potential benefits in terms of preserving the product's quality and microbial content.

The established technological process can be a valuable reference for industrial yogurt production. Further studies and improvements in freeze-drying technology may lead to even more efficient and sustainable yogurt production in the future.

### 5. References

- Anandharamakrishnan, C., (2017). Handbook of drying for dairy products. John Wiley & Sons, Ltd, 2017. doi: 10.1002/9781118930526.
- AOAC International, AOAC-927.05 Moisture in Dried Milk. 2000.
- Athanasios, I. Liapis and Roberto Bruttini, (2020). "Freeze drying," in Handbook of industrial drying, 4th ed. CRC press, pp. 309–343, 2020.

- Bamforth, C. W. and Cook, D. J., (2019). "Yoghurt and Other Fermented Milk Products," in *Food, Fermentation, and Micro-organisms*, 2nd Edition. Wiley-Blackwell, pp. 183–186, 2019.
- Bhushani, A. and Anandharamakrishnan, C., (2017). "Freeze Drying," in *Handbook of Drying for Dairy Products*, John Wiley & Sons, pp. 95–121, 2017.
- Dzung, N.T and Phuong, V.D., (2016). "The Multi-objective Optimization by the Restricted Area Method to Determine the Technological Mode of Cold Drying Process of Carrot Product," *Research Journal of Applied Sciences, Engineering and Technology*, vol. 13, no. 1, pp. 64–74, 2016, doi: 10.19026/rjaset.13.2891.
- Dzung, N.T and Hai, D.T.H, (2016). "The Multi-objective Optimization by the Utopian Point Method to Determine the Technological Mode of Infrared Radiation Drying Process of Jackfruit Product in Viet Nam" *Research Journal of Applied Sciences, Engineering and Technology*, vol. 13, no. 1, pp.75-84, 2016.
- Dzung, N. T. (Ed.), Chuyen, H. Van, Linh, V. T. K, Nhiem, L.T., Hoan, P. T., Linh, D.T.K, Quang, N.D., Duyen N.D.M, Tung, P.T., (2022) *Freeze drying*. VNU-HCM Press, pp. 187-192, 2022.
- Duan, X., Yang, X., Ren, G., Pang, Y., Liu, L., and Liu, Y., (2016). "Technical aspects in freeze-drying of foods," *Drying Technology*, vol. 34, no. 11. 2016. doi: 10.1080/07373937.2015.1099545.
- Fellows, P., (2000). *Food Processing Technology*, 2nd ed. Woodhead Publishing Limited and CRC Press LLC, 2000.
- German, J. B., (2014). "The future of yogurt: Scientific and regulatory needs," *American Journal of Clinical Nutrition*, vol. 99, no. 5, pp. 1271–1278, May 2014, doi: 10.3945/AJCN.113.076844.
- Gallardo-Rivera, C. et al., (2021). "Effect of three types of drying on the viability of lactic acid bacteria in foam-mat dried yogurt," *Processes*, vol. 9, no. 12, 2021, doi: 10.3390/pr9122123.
- G.Wilhelm, Oetjen, and Peter Haseley, *Freeze-drying*. Wiley-VCH, 2004.
- Karmi, O., Zayed, A., Baragethi, S., Qadi, M., and Ghanem, R., (2011). "Measurement of Vitamin B 12 concentration: a review on available methods," *IIOAB Journal*, vol. 2, no. 2, pp. 23–32, 2011.
- Lim, Y., Hong, S., Shin, Y. K. Kang, S. H., and Center D., (2015). "Changes in the viability of lactic acid bacteria during storage of freeze-dried yogurt snacks," *Journal of Dairy Science and Biotechnology*, vol. 33, no. 3, p. 203, 2015.
- Mawilai, P., Chaloeichitratham, N., and Pornchaloempong, P., (2019). "Processing feasibility and qualities of freeze dried mango powder for SME scale," in *IOP Conference Series: Earth and Environmental Science*, 2019. doi: 10.1088/1755-1315/301/1/012059.
- Marette, A., Picard-Deland, É., and Fernandez, M., (2017). *Yogurt: roles in nutrition and impacts on health*. 2017.
- Sogi, D. S., Siddiq, M., and Dolan, K. D., (2015). "Total phenolics, carotenoids and antioxidant properties of Tommy Atkin mango cubes as affected by drying techniques," *LWT*, vol. 62, no. 1, 2015, doi: 10.1016/j.lwt.2014.04.015.
- TCVN 7906:2008, "Microorganisms in food and animal feeding stuffs - Quantitative determination of mesophilic lactic acid bacteria - Colony-count technique at 30°C," *TCVN, 7906:2008*, 2008.
- Yildiz, F., (2010). *Development and manufacture of yogurt and other functional dairy products*. CRC Press/Taylor & Francis, 2010.

#### Conflict of interest

The author declares no conflict of interest.

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