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STUDY OF DETERMINING THE TECHNOLOGICAL MODE IN THE FREEZE DRYING PROCESS OF ROYAL JELLY IN VIET NAM

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ABSTRACT

The aim of this study was to find the optimal technological mode for the freeze drying of Royal jelly. By the experimental method, the multi-objective optimization problem to describe the freeze drying process of Royal jelly was built. Anh by the restricted area method, solving the multi-objective optimization problem were found out the technological mode of the freeze drying process of Royal jelly as follow: the optimal temperature of freeze drying chamber was $Z_1^{opt} = 20.58^0C$, the optimal pressure of the freeze drying chamber was $Z_2^{opt} = 0.411$mmHg and the time of the freeze drying of Royal jelly $Z_3^{opt} = 18.283$h. Corresponding to these optimal factors, the energy consumption for 1 kg final product reached the minimum value of $y_{1P}^R = 6.32$kWh/kg, the residual water content in Royal jelly reached the minimum value of $y_{2P}^R = 4.19\%$ under 4.5% (< 4.5%), the loss of protein, carbohydrate, lipid, mineral salts, 10-HDA, vitamin B_5, free fatty acids and viscosity reached the minimum value of $y_{3P}^R = 1.77\%$, $y_{4P}^R = 1.83\%$, $y_{5P}^R = 2.07\%$, $y_{6P}^R = 2.36\%$, $y_{7P}^R = 0.78\%$, $y_{8P}^R = 2.47\%$, $y_{9P}^R = 2.67\%$, $y_{10P}^R = 3.58\%$.

1. Introduction

It is common knowledge that Royal jelly is rare natural product, it contains a lot of important nutritional substances for human's health such as protein, lipid, carbohydrate, mineral salts, free fatty acids, free amino acids, vitamins and enzymes. In addition, Royal jelly contains bioactive compounds that have extremely good effect on human's health such as 10-HDA (Anna et al., 2009). Protein components inside Royal jelly contain hardly all essential amino acids for humans, including 29 amino acids and derivatives. Besides, protein inside of Royal jelly also contains some enzymes such as protease, glucose oxidase, phosphatase, cholinesterase and an insulin-like substance (Antinelli et al., 2003), these enzymes is essential for biochemical processes in human's body. Carbohydrate components inside Royal jelly are mainly fructose, glucose and sucrose. But ratio of fructose in carbohydrate components is higher than ratio of glucose and sucrose. In addition, other sugars present in much lower quantities are maltose, trehalose, melibiose, ribose and erlose (Lercker et al., 1992; Gaëlle and Hervé, 2012), they are types of bioactive carbohydrate that have very good effect on human. Lipid components inside Royal jelly has low rate, but it contains bioactive compounds rare in the nature, free fatty acids and fatty acids inside lipid components have unusual and uncommon structures, they are mostly short carbon chain (from 8 to 10 carbon atoms) of hydroxyl fatty acids or dicarboxylic acids. These free fatty acids are most of the recorded biological properties of Royal jelly (Schmidt et al., 1992) such as 10-HDA (10-hydroxy-2-decenoic acid) and its isomer. Besides, the lipid inside Royal jelly contains some neutral lipids including: sterols (cholesterol, esgosterol) and an unsaponifiable fraction of hydrocarbons similar to beeswax extracts (Lercker et al., 1992; Hattori et al., 2007). In addition, Royal jelly contains a lot of mineral salts, the total ash in Royal jelly has low rate about 2 to 3 % of dry weight. The major mineral salts are, in descending order: K, Ca, Na, Zn, Fe, Cu and Mn, with a strong prevalence of potassium (Andreas Stocker et al., 2005). Besides, Royal jelly is extremely rich in water-soluble vitamins and fat-soluble vitamins (Schmidt and Buchmann, 1986). For the water-soluble vitamins include B_1, B_2, B_3 (PP), B_5, B_6, B_c and H. For the fat-soluble vitamins include A, D, E, K (Benfenati et al., 1992). Therefore, it can be seen that Royal jelly supply complete mineral salts and vitamins for humans (Lercker et al., 1992, Benfenati et al., 1992).

Figure 1. The Royal jelly product in Viet Nam

According to the results of Mohamed, F. R., et al. (2012), it shown that the most important nutritional compounds of Royal jelly including protein, carbohydrate and lipid have balanced rate between essential amino acids with different amino acids, between fructose, glucose with different sugars, between unsaturated fatty acids with saturated fatty acids that are very rare in nature. This nature product is very good and very perfect for growing up and development processing of human’s health. According to Antinelli, J. F., et al. (2003); Anna G.S. (2009); Isidorov et al., (2011). Royal jelly was also called as a natural pharmaceutical product, it has the ability of anti-aging; prevent the formation of free radicals from biochemical reactions in human’s body. It can help prevent the heart disease or heart disorder, psychophysiological disorder, nervous disorder, digestive disorder, cancer and many other diseases. It has the capable of restoring and protecting human’s skin as well as increase energy and restores human’s health. In addition, it can cure women of gynaecological disease. According to analytical data of Dzung (2013), the important chemical composition of Royal jelly in Viet Nam contains many different substances but their main constituents are water, protein, carbohydrate (sugars), lipids and mineral salts, impurity, 10-HDA, vitamin B_5 and free fatty acids. It can be seen in Table 1.

From Table 1, it is obvious that Royal jelly is rich in nutritional components. It is an advantageous environment in order that microorganism grows up and develops. If Royal jelly is not preserved, it will be easily decomposed or hydrolyzed and oxidized, it will be no longer value of use (Isidorov et al., 2011; Hiroyuki M. et al., 2012). Therefore, Royal jelly needs to be preserved in suitable environment in order to be prolonged use. Currently, there are two methods that often use to preserve Royal jelly; those are the freezing method and the freeze drying method. The final products have always a very good quality when they are made by these two technologies (Mohamed, F. R., et al., 2012). But the freezing method, Royal jelly after freezing process must be preserved in environment that has suitable temperature about -20^0C to -18^0C and the range of this temperature must be maintained during use time and export time. As a result, it makes to increase the expenditure of preservation process of Royal jelly. For the freeze drying method are used the most popular. The Royal jelly after the freeze drying placed in nylon bags, vacumn seaming is preserved in usual environment of 25^0C. Therefore, it will be not lost the expenditure for preservation process (Dzung et al., 2014; 2015).

Table 1. The chemical composition of Royal jelly in Viet Nam

No	**Substance**	**The ratio of initial material weight**	**The ratio of dry weight**
1	Water	59.2%	-
2	Proteins	14.26%	34.95%
3	Carbohydrate (sugars)	15.95%	39.09%
4	Lipids	4.00%	9.80%
5	Minerals	1.10%	2.70%
6	Impurity (wax, …)	2.39%	5.86%
7	10-HDA	3.1 %	7.60 %
8	Vitamin B_5	4.05 mg/100g	9.93 mg/100g
9	Free fatty acids	558.95 mg/100g	1370 mg/100g
Thermophysical parameter of Royal jelly in Viet Nam			
10	Viscosity at 25^0C	11817.9 cP	-

However, the problem posed here is how to determine the technological mode for the freeze drying process of Royal jelly in order that Royal jelly after freeze drying have the best quality, the residual water content of Royal jelly is under 4.5% (Dzung et al., 2015), the energy consumption of 1 kg final product reaches the minimum value. This is a question that had not any research to mention for a long time ago. To answer this problem, in this study the multi-objective optimization problem describing about the relationship between objective functions, including: y_1 (kWh/kg) – the energy consumption of 1 kg final product of Royal jelly after freeze drying; y_2 (%) – the residual water content of Royal jelly after freeze drying; y_3 (%) – the loss of total protein; y_4 (%) – the loss of carbohydrate; y_5 (%) – the loss of lipid; y_6 (%) – the loss of mineral salts; y_7 (%) – the loss of 10-HDA; y_8 (%) – the loss of vitamin B_5; y_9 (%) – the loss of free fatty acids and y_{10} (%) – the loss of viscosity of Royal jelly after freeze drying; with technological factors, including: Z_1 (^0C) – the temperature of freeze drying chamber, Z_2 (mmHg) – the pressure of freeze drying chamber; Z_3 (h) – the time of freeze drying process is built by experimental method. The multi-objective optimization problem is expressed as follow: Finding $Z^{opt} = \{Z_1^{opt}, Z_2^{opt}, Z_3^{opt}\} \in \Omega_Z$ in order that $y_{jmin} = f(Z_1^{opt}, Z_2^{opt}, Z_3^{opt}) = \min\{f_j(Z_1, Z_2, Z_3)\}$. After that by the restricted area method, solving this multi-objective optimization problem is determined the technological mode for the freezing process of Royal jelly, (Dzung, 2012).

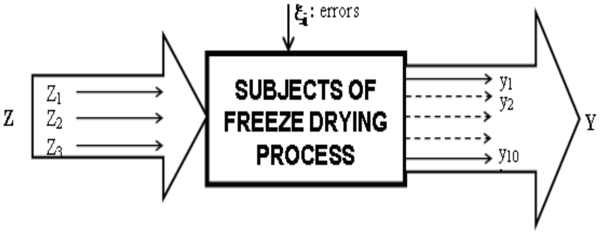

Figure 2. Diagram of subjects of freezing process

2. Materials and methods

2.1. Materials

The Royal jelly is harvested from bees's nest to grow up at Bao Loc area in Lam Dong province of Viet Nam. It is the pure natural product and does not mix any chemical composition. It is very thick solution, has pale yellow and sour. The basic composition of Royal jelly is presented in Table 1, (Dzung, 2013; 2014; 2015).

The Royal jelly is poured into trays, they are made by glass. According to results of Dzung (2015), it shown that Royal jelly layer in glass trays at optimal technological mode of the freezing process of Royal jelly have depth of 12.93mm.

Before carrying out the freeze drying process of Royal jelly, Royal jelly is frozen at the optimal technological mode: the temperature of freezing environment was -

40.46^0C; the time of freezing process was 1.63h; the thickness of Royal jelly in the tray was 12.93mm. Corresponding to these technological parameters, the freezing temperature of Royal jelly was -18.33^0C and water inside Royal jelly was completely crystallized $\omega = 1$ or $\omega = 100\%$, (Dzung, 2014; 2015).

2.2. Apparatus

Equipments used to research the technological mode for the freeze drying process of Royal jelly are listed (Dzung, 2012a & b, 2013; 2014; 2015):

- Equipments used to determine weigh of Royal jelly by Satoriusbasic Type BA310S: range scale (0 ÷ 350)g, error: ± 0.1g = ± 0.0001 kg.
- The Freeze Drying System DS-3 (Fig 3a) and The Refrigeration System DL-3 (Fig 3b) that were controlled automatically by computer. It could reduce the temperature of environment to $(-50 \div -45)^0$C. The temperature, pressure and time profile of freeze drying process are measured by computer.

Figure 3a. The freeze drying system DS-3 with the auto-freezing $(-50 \div -45)^0$C

- Kjeldahl, Soxhlet, GC-MS, HPLC, Viscometer and other equipment were used to analyse and determine protein, carbohydrate, lipid, ash, 10-HDA, free fatty acids, vitamins and viscosity of Royal jelly during freeze drying process.

Figure 3b. The Refrigeration system DL-3 with the auto freezing $(-45 \div -40)^0$C

2.3. Methods

Using in this study to include some method as follow (Dzung, 2012; 2013; 2014; 2015):

- Determining the temperature (Z_1, ^{0}C), pressure (Z_2, mmHg) of freeze drying chamber and the time of freezing process (Z_3, h) of Royal jelly by the automatic measure and control system on computer of the Freeze Drying System DS-3.

- Determining the energy consumption (y_1, kWh/kg final product) of 1 kg Royal jelly after freeze drying process by the equation (1), (Holman J. , 1992; Figura et al., 2007).

$$y_1 = \frac{P.\tau}{G} = \frac{U.I.\tau.\cos\varphi}{G}\text{, kWh/kg} \qquad (1)$$

Where: G (kg) – weight of the final product; U (V) – number of Voltmeter; I (A) – number of Amperemeter; τ (s) – second; cosφ – power factor.

- Determining the residual water content of the final product of Royal jelly after freeze drying (y_2, %) by the mass sensor controlled by computer, (Dzung, 2012a & b).

$$y_2 = 100 - \frac{G_0}{G_e}\left(100 - W_0\right) \qquad (2)$$

Where: G_0 (kg) – weight of the initial material of Royal jelly used for freeze drying; G_e (kg) – weight of the final product of Royal jelly after freeze drying; W_0 (%) – the residual water content of the initial material of Royal jelly.

- Determining the total protein inside Royal jelly before and after freeze drying by the Kjeldahl method (FAO, 14/7, 1986).
- Determining the total carbohydrate inside Royal jelly before and after freeze drying by the hight - performance anion - exchange chromatography method (TCVN 4594:1988).
- Determining the lipid inside Royal jelly before and after freeze drying by the Soxhlet method (FAO, 14/7, 1986).
- Determining ash inside Royal jelly before and after freeze drying by the AOAC 2000 (971.14) method (FAO, 14/7, 1986) as follow: Royal jelly samples were burned into ash by heat. Then, ash was determined by weight method.
- Determining the 10-HDA inside Royal jelly before and after freeze drying by the Gas Chromatography method (GC-ISO/CD 5509:94).
- Determining vitamin B_5 inside Royal jelly before and after freeze drying by the HPLC/UV (High performance liquid chromatography/UV-vis) method (TK. AOAC 985.33).
- Determining free fatty acids inside Royal jelly before and after freeze drying by the AOCS (1997) method (TCVN 6127-2010).
- Determining viscosity of Royal jelly before and after freeze drying by viscometer of Brookfield. Royal jelly after freeze drying is soaked into water in order to determine viscosity, the weight of soaked water is equal weight of separate water from freeze drying process.
- Determining the loss of total protein; the loss of carbohydrate; the loss of lipid; the loss of mineral salts; the loss of 10-HDA; the loss of vitamin B_5; the loss of free fatty acids and the loss of viscosity of the final product (y_j, %) by equation (2), with j = 3 ÷ 10.

$$y_j = \frac{m_1 - m_2}{m_1}100\% = \frac{\Delta m}{m_1}100\%; \quad j = 3 \div 10 \qquad (3)$$

Where: the total protein, carbohydrate, lipid, mineral salts, 10-HDA, free fatty acids, free amino acids, vitamin and and viscosity of the material initial and after freeze drying respectively m_1 (%) and m_2 (%) were calculated according to weight of dry matter. If it is viscosity, unit of m_1 and m_2 will be cP (mPa.S = 10^{-3} Pa.S). In the fact that the final product of Royal jelly after freeze drying achieves the best quality means $y_{jmin} = 0$. In the fact, $y_j > 0$.

- Using quadratic orthogonal experimental planning method (Dzung, 2012a; 2015) to build the mathematical model about relationships between y_j (j = 1÷ 10) and technological factors effect on the freezing process (Z_1, Z_2, Z_3). These mathematical models of y_j (j = 1÷ 10) were written as follow (Dzung, 2012a; 2015):

$$y_j = b_0 + \sum_{u=1}^{k} b_u x_u + \sum_{u \neq i; u=1}^{k} b_{ui} x_u x_i + \sum_{u=1}^{k} b_{uu}\left(x_u^2 - \lambda\right) \qquad (4)$$

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

$$x_i = (Z_i - Z_i^0)/\Delta Z_i; \; Z_i = x_i.\Delta Z_i + Z_i^0 \qquad (5)$$

Where: $Z_i^0 = (Z_i^{max} + Z_i^{min})/2$;

$$\Delta Z_i = (Z_i^{max} - Z_i^{min})/2; \qquad (6)$$

$$Z_i^{min} \le Z_i \le Z_i^{max};\ i = 1 \text{ to } 3$$

The experimental number is determined:

$$N = n_k + n_* + n_0 = 2^k + 2k + n_0 = 18 \quad (7)$$

With: $k = 3$; $n_k = 2^k = 2^3 = 8$;

$n_* = 2k = 2x3 = 6$; $n_0 = 4$

The value of the star point:

$$\alpha = \sqrt{\sqrt{N.2^{(k-2)}} - 2^{(k-1)}} = 1.414 \quad (8)$$

The condition of the orthogonal matrix:

$$\lambda = \frac{1}{N}\left(2^k + 2\alpha^2\right) = 2/3 \quad (9)$$

- Using the mathematical tools to solve the multi-objective optimization problem to determine the technological mode for the freeze drying process of Royal jelly.

3. Results and discussions

3.1. Develop the mathematical models of the freeze drying process of Royal jelly

The constituent objective functions of the freeze drying process including: y_1 (kWh/kg) – the energy consumption of 1 kg final product of Royal jelly after freeze drying; y_2 (%) – the residual water content of Royal jelly after freeze drying; y_3 (%) – the loss of total protein; y_4 (%) – the loss of carbohydrate; y_5 (%) – the loss of lipid; y_6 (%) – the loss of mineral salts; y_7 (%) – the loss of 10-HDA; y_8 (%) – the loss of vitamin B_5; y_9 (%) – the loss of free fatty acids and y_{10} (%) – the loss of viscosity of final product of Royal jelly of freeze drying process depended on the technological factors, including: temperature of freeze drying chamber (Z_1, ^{0}C), pressure of freeze drying chamber (Z_2, mmHg), time of freeze drying (Z_3, h).

Therefore, these constituent objective functions were determined by the experimental planning method with the quadratic orthogonal experimental matrix ($k = 3$, $n_0 = 4$). In addition, the experimental factors were established by conditions of the technological freeze drying (Dzung, 2012a; 2013; 2015), they were summarized in Table 2.

Table 2. The technological factors levels design

Parameters		Z_1, (^{0}C)	Z_2, (mmHg)	Z_3, (h)
Le-vels	- α (-1.414)	17.93	0.008	17.172
	Low (-1)	20	0.137	18
	Central (0)	25	0.447	20
	High (+1)	30	0.758	22
	+ α (1.414)	32.07	0.886	22.828
Deviation ΔZ_i		5	0.3105	2

The experiments were carried out with all of the factor levels in Table 2 to determine the value of the objective functions that describe relationships between the energy consumption of 1 kg final product of freeze drying Royal jelly; the residual water content of final product of Royal jelly after freeze drying; the loss of total protein; the loss of carbohydrate; the loss of lipid; the loss of mineral salts; the loss of 10-HDA; the loss of free fatty acids; the loss of vitamin and the loss of viscosity of the final product of Royal jelly after freeze drying and technological factors, (Dzung, 2012a; 2015). The results were summarized in Table 3a, 3b, 3c and 3d.

Table 3a. The orthogonal experimental matrix level 2 (k = 3, n_0 = 4)

N		x_0	x_1	x_2	x_3
2^k	1	1	1	1	1
	2	1	-1	1	1
	3	1	1	-1	1
	4	1	-1	-1	1
	5	1	1	1	-1
	6	1	-1	1	-1
	7	1	1	-1	-1
	8	1	-1	-1	-1
2k	9	1	1.414	0	0
	10	1	-1.414	0	0
	11	1	0	1.414	0
	12	1	0	-1.414	0
	13	1	0	0	1.414
	14	1	0	0	-1.414
n_0	15	1	0	0	0
	16	1	0	0	0
	17	1	0	0	0
	18	1	0	0	0

Table 3b. The orthogonal experimental matrix level 2 (k = 3, n_0 = 4)

x_1x_2	x_1x_3	x_2x_3	$x_1^2 - \lambda$	$x_2^2 - \lambda$	$x_3^2 - \lambda$	y_1
1	1	1	0.333	0.333	0.333	7.85
-1	-1	1	0.333	0.333	0.333	7.62
-1	1	-1	0.333	0.333	0.333	8.18
1	-1	-1	0.333	0.333	0.333	8.02
1	-1	-1	0.333	0.333	0.333	6.41
-1	1	-1	0.333	0.333	0.333	6.36
-1	-1	1	0.333	0.333	0.333	6.37
1	1	1	0.333	0.333	0.333	6.29
0	0	0	1.333	-0.667	-0.667	7.18
0	0	0	1.333	-0.667	-0.667	6.78
0	0	0	-0.667	1.333	-0.667	6.70
0	0	0	-0.667	1.333	-0.667	7.68
0	0	0	-0.667	-0.667	1.333	9.20
0	0	0	-0.667	-0.667	1.333	5.98
0	0	0	-0.667	-0.667	-0.667	7.00
0	0	0	-0.667	-0.667	-0.667	7.05
0	0	0	-0.667	-0.667	-0.667	7.22
0	0	0	-0.667	-0.667	-0.667	7.06

Table 3d. The orthogonal experimental matrix level 2 (k = 3, n_0 = 4)

N		y_2	y_3	y_4	y_5
2^k	1	2.94	4.03	3.25	3.06
	2	3.58	3.89	2.92	3.47
	3	2.59	4.12	2.66	2.96
	4	3.49	3.72	2.12	2.65
	5	4.11	2.83	2.58	2.45
	6	4.45	2.63	1.97	2.24
	7	4.19	2.98	2.35	2.14
	8	4.51	1.69	1.97	2.04
2k	9	3.08	3.20	3.02	3.16
	10	3.83	2.32	2.17	2.96
	11	3.80	3.00	2.84	2.65
	12	3.02	2.63	2.05	2.55
	13	2.57	4.23	2.84	3.27
	14	4.73	1.32	2.07	2.14
n_0	15	3.34	2.09	1.87	1.94
	16	3.18	2.32	2.00	1.84
	17	3.25	2.23	1.82	1.63
	18	3.17	2.52	1.94	1.73

Table 3c. The orthogonal experimental matrix level 2 (k = 3, n_0 = 4)

y_6	y_7	y_8	y_9	y_{10}
2.26	2.89	4.13	4.01	18.13
2.11	1.71	3.32	3.94	5.19
3.70	2.50	3.93	3.72	9.89
3.41	1.58	3.22	2.92	8.65
2.96	2.24	3.42	3.21	10.26
2.52	0.79	2.82	2.99	5.15
3.26	1.05	2.48	2.85	2.84
2.78	0.92	2.21	2.70	5.69
2.30	2.11	4.93	4.74	13.27
2.00	1.32	2.72	2.48	6.18
1.96	1.71	2.11	3.14	13.14
3.67	1.84	2.01	2.63	9.97
3.22	1.97	4.63	4.16	16.19
2.48	1.45	2.82	1.82	6.76
2.41	1.05	2.42	3.65	3.54
2.33	0.79	2.52	3.80	1.36
2.41	0.92	2.62	3.50	1.57
2.52	1.18	2.32	3.43	3.50

The mathematical model of regression equations (y_j, j = 1 to 10) from Eq. (10) to Eq. (19) were obtained after processing the experimental data, calculating the coefficients, testing the significance of the coefficients by the Student criterion, and testing the regression equations for the fitness of the experimental results by Fisher criterion (Dzung, 2012a, Dzung, 2013; Dzung, 2015).

Results received were the mathematical models as follow:

- *The energy consumption of 1 kg final product of Royal jelly after freezing process*:

$$y_1 = f_1(x_1, x_2, x_3) = 7.125 + 0.091x_1 - 0.166x_2 + 0.899x_3 - 0.104x_2x_3 - 0.123x_1^2 + 0.18x_3^2 \tag{10}$$

- *The residual water content of final product of Royal jelly after freezing process*:

$$y_2 = f_2(x_1, x_2, x_3) = 3.216 - 0.271x_1 + 0.116x_2 - 0.643x_3 - 0.111x_1x_3 + 0.139x_1^2 + 0.118x_2^2 + 0.237x_3^2 \tag{11}$$

- *The loss of total protein of final product of Royal jelly after freezing process*:

$$y_3 = f_3(x_1, x_2, x_3) = 2.245 + 0.274x_1 + 0.814x_3 + 0.303x_1^2 + 0.332x_2^2 + 0.311x_3^2 \tag{12}$$

- *The loss of carbohydrate of final product of Royal jelly after freezing process*:

$$y_4 = f_4(x_1, x_2, x_3) = 1.978 + 0.225x_1 + 0.228x_2 + 0.263x_3 + 0.144x_2x_3 + 0.239x_1^2 + 0.162x_2^2 + 0.169x_3^2 \tag{13}$$

- *The loss of lipid of final product of Royal jelly after freezing process*:

$$y_5 = f_5(x_1, x_2, x_3) = 1.934 + 0.131x_2 + 0.404x_3 + 0.417x_1^2 + 0.186x_2^2 + 0.237x_3^2 \tag{14}$$

- *The loss of mineral salts of final product of Royal jelly after freezing process*:

$$y_6 = f_6(x_1, x_2, x_3) = 2.328 + 0.149x_1 - 0.475x_2 + 0.084x_3 - 0.273x_2x_3 + 0.257x_2^2 + 0.276x_3^2 \tag{15}$$

- *The loss of 10-HDA of final product of Royal jelly after freezing process*:

$$y_7 = f_7(x_1, x_2, x_3) = 1.075 + 0.401x_1 + 0.369x_3 + 0.198x_1x_2 + 0.231x_1^2 + 0.262x_2^2 + 0.231x_3^2 \tag{16}$$

- *The loss of vitamin B_5 of final product of Royal jelly after freezing process*:

$$y_8 = f_2(x_1, x_2, x_3) = 2.556 + 0.46x_1 + 0.166x_2 + 0.519x_3 - 0.156x_2x_3 + 0.552x_1^2 - 0.335x_2^2 + 0.501x_3^2 \tag{17}$$

- *The loss of free fatty acids of final product of Royal jelly after freezing process*:

$$y_9 = f_{10}(x_1, x_2, x_3) = 3.606 + 0.37x_1 + 0.224x_2 + 0.512x_3 - 0.244x_2^2 - 0.189x_3^2 \tag{18}$$

- *The loss of viscosity of final product of Royal jelly after freezing process*:

$$y_{10} = f_{11}(x_1, x_2, x_3) = 4.896 + 2.205x_1 + 1.346x_2 + 2.605x_3 + 2.457x_1x_2 + 1.488x_1x_3 + 2.235x_2^2 + 2.194x_3^2 \tag{19}$$

3.2. Building one-objective optimization problems for the freeze drying process of Royal jelly

It was obvious that all objective functions (y_j, j = 1 to 10) for the freeze drying process of Royal jelly depended on the technological parameters (x_i, i = 1 to 3). If every objective function was individually surveyed, these one-objective functions along with the technological parameters would constitute the one-objective optimization problems. Because all the one-objective functions were to find the minimal value, the one-objective optimization problems were restated as follow (Dzung, 2012a, 2013 and 2015): Finding in common the test $x^{jopt} = (x_1^{jopt}, x_2^{jopt}, x_3^{jopt}) \in \Omega_x = \{-1.414 \le x_1, x_2, x_3 \le 1.414\}$ in order that:

$$\begin{cases} y_j = f_{j\min}\left(x_1^{jopt}, x_2^{jopt}, x_3^{jopt}\right) \\ \qquad\qquad = \min f_j\left(x_1, x_2, x_3\right) \\ j = 1 \div 10;\ \forall x \in \Omega_x = \\ \quad = \left\{-1.414 \le x_1, x_2, x_3 \le 1.414\right\}; \end{cases} \tag{20}$$

3.3. Building multi-objective optimization problems for freeze drying process of Royal jelly

The establishment of the technological mode during freeze drying process of Royal jelly was based on factors including: economic, technicality and quality of the final product, (Dzung, 2012a). The final product of Royal jelly after freeze drying has the best quality, the energy consumption of 1 kg final product reached the minimum value and the residual water content of the final product reached the minimum value but under 4.5%. In addition, the final product of Royal jelly after freeze drying must satisfy all conditions of economic and technical criterion as well as quality criterion for technology of the freeze drying (Dzung, 2012a). For example:

- *$y_1 < C_1 = 6.5$ kWh/kg, if the energy consumption of 1 kg product is over 6kWh ($y_1 > C_1 = 6.5$), it will increase the product price and difficult commercialization.*
- *$y_2 < C_2 = 4.5\%$, if the residual water content of the final product is over 4.5% ($y_2 > C_2 = 4.5\%$), the microorganisms will be capable to grow, develop and damage products.*
- *$y_j < C_j = 3.0\%$ with $j = 3 \div 9$, if the loss of total protein; the loss of carbohydrate; the loss of lipid; the loss of mineral salts; the loss of 10-HDA; the loss of free fatty acids and the loss of vitamin are over 3.0% ($y_j > C_j = 3.0\%$; $j = 3 \div 9$), the final product of Royal jelly after the freeze drying will reduce quality.*
- *$y_{10} < C_{10} = 5.0\%$, if the loss of viscosity of the final product is over 5.0% ($y_{10} > C_{10} = 5.0\%$), the protein inside Royal jelly after freeze drying will be denatured, not be able to recover the original its quality. It make to reduce ability to link between water and protein, this is cause to reduce viscosity of Royal jelly after freeze drying and reverted to the original state. Finally, it makes to reduce quality of final product.*

It is obvious that the multi-objective optimization problem during the freeze drying of Royal jelly appeared in this case. The technological parameters (x_1, x_2 and x_3) of the freeze drying process of Royal jelly have simultaneously influenced on eleven objective functions (y_j, $j = 1 \div 10$) with the identified domain $\Omega_x = \{-1.414 \le x_1, x_2, x_3 \le 1.414\}$. Therefore, the mathematical model of eleven-objective optimization problem to determine the technological mode of the freeze drying process of Royal jelly was restated as follow: Finding in common the root $x = (x_1^{opt}, x_2^{opt}, x_3^{opt}) \in \Omega_x = \{-1.414 \le x_1, x_2, x_3 \le 1.414\}$ in order that (Dzung, 2012a & 2015):

$$\begin{cases} y_j = f_{j\min}\left(x_1^{opt}, x_2^{opt}, x_3^{opt}\right) \\ \qquad\qquad = \min f_j\left(x_1, x_2, x_3\right) \\ \forall x \in \Omega_x = \\ \quad = \left\{-1.414 \le x_1, x_2, x_3 \le 1.414\right\} \\ y_j < C_j; \quad j = 1 \div 10 \end{cases} \tag{21}$$

3.4. Solving one-objective optimization problems for freeze drying process of Royal jelly

According to the results of Dzung et al (2012a; 2015), if all the one-objective optimization problems (20) have the same roots: $(x_1^{jopt}, x_2^{jopt}, x_3^{jopt}) = (x_1^{kopt}, x_2^{kopt}, x_3^{kopt})$ with $k \neq j$, these roots called are utopian roots and also roots of multi-objective optimization problem (21). The optimal plan of utopian roots called is utopian plan. If the utopian roots and the utopian plan do not exist, multi-objective optimization problem (21) will be solved to find the optimal Pareto roots and the optimal Pareto plan.

Therefore, solving one-objective optimization problems (20) were found to achieve: $y_{jmin} = \min f_j(x_1, x_2, x_3)$, $j = 1 \div 10$, with the identified domain $\Omega_x = \{-1.414 \le x_1, x_2, x_3 \le 1.414\}$. By using the meshing method programmed in Matlab R2008a software, the results of the optimal parameters of every objective function from (10) to (19) limited in the experimental domain were summarized in Table 4, (Dzung, 2012a; 2015):

Table 4. Minimum roots of each one-objective optimization problems

j	y_{jmin}	$x_1^{j\ opt}$	$x_2^{j\ opt}$	$x_3^{j\ opt}$
1	5.812	-1.414	1.414	-1.414
2	2.425	1.414	-0.492	1.414
3	1.651	-0.452	0.000	-1.309
4	1.782	-0.533	-0.441	-0.590
5	1.739	0.000	-0.352	-0.852
6	1.863	-1.414	1.182	0.447
7	0.720	-1.036	0.391	-0.799
8	1.740	-0.482	-1.000	-0.564
9	1.738	-1.307	-1.150	-1.112
10	1.243	-1.414	0.476	-0.114

In the Table 4, it is obvious that the utopian point was indentified: $f^{UT} = (f_{1min}, f_{2min}, f_{3min}, f_{4min}, f_{5min}, f_{6min}, f_{7min}, f_{8min}, f_{9min}, f_{10min})$ = (5.812, 2.425, 1.651, 1.782, 1.739, 1.863, 0.720, 1.740, 1.738, 1.243). However, the utopian root and the utopian plan did not exist, because of $x^{jopt} = (x_1^{jopt}, x_2^{jopt}, x_3^{jopt}) \neq x^{kopt} = (x_1^{kopt}, x_2^{kopt}, x_3^{kopt})$ with $j, k = 1 \div 10$, $j \neq k$ (Dzung, 2011, 2012a, 2012c & 2015).

From results of solving one-objective optimization problems (21), it was obvious that the utopian root and utopian plan do not exist. Therefore, multi-objective optimization problems (21) need to have to be solved to find the optimal Pareto root and the optimal Pareto plan in order that optimal Pareto effect $y_P^R = (y_{1P}^R, y_{2P}^R, y_{3P}^R, y_{4P}^R, y_{5P}^R, y_{6P}^R, y_{7P}^R, y_{8P}^R, y_{9P}^R, y_{10P}^R)$ closest to the utopian point f^{UT} and the furthest from the restricted area $\{y_j \ge C_j\}$.

3.5. Solving multi-objective optimization problems for freeze drying process of Royal jelly

The purpose of the experiment was to reach the targets of the freeze drying process which were expressed by 10 regression equations from (10) to (19), but the tests satisfying all function values ($y_{1min}, y_{2min}, y_{3min}, y_{4min}, y_{5min}, y_{6min}, y_{7min}, y_{8min}, y_{9min}, y_{10min}$) could not be found. Hence, the idea of the multi-objective optimization problem (22) was to find the optimal Pareto root for $y_P^R = (y_{1P}^R, y_{2P}^R, y_{3P}^R, y_{4P}^R, y_{5P}^R, y_{6P}^R, y_{7P}^R, y_{8P}^R, y_{9P}^R, y_{10P}^R)$ closest to the utopian point and the furthest from the restricted area, but $y_j = y_j(x) = f_j(x_1, x_2, x_3)$, with $j = 1 \div 10$ must satisfy technological conditions with requirements in $\{y_j < C_j\}$, (Dzung, 2011, 2012a & 2015).

By the restricted area method, solving the multi-objective optimization problem of the freeze drying (21) as the followings: Establishing the R*-objective combination function $R^*(y_1, y_2, y_3, y_4, y_5, y_6, y_7, y_8, y_9, y_{10}) = R^*(x_1, x_2, x_3) = R^*(x)$ as follow, (Dzung, 2012a, 2012c & 2015):

$$\begin{cases} R^*(x) = R^*(x_1, x_2, x_3) \\ \quad = \sqrt[10]{\prod_{j=1}^{10} r_j(x_1, x_2, x_3)} = \sqrt[10]{\prod_{j=1}^{10} r_j(x)} \\ \Omega_x = \{-1.414 \le x_1,\ x_2,\ x_3 \le 1.414\}; \\ x = (x_1, x_2, x_3) \end{cases} \quad (22)$$

Where: $r_j(x) = \left(\dfrac{C_j - y_j(x)}{C_j - y_{jmin}} \right)$

$$\text{when} \quad y_j(x) < C_j \quad (23)$$

$$r_j(x) = 0 \ \text{when} \ \ y_j(x) \ge C_j \quad (24)$$

$r_1(x) = (6.5 - y_1(x))/(6.5 - 5.812)$

when $y_1(x) < 6.5$

$r_1(x) = 0$ when $y_1(x) \ge 6.5$

$r_2(x) = (4.5 - y_2(x))/(4.5 - 2.425)$

when $y_2(x) < 4.5$

$r_2(x) = 0$ when $y_2(x) \ge 4.5$

$r_3(x) = (3.0 - y_3(x))/(3.0 - 1.651)$

when $y_3(x) < 3.0$

$r_3(x) = 0$ when $y_3(x) \geq 3.0$

$r_4(x) = (3.0 - y_4(x))/(3.0 - 1.782)$

when $y_4(x) < 3.0$

$r_4(x) = 0$ when $y_4(x) \geq 3.0$

$r_5(x) = (3.0 - y_5(x))/(3.0 - 1.739)$

when $y_5(x) < 3.0$

$r_5(x) = 0$ when $y_5(x) \geq 3.0$

$r_6(x) = (3.0 - y_6(x))/(3.0 - 1.863)$

when $y_6(x) < 3.0$

$r_6(x) = 0$ when $y_6(x) \geq 3.0$

$r_7(x) = (3.0 - y_7(x))/(3.0 - 0.720)$

when $y_7(x) < 3.0$

$r_7(x) = 0$ when $y_7(x) \geq 3.0$

$r_8(x) = (3.0 - y_8(x))/(3.0 - 1.740)$

when $y_8(x) < 3.0$

$r_8(x) = 0$ when $y_8(x) \geq 3.0$

$r_9(x) = (3.0 - y_9(x))/(3.0 - 1.738)$

when $y_9(x) < 3.0$

$r_9(x) = 0$ when $y_9(x) \geq 3.0$

$r_{10}(x) = (5.0 - y_{10}(x))/(5.0 - 1.243)$

when $y_{10}(x) < 5.0$

$r_{10}(x) = 0$ when $y_{10}(x) \geq 5.0$

From (23), it was obvious that if $y_j(x) \to y_{j\,\min}$ and $\forall y_j(x) < C_j$, $r_j(x) \to r_{j\max} = 1$. By choosing R*(x) as the objective function, the m-objective optimization problem is restated as: Find $x^R = (x_1^R, x_2^R, x_3^R) \in \Omega_x$ in order that R*(x) reaches the maximum value (Dzung, 2012a & 2015).

$$\begin{cases} R^*_{\max} = R^*(x^R) = R^*(x_1^R, x_2^R, x_3^R) \\ = \max\left\{R^*(x)\right\} = \max\left\{\sqrt[10]{\left[\prod_{j=1}^{10} r_j(x)\right]}\right\} \\ \Omega_x = \left\{-1.414 \leq x_1,\ x_2,\ x_3 \leq 1.414\right\}; \\ x = (x_1, x_2, x_3) \end{cases} \quad (25)$$

From (23), it can be seen: $0 \leq R^*(x^R) \leq 1$. If $R^*(x^R) = 1$, $x^R = x^{UT}$ – the utopian root. If $R^*(x^R) = 0$, one of the values of $y_j(x)$ violates $\{y_j < C_j\}$, which means that $y_j(x)$ belongs to the restricted area $C = \{y_j \geq C_j\}$, (Dzung, 2012a).

The eleven-objective optimization problem needed to indentify $x^R = (x_1^R, x_2^R, x_3^R) \in \Omega_x$ in order that $R^*(x_1^R, x_2^R, x_3^R) = \mathrm{Max}\{R^*(x_1, x_2, x_3)\}$. The maximum value of (25) was determined by the meshing method programmed in Matlab R2008a software (Dzung, 2012a):

$$R^*(x)_{\max} = \mathrm{Max}\{R^*(x_1, x_2, x_3)\}$$
$$= R^*(x_1^R, x_2^R, x_3^R) = 0.476$$

Where: $x_1^R = -0.883$;
$x_2^R = -0.115$;
$x_3^R = -0.859$;

Then, transforming into real variables:

$Z_1^{opt} = 20.58^0C$;
$Z_2^{opt} = 0.411mmHg$;
$Z_3^{opt} = 18.283h$

Substituting x_1^R, x_2^R, x_3^R into these equations from (10) to (19), the results were obtained as:

$y_{1P}^R = 6.32$; $y_{2P}^R = 4.19$;
$y_{3P}^R = 1.77$; $y_{4P}^R = 1.83$;
$y_{5P}^R = 2.07$; $y_{6P}^R = 2.36$;
$y_{7P}^R = 0.78$; $y_{8P}^R = 2.47$;
$y_{9P}^R = 2.67$; $y_{10P}^R = 3.58$;

Where: $x^R = (x_1^R, x_2^R, x_3^R)$ called optimal Pareto root and $f_p^R = y_P^R = (y_{1P}^R, y_{2P}^R, y_{3P}^R, y_{4P}^R, y_{5P}^R, y_{6P}^R, y_{7P}^R, y_{8P}^R, y_{9P}^R, y_{10P}^R)$ called the optimal Pareto effect.

For this reason, through the calculation from the experimental models from Eq. (10) to Eq. (19), technological parameters of the freeze drying process which satisfied the maximum R*-Optimal combination criterion were determined as: temperature of freeze drying chamber was $Z_1^{opt} = 20.58^0C$, pressure of freeze drying chamber was $Z_2^{opt} = 0.411mmHg$, time of freeze drying was $Z_3^{opt} = 18.283h$. Corresponding to: the energy consumption of 1 kg final product was $y_{1P}^R = 6.32$ kWh/kg; the residual water content of the product was $y_{2P}^R = 4.19\%$ ($< 4.5\%$); the loss of total protein was $y_{3P}^R = 1.77\%$; the loss of carbohydrate was $y_{4P}^R = 1.83\%$; the loss of lipid was $y_{5P}^R = 2.07\%$; the loss of mineral salts was $y_{6P}^R = 2.36\%$; the loss of 10-HDA was $y_{7P}^R = 0.78\%$; the loss of vitamin B_5 was $y_{8P}^R = 2.47\%$; the loss of free fatty acids was $y_{9P}^R = 2.67\%$; the loss of viscosity was $y_{10P}^R = 3.58\%$. Compared with the experimental results from the Table 3a & Table 3b, these results above were suitable and satisfying with the objectives of the problem.

3.6. Experiment to test the optimal Pareto root of multi-objective optimization problem

Carrying out the freeze drying process of Royal jelly at the optimal Pareto root: temperature of freeze drying chamber of $Z_1^{opt} = 20.58^0C$, pressure of freeze drying chamber of $Z_2^{opt} = 0.411$mmHg, and time of freeze drying $Z_3^{opt} = 18.283$ hours, the experimental results were determined as: energy consumption of 1 kg final product was $y_1 = 6.34$ kWh/kg; the residual water content of the product was $y_2 = 4.17\%$ ($< 4.5\%$); the loss of total protein was $y_3 = 1.80\%$; the loss of carbohydrate was $y_4 = 1.79\%$; the loss of lipid was $y_5 = 2.05\%$; the loss of mineral salts was $y_6 = 2.34\%$; the loss of 10-HDA was $y_7 = 0.79\%$; the loss of vitamin B5 was $y_8 = 2.45\%$; the loss of free fatty acids was $y_9 = 2.69\%$; the loss of viscosity was $y_{10} = 3.61\%$.

Consequently, it was very noticeable that the results from the optimization problems of the freeze drying process had the approximation to the experimental results. When the pressure of freeze drying chamber was fixed: $x_2 = -0.115$, respectively $Z_2 = 0.411$ mmHg, the relationship between y_1, y_2, y_3, y_4, y_5 y_6, y_7, y_8, y_9 and y_{10} combination function with 2 variables x_1, x_3 was performed geometrically in 3D (Figures 4, 5, 6, 7, 8, 9, 10, 11, 12, 13). When x_1 was fixed with constant values, the variation of x_3 was shown in Figures 14, 15, 16, 17, 18, 19, 20, 21, 22, 23.

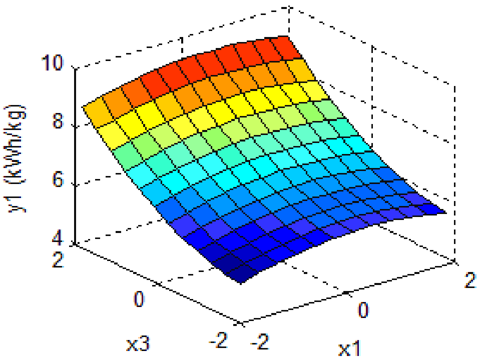

Fig 4. The energy consumption of 1kg final product y1 (kWh/kg)

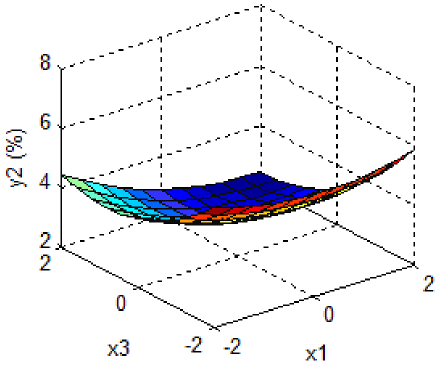

Fig 5. The residual water content of final product y2 (%)

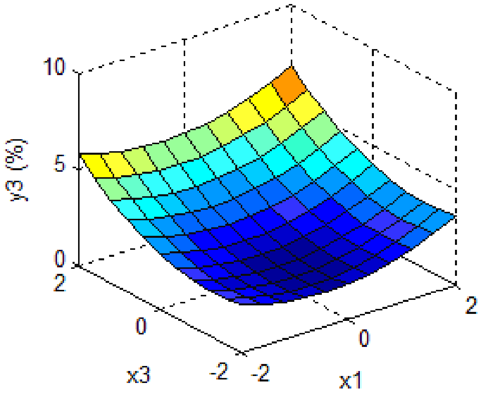

Fig 6. The loss of total protein of final product y3 (%)

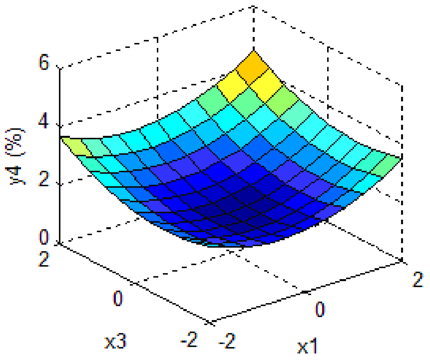

Fig 7. The loss of carbohydrate of final product y4 (%)

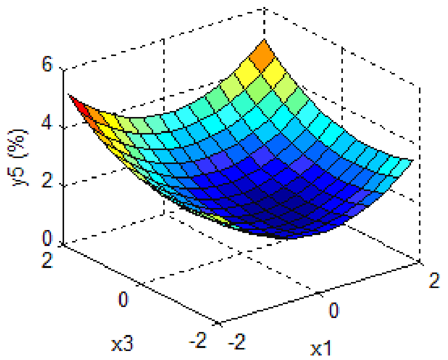

Fig 8. The loss of lipid of final product y5 (%)

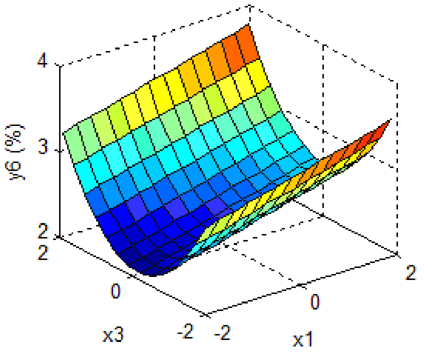

Fig 9. The loss of mineral salts of final product y6 (%)

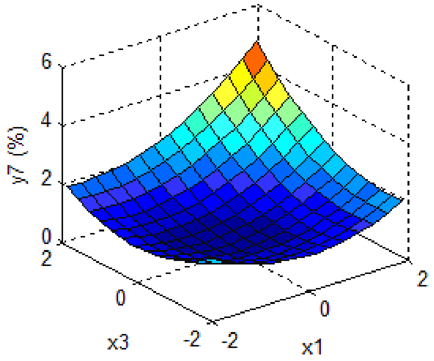

Fig 10. The loss of 10-HDA of final product y7 (%)

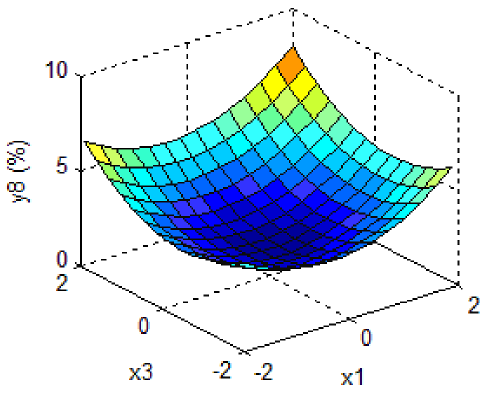

Fig 11. The loss of vitamin B5 of final product y8 (%)

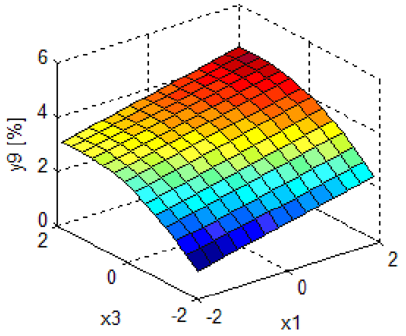

Fig 12. The loss of free fatty acids of final product y9 (%)

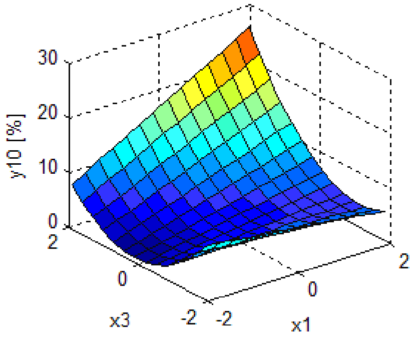

Fig 13. The loss of viscosity of final product y10 (%)

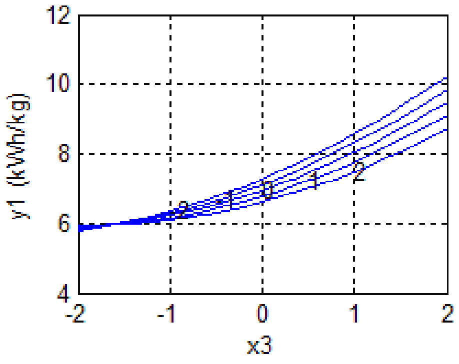

Fig 14. The energy consumption of final product y1 (kWh/kg)

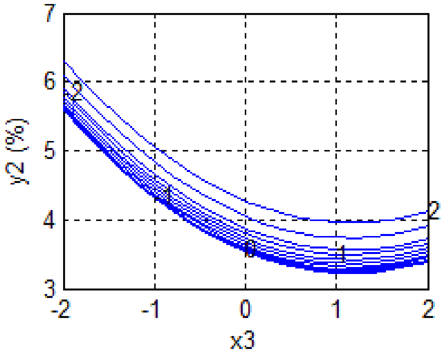

Fig 15. The residual water content of final product y2 (%)

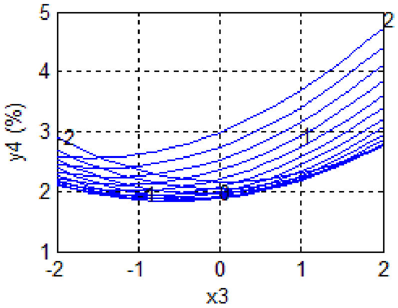

Fig 17. The loss of carbohydrate of final product y4 (%)

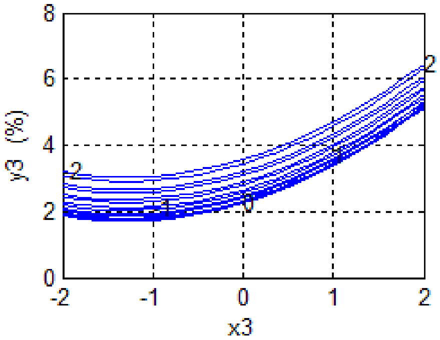

Fig 16. The loss of total protein of final product y3 (%)

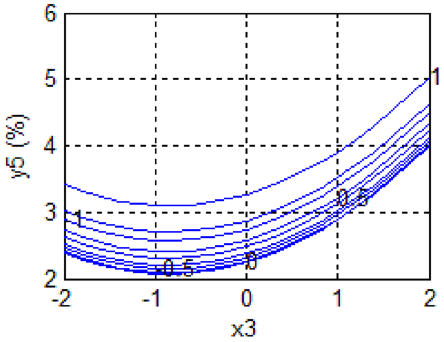

Fig 18. The loss of lipid of final product y5 (%)

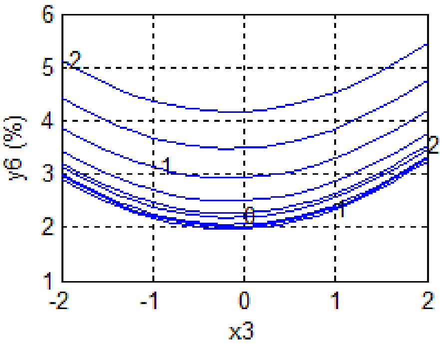

Fig 19. The loss of mineral salts of final product y6 (%)

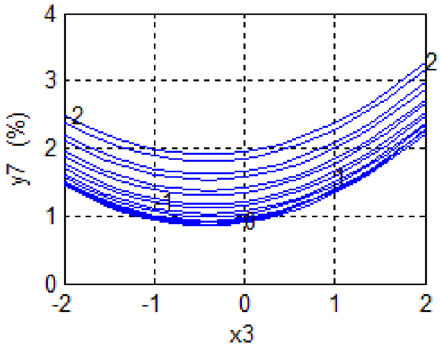

Fig 20. The loss of 10-HDA of final product y7 (%)

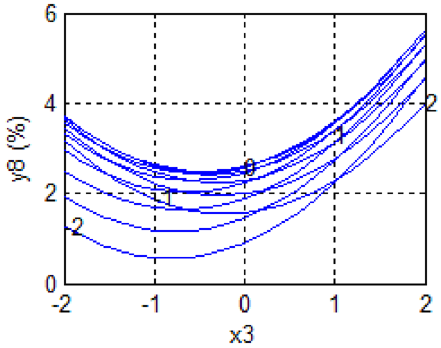

Fig 21. The loss of vitamin B5 of final product y8 (%)

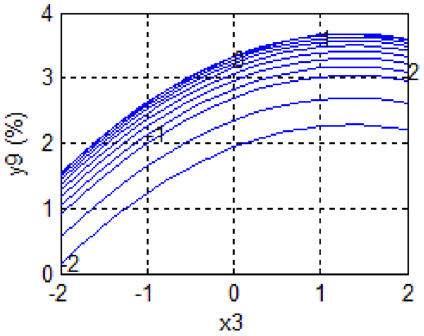

Fig 22. The loss of free fatty acids of final product y9 (%)

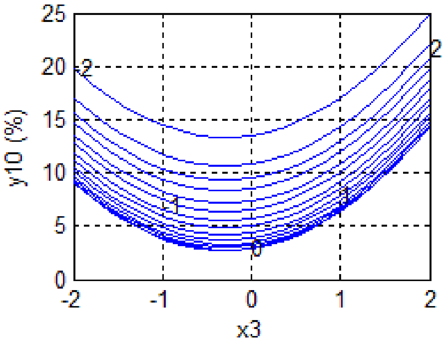

Fig 23. The loss of viscosity of final product y10 (%)

All Figures on above was obvious that objective functions were varied by effect factors during the freeze drying process of Royal jelly. This varying objective functions were completely suitable with experimental results. Therefore, it proved that relationships between objective functions with effect factors very well described for the freeze drying process of Royal jelly.

3.7. Determining technological mode of freezing process of Royal jelly

From results on above, it allowed to set up the technological mode during the freeze drying process of Royal jelly in Table 5 as follow:

Table 5. The technological mode of the freeze drying process of Royal jelly

No	Technological Parameters	Symbol and unit	Value
1	The temperature of the freezing environment	$Z_1 = T_{\infty}$, (^{0}C)	20.58
2	The time of freezing process	$Z_2 = P$, (mmHg)	0.411
3	the thickness of Royal jelly in the tray	$Z_3 = \tau$, (h)	18.283
The standards of final product of Royal jelly after freeze drying			
4	The energy consumption of 1 kg final product	y_{1P}^R, (kWh/kg)	6.32
5	The residual water content of final product	y_{2P}^R, (%)	4.19
6	The loss of total protein of final product	y_{3P}^R, (%)	1.77
7	The loss of carbohydrate of final product	y_{4P}^R, (%)	1.83
8	The loss of lipid of final product	y_{5P}^R, (%)	2.07
9	The loss of mineral salts of final product	y_{6P}^R, (%)	2.36
10	The loss of 10-HDA of final product	y_{7P}^R, (%)	0.78
11	The loss of vitamin B5 of final product	y_{8P}^R, (%)	2.47
12	The loss of free fatty acids of final product	Y_{9P}^R, (%)	2.67
13	The loss of viscosity of final product	y_{10P}^R, (%)	3.58

From Table 5, it was obvious when Royal jelly was carried out at the optimal technological mode of freeze drying process, the quality of Royal jelly after freeze drying had very good as the same quality of Royal jelly before freeze drying. In the fact that has not any drying method can create product to have good quality as the same product of the freeze drying process. Therefore, the technological mode of freeze drying process of Royal jelly was found out on above, it can be completely applied for Royal jelly preservation in order to be prolonged use time and export time (Dzung, 2012a).

4. Conclusions

The mathematical models (10) to (19) which were established from the experiments quite well described the relationship between the temperature of freeze drying chamber; the pressure of freeze drying chamber; the time of freeze drying process of Royal jelly with the energy consumption of 1 kg final product of freeze drying Royal jelly; the residual water content of final product of Royal jelly after freeze drying; the loss of total protein; the loss of carbohydrate; the loss of lipid; the loss of mineral salts; the loss of 10-HDA; the loss of vitamin B_5; the loss of free fatty acids and the loss of viscosity of final product of Royal jelly (Dzung, 2012a).

The system of equation (21) was the multi-objective optimization problems of the freeze drying process of Royal jelly. This mathematical model was suitably used for calculating and setting up the technological mode of the freeze drying process of Royal jelly (Dzung, 2012a).

Solving the multi-objective optimization problems (21) determined the technological mode of the freeze drying process of Royal jelly (Dzung, 2012a). The results were presented in Table 5.

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