A Comparison of Factors that Influence the Lyophilization Process

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Summary

The lyophilization (or freeze drying) process for agro-foods products depends on a series of technological factors that are in an inter-dependence with the process performance. This paper presents an expert method and its application. This method characterizes the influence factors of the lyophilization process, after the importance level of some factors in correlation with other factors, is defined. Only the most important factors were considered; influence considerations were made in relation to some adjustment factors of the lyophilization system. These research results were necessary for re-consideration and re-design of agro-foods lyophilization systems.

Key words

lyophilization, factors, importance, optimization, process

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Received: November 23, 2006 | Accepted: March 22, 2007

Introduction

Freeze-drying has a great impact upon the production of dehydrated foods because of the superior quality of the product obtained and promises continued expansion of the number of applications (Estiaghi, 1994). Substances that are not damaged by freezing can usually be lyophilized (freeze-dried) so that refrigerated storage is unnecessary (Jennings, 1999). To continue the optimization of foodstuffs lyophilization system utilization it is necessary to study the correlations between factors that influence the lyophilization process and to establish the order of importance.

The determination of the critical events occurring in the frozen material is essential for scientists currently involved with freeze drying development work, so they can select lyophilization parameters on a rational and product-specific basis (Ray, 1999).

Material and methods

In this paper, we continued study of the role of the essential factors in the lyophilization process using an expert method (Tucu, 1995), which was based on the opinions of a number "*m*" experts, representatives of companies producing lyophilization installations, research teams, teaching staff and users. The experts expressed their opinions by ordering factors, based on the "ranks" (1, 2, 3, 4… k, where *k* = number of factors), of one factor to another, to the factors of the process, as a function of their influence upon the state factor.

The results were assembled on separate sheets for each group of state variables and influence factors, synthesized in the factor-ordering matrices. In the factor-ordering matrix each line contained a row of natural numbers disposed differently: x_{ij} represents the rank attributed by the specialist "*i*" to the factor "*j*". In case of increase in the resolution of determination, fractional numbers can be used too, observing the condition:

$$\sum_{j=1}^{k} x_{ij} = \frac{1}{2} \cdot k \cdot (k+1) \qquad (1)$$

An arithmetical mean of the number in a row is:

$$M_{a_j} = \frac{(k+1)}{2} \qquad (2)$$

where a_j is the mean value of the rank j, that is

$$a_j = \frac{A_j}{m} \qquad (3)$$

where *m* represents the number of experts, and A_j was calculated with the relation:

$$A_j = \sum_{i=1}^{m} x_{ij}\ , \qquad (4)$$

The share coefficient of the factor is (Taloi, 1987):

$$M_j = \frac{A_j}{\sum_{j=1}^{k} A_j} \qquad (5)$$

where *k* is the number of factors.

The indicators, called importance level factors (ILF), which represent the measure in which these factors influence other factors/variables, were determined with the relation:

$$ILF = \frac{1}{M_j} \qquad (6)$$

For the interaction between the influence factors, histograms with the results shown in the table of expert opinions were presented.

In order to continue an important method of optimization of the lyophilization process, we used the opinions of nine experts: from a range of lyophilization installations product firms, from research groups, teachers and users. They considered some factors/variables of the influence system for the lyophilization process (Anghel, 2006):

a. The group of factors as variables relating to the *lyophilization technique system*, including the characteristics considered to be the most important in defining their qualities (Bacauanu, 2005):

- duration of lyophilization by unit of product – D_1
- the exposed surface of thermal exchange – S_1
- cooling velocity – V_1
- the quality factor of the thermal transfer – Q_1
- the corrosion resistance of the material messed up in contact with foodstuff – R_1
- the condition of the metal surfaces in contact with the product – M_1
- the vacuum capacity of the lyophilization space – C_1

b. The group of adjustment factors, variables of the system:

- freezing velocity – M_4
- the thermal transfer surface of the food (Carapelle et al., 2001) – D_4
- compensation for the breakage mechanical effects of the structural elements – B_4
- compensation for the heat losses of the environment – T_4
- setting the food in the lyophilization system – P_4
- the sensitivity threshold of the lyophilization system – S_4

Table 1.
Determination of importance level of adjustment factors in relation to duration of lyphilization by unit of product - D_1

Adjustment factor	M_4	D_4	B_4	T_4	P_4	S_4
Expert nr.	Importance level considered by each expert (1÷6)					
1	2	4	5	3	1	6
2	1	3	6	4	2	5
3	1	4	6	3	2	5
4	1	4	6	3	2	5
5	1	4	6	3	2	5
6	1	4	6	2	3	5
7	1	4	6	3	2	5
8	1	4	6	3	2	5
9	1	5	6	3	2	4
A_j	10	36	53	27	18	45
ILF	18.868	5.236	3.571	6.993	10.526	4.202

Table 2.
Determination of importance level of adjustment factors in relation to the exposed surface of thermal exchange - S_1

Adjustment factor	M_4	D_4	B_4	T_4	P_4	S_4
Expert nr.	Importance level considered by each expert (1÷6)					
1	2	3	5	4	6	1
2	1	4	6	3	5	2
3	1	4	5	2	6	3
4	1	4	5	3	6	2
5	1	3	5	4	6	2
6	1	4	5	2	6	3
7	1	4	5	3	6	2
8	1	4	5	2	6	3
9	2	4	6	3	5	1
A_j	11	34	47	26	52	19
ILF	17.182	5.559	4.021	7.269	3.635	9.947

Table 3.
Determination of importance level of adjustment factors in relation to the cooling velocity, - V_1

Adjustment factor	M_4	D_4	B_4	T_4	P_4	S_4
Expert nr.	Importance level considered by each expert (1÷6)					
1	1	6	2	4	3	5
2	2	6	1	5	4	3
3	3	5	1	6	2	4
4	2	6	1	5	3	4
5	2	6	1	5	3	4
6	2	6	1	5	4	3
7	3	5	1	6	2	4
8	2	6	1	4	3	5
9	1	6	2	5	3	4
A_j	18	52	11	45	27	36
ILF	10.526	3.636	17.241	4.202	6.994	5.236

Table 4.
Determination of importance level of adjustment factors in relation to the quality factor of the thermal transfer, - Q_1

Adjustment factor	M_4	D_4	B_4	T_4	P_4	S_4
Expert nr.	Importance level considered by each expert (1÷6)					
1	1	6	2	4	3	5
2	2	6	1	5	4	3
3	3	5	1	6	2	4
4	2	6	1	5	3	4
5	2	6	1	5	3	4
6	2	6	1	5	4	3
7	3	5	1	6	2	4
8	2	6	1	4	3	5
9	1	6	2	5	3	4
A_j	18	52	11	45	27	36
ILF	10.526	3.636	17.241	4.202	6.994	5.236

Table 5
Determination of importance level of adjustment factors in relation to the corrosion resistance, - R_1

Adjustment factor	M_4	D_4	B_4	T_4	P_4	S_4
Expert nr.	Importance level considered by each expert (1÷6)					
1	1	2	3	5	4	6
2	2	1	3	6	4	5
3	2	1	4	5	3	6
4	2	1	5	4	3	6
5	3	1	4	5	2	6
6	1	2	4	5	3	6
7	2	1	5	4	3	6
8	2	1	4	6	3	5
9	3	1	4	5	2	6
A_j	18	11	36	45	27	52
ILF	10.753	17.241	5.208	4.202	6.993	3.636

Table 6.
Determination of importance level of adjustment factors in relation to the condition of the metal surfaces in contact with the product, - M_1

Adjustment factor	M_4	D_4	B_4	T_4	P_4	S_4
Expert nr.	Importance level considered by each expert (1÷6)					
1	2	3	5	4	6	1
2	1	4	6	3	5	2
3	1	4	5	2	6	3
4	1	4	5	3	6	2
5	1	3	5	4	6	2
6	1	4	5	2	6	3
7	1	4	5	3	6	2
8	1	4	5	2	6	3
9	2	4	6	3	5	1
A_j	11	34	47	26	52	19
ILF	17.241	5.555	4.016	7.246	3.636	9.901

Results and discussion

With the expert opinions, the relations (1) - (6) were scanned, and finally resulted in Tables 1 - 7, with the values of the indicators of importance level, ILF of adjustment factors (M_4, D_4, B_4, T_4, P_4, S_4) with each most important factors for a good quality of lyophilizated products, (D_1, S_1, V_1, Q_1, R_1, M_1, C_1). The results of these correlations are presented in the form of histograms shown in figures 1 - 7, with some linear and some non-linear distributions.

The order of adjustment factors as a function of the importance level related to the duration of the lyophiliza-

Table 7.
Determination of importance level of adjustment factors in relation to the vacuum capacity of the lyophilization space,- C_1

Adjustment factor	M_4	D_4	B_4	T_4	P_4	S_4
Expert nr.	Importance level considered by each expert (1÷6)					
1	2	3	5	4	6	1
2	1	4	6	3	5	2
3	1	4	5	2	6	3
4	1	4	5	3	6	2
5	1	3	5	4	6	2
6	1	4	5	2	6	3
7	1	4	5	3	6	2
8	1	4	5	2	6	3
9	2	4	6	3	5	1
A_j	11	34	47	26	52	19
ILF	17.241	5.555	4.016	7.246	3.636	9.901

tion per unit of product – D_1 are presented in Table 1 and Figure 1. We noted that velocity of freezing is very important for the duration of lyophilization while setting of the food in system has low importance. Also the freezing velocity is very important for the exposed surface of thermal exchange, or for quality factor of the thermal transfer, but it is not so important for vacuum capacity or corrosion resistance. A similar situation was noticed in relation with the compensation for the breakage mechanical effects of the structural elements (B_4), which was very important for vacuum capacity (C_1) and the cooling velocity (V_1), but in rest has a minimal importance in comparison with the rest of adjustment factors. Similarly, this method of analysis can be used in the study of correlations between all of the influence factors and output variables.

Conclusions

The choice of variables proved correct, as the experts considered each opinion as having a significant share of input variables, as an influence factor for the output variables considered. It is most important to know, within the system of influence factors for the lyophilization process, the order of importance in the relation of each factor with the others (Liapis et al., 1995). But this study represents just one step in the process of lyophilization optimization. The method used can be employed for the reconsideration of current design methods, or for re-design of the lyophiliser using the practical experience of the experts.

For a complete optimization it is necessary to consider all criteria, in an order established also by experts, taking into account both the optimal conditions of the technique as well as any restrictions imposed by the lyophilization process, including economic considerations.

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