Dynamics model for the thermal performance from a lyophilization process, based on a complete transfer functions matrix

Modelo dinámico para el rendimiento térmico de un proceso de liofilización, basado en una matriz de funciones de transferencia completa

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Abstract. – *During the beginning of the XX century lyophilization was developed as an alternative technology to extend the storage time for fruit and vegetables or other kind of food; however, the energetic consumption of this technology makes it not an option for common food producers, less over for those one that work by the open field cultivation technique. The main energy consumption in a lyophilization systems are the motors from the vacuum pump and from the refrigerant compressors; due to the temperature range needs the lyophilization systems use to have more than one cooling thermodynamic system based on vapor compression. This paper describes an experimental methodology to get a complete state transfer functions matrix, based on the graphical analysis of the concerned transfer functions magnitude spectra. This experimental data came from a set of test performed at the National Laboratory for Cooling Technology Research (LaNITeF) at the Engineering Center for Industrial Development (CIDESI). The intention of this transfer functions matrix is to be applied in a control strategy to then optimize the energetic performance of the concerned lyophilization system. This function transfer matrix is considered complete because there is not a dynamic order reduction considering its degrees of freedom. The transfer functions matrix describes the dynamic relationship between both the inputs variables that describe the energetic consumption of the lyophilization system, and the ambient conditions, as well as the output variables that represent*

the dynamical states vector with the variables of interest from the concerned process. The simulation from an experimental scenario worked as the graphical validation of the transfer functions matrix characterized experimentally, so the main conclusion of this scientific work is that this transfer functions matrix can be used as dynamic model to implement control and optimization algorithms.

Keywords: Transfer functions matrix; Thermal performance; Lyophilization process.

Resumen. - Durante el comienzo del siglo XX, la liofilización se desarrolló como una tecnología alternativa para extender el tiempo de almacenamiento de frutas y verduras u otro tipo de alimentos; Sin embargo, el consumo energético de esta tecnología hace que no sea una opción para los productores de alimentos comunes, menos para aquellos que trabajan con la técnica de cultivo en campo abierto. El principal consumo de energía en un sistema de liofilización son los motores de la bomba de vacío y de los compresores de refrigerante; Debido al rango de temperatura que necesitan los sistemas de liofilización para tener más de un sistema termodinámico de enfriamiento basado en la compresión de vapor. Este artículo describe una metodología experimental para obtener una matriz completa de funciones de transferencia de estado, basada en el análisis gráfico de los espectros de magnitud de las funciones de transferencia en cuestión. Estos datos experimentales provienen de un conjunto de pruebas realizadas en el Laboratorio Nacional de Investigación de Tecnología de Refrigeración (LaNITeF) en el Centro de Ingeniería para el Desarrollo Industrial (CIDESI). La intención de esta matriz de funciones de transferencia es aplicarla en una estrategia de control para luego optimizar el rendimiento energético del sistema de liofilización en cuestión. Esta matriz de transferencia de funciones se considera completa porque no hay una reducción de orden dinámico considerando sus grados de libertad. La matriz de funciones de transferencia describe la relación dinámica entre las variables de entrada que describen el consumo energético del sistema de liofilización y las condiciones ambientales, así como las variables de salida que representan el vector de estados dinámicos con las variables de interés del proceso en cuestión. La simulación de un escenario experimental funcionó como la validación gráfica de la matriz de funciones de transferencia caracterizada experimentalmente, por lo que la conclusión principal de este trabajo científico es que esta matriz de funciones de transferencia puede usarse como modelo dinámico para implementar algoritmos de control y optimización.

Palabras clave: Matriz de funciones de transferencia; Rendimiento térmico; Proceso de liofilización.

1.   Introduction

Because of the cooling speed and load capacity, traditional refrigeration and freezing process based on the vapor compression technologies are the most used alternatives for the perishables products conservation (production, storage, transportation, distribution and exhibition) [3 – 5].

It is estimated that just in Mexico the 30 % of the food production is wasted because of several issues with the cold chain and refrigeration [1]; and it occurs almost the same with the final availability for the perishable medicine in rest of Latin America [2].

But the wastes from the production of food and medicine is not the only opportunity for the refrigeration process; because, nowadays the temporal cultivation of fruits and vegetable through open field techniques, does not represent a convent opportunities for communities that have this economical access naturally. And the due to the inefficiency of the conventional cooling technology, the producers do not have the opportunity to apply it to extend their market presence by transporting or exporting their products and then increasing their utility [6 INEGI].

It is well known that the natural cooling process into the vapor compression technologies affects the electrochemical properties of food and medicine [6, 7]; so, that, since the beginning of the XX century the lyophilization process appears [8] by being an alternative for the food and medicine preservation process, because its preserves their structural composition by a fast cooling process into a vacuum ambient for the water extraction [9].

Both lyophilization sub-stages mainly defined as the fast cooling and the vacuum ambient are high energy expending process; due to the motors power that move the refrigerant compressors and the vacuum pumps. Therefore, a mathematical model for the dynamic behavior of the thermal and energetic variables from a lyophilization device are proposed, with the intention to use it in future works for the tuning of control algorithms that can optimize this lyophilization process energetic consumption.

The mathematical model for the specific lyophilization process described in this document comes from a state space model, and consists in a transfer function matrix characterized by the spectral response of the thermodynamic variables involved on the concerned process.

All experimental effort and analysis was developed at the National Laboratory for Cooling Technology Research (LaNITeF) at the Engineering Center for Industrial Development (CIDESI), whom is part of the Public Research Centers and the National Laboratories Network form the National Council for Science and Technology (CONACYT).

The lyophilization process modeling is described in this document by starting with the lyophilization process description and

its variables identification in the second chapter. Then the experimental testing of the lyophilization process is documented into the third chapter, and just after it, on section forth the experimental results analysis is. This results analysis works as reference for the conclusions synthesis in the last chapter.

2.   Lyophilization

Mainly, the lyophilization process consist in three sub-process:

1.   "Conduction" to remove most of the food water.
2.   "Diffusion stage 1" to consolidate the food electrochemical properties.
3.   "Diffusion stage 2" to consolidate the food physical properties.

In general, the food lyophilization consist on its gases solidification by passing through the liquid phase just with the water concentration needed to preserves its original molecular structure [9 - 11].

To clarify the temperature behavior of a lyophilization process, Fig. 1 illustrates the temperature of the cooling chamber from the lyophilization device used on this paper, working with 87.37 grams of fruit load.

Fig. 2 shows the schematic diagram of the lyophilization systems used in this researching work, where it is possible to see that this device consists of two vapor compression refrigeration systems, one for the R-23 refrigerant gas (system 1) and the second one for the R-507a (system 2) refrigerant.
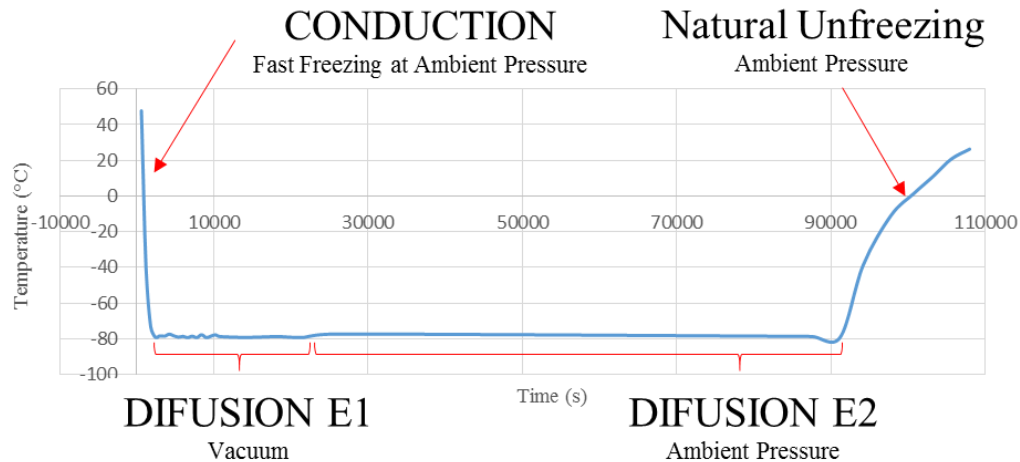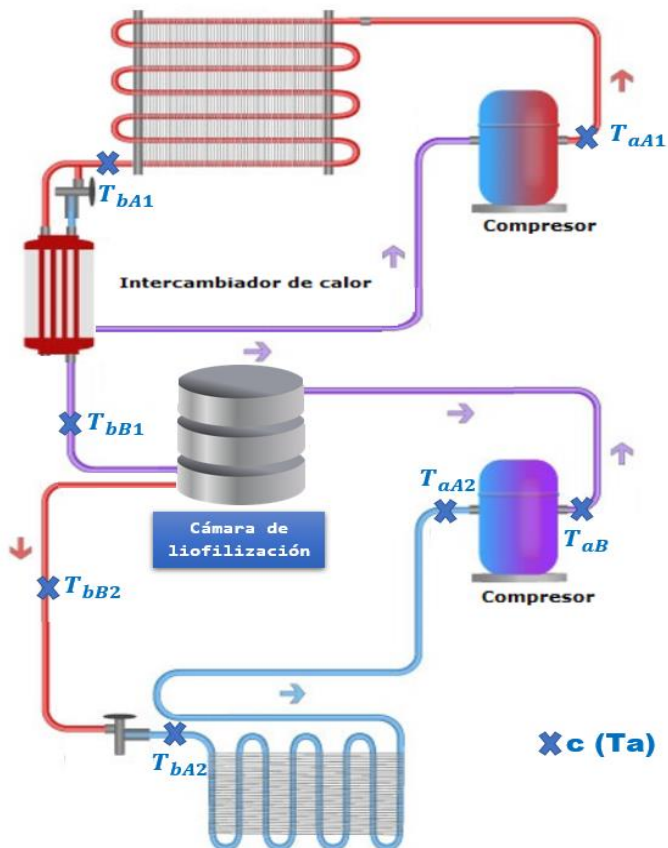

Figure 1. Cooling chamber temperature.

Figure 2. Schematic diagram of the lyophilization device.

Table 1 lists the variables involved in the dynamics model for the lyophilization process, considering the thermodynamic states of the refrigerant gases as the model outputs and as inputs the variables related with both the energetic consumption as well as the ambient conditions.

Table 1. Lyophilization process model variables.

Variable	Description	Units
T_{aA1}	High Temperature at High Pressure on System 1	°C
T_{bA1}	Low Temperature at High Pressure on System 1	°C
T_{bB1}	Low Temperature at Low Pressure on Systems 1	°C
$T_{aB} = T_{aB1} = T_{aB2}$	Common High Temperature at Low Pressure	°C
T_{aA2}	High Temperature at High Pressure on System 2	°C
T_{bA2}	Low Temperature at High Pressure on System 2	°C
T_{bB2}	Low Temperature at Low Pressure on System 2	°C
T_a	Ambient Temperature	°C
P_a	Atmospheric Pressure	Bar
E_{c1}	Compressor Energy Consumption System 1	W
E_{c2}	Compressor Energy Consumption System 2	W
E_{bv}	Vacuum Pump Energy Consumption	W
E_{fc}	Heat Source Energy Consumption	W

3. Lyophilization dynamics

As it has been clarified the intention of this mathematical model is to be the base of an energetic optimization strategy, by modifying the dynamic response of the described lyophilization system. Then the dynamic model proposed as state space, was defined as:

$$\frac{d}{dt}\begin{bmatrix} T_{aA1}(t) \\ T_{bA1}(t) \\ T_{bB1}(t) \\ T_{aB}(t) \\ T_{aA2}(t) \\ T_{bA2}(t) \\ T_{bB2}(t) \end{bmatrix} = [A]_{7\times7}\begin{bmatrix} T_{aA1}(t) \\ T_{bA1}(t) \\ T_{bB1}(t) \\ T_{aB}(t) \\ T_{aA2}(t) \\ T_{bA2}(t) \\ T_{bB2}(t) \end{bmatrix} + [B]_{7\times6}\begin{bmatrix} E_{c1}(t) \\ E_{c2}(t) \\ E_{bv}(t) \\ E_{fc}(t) \\ T_a(t) \\ P_a(t) \end{bmatrix}, \tag{1}$$

where A and B are the dynamics matrix and the input matrix respectively, then applying the Fourier transfer and by solving for the state vector, we have:

$$
\begin{bmatrix}
T_{aA1}(\omega) \\
T_{bA1}(\omega) \\
T_{bB1}(\omega) \\
T_{aB}(\omega) \\
T_{aA2}(\omega) \\
T_{bA2}(\omega) \\
T_{bB2}(\omega)
\end{bmatrix}
= [j\omega[I]_{7\times7} - [A]_{7\times7}]^{-1}
\begin{bmatrix}
E_{c1}(\omega) \\
E_{c2}(\omega) \\
E_{bv}(\omega) \\
E_{fc}(\omega) \\
T_a(\omega) \\
P_a(\omega)
\end{bmatrix},
\tag{2}
$$

where the matrix in the between of the state vector and the input vector is known as the transfer function matrix, which can be re-written as:

$$
\begin{bmatrix}
T_{aA1}(\omega) \\
T_{bA1}(\omega) \\
T_{bB1}(\omega) \\
T_{aB}(\omega) \\
T_{aA2}(\omega) \\
T_{bA2}(\omega) \\
T_{bB2}(\omega)
\end{bmatrix}
= \frac{1}{P(\omega)}
\begin{bmatrix}
Z_{1,1}(\omega) & Z_{1,2}(\omega) & Z_{1,3}(\omega) & Z_{1,4}(\omega) & Z_{1,5}(\omega) & Z_{1,6}(\omega) \\
Z_{2,1}(\omega) & Z_{2,2}(\omega) & Z_{2,3}(\omega) & Z_{2,4}(\omega) & Z_{2,5}(\omega) & Z_{2,6}(\omega) \\
Z_{3,1}(\omega) & Z_{3,2}(\omega) & Z_{3,3}(\omega) & Z_{3,4}(\omega) & Z_{3,5}(\omega) & Z_{3,6}(\omega) \\
Z_{4,1}(\omega) & Z_{4,2}(\omega) & Z_{4,3}(\omega) & Z_{4,4}(\omega) & Z_{4,5}(\omega) & Z_{4,6}(\omega) \\
Z_{5,1}(\omega) & Z_{5,2}(\omega) & Z_{5,3}(\omega) & Z_{5,4}(\omega) & Z_{5,5}(\omega) & Z_{5,6}(\omega) \\
Z_{6,1}(\omega) & Z_{6,2}(\omega) & Z_{6,3}(\omega) & Z_{6,4}(\omega) & Z_{6,5}(\omega) & Z_{6,6}(\omega) \\
Z_{7,1}(\omega) & Z_{7,2}(\omega) & Z_{7,3}(\omega) & Z_{7,4}(\omega) & Z_{7,5}(\omega) & Z_{7,6}(\omega)
\end{bmatrix}
\begin{bmatrix}
E_{c1}(\omega) \\
E_{c2}(\omega) \\
E_{bv}(\omega) \\
E_{fc}(\omega) \\
T_a(\omega) \\
P_a(\omega)
\end{bmatrix},
\tag{3}
$$

where:

$P(\omega)$ is the polynomial conformed by the natural frequencies (Eigen values), and $Z_{n,m}(\omega)$ represents the zeros polinomiun from the concerned spectral relationship between *n*-output and *m*-input variables.

4.   Experimental characterization

With the intention to know both poles (eigenvalues) and zeros, a set of experiments where performed in the lyophilization device, where all model variables listed on table 1 were measured and processed to know the spectral response of each them. Figs 3 to 8 and 9 to 15 plot the inputs and outputs variables behavior respectively.
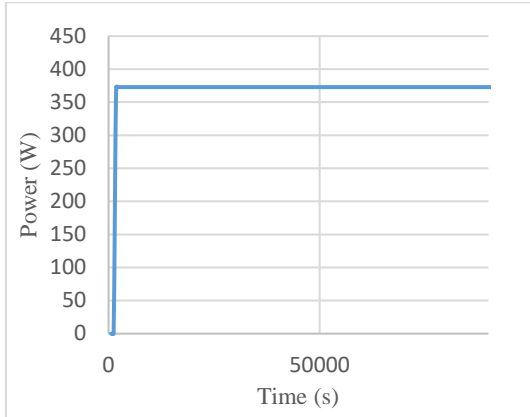
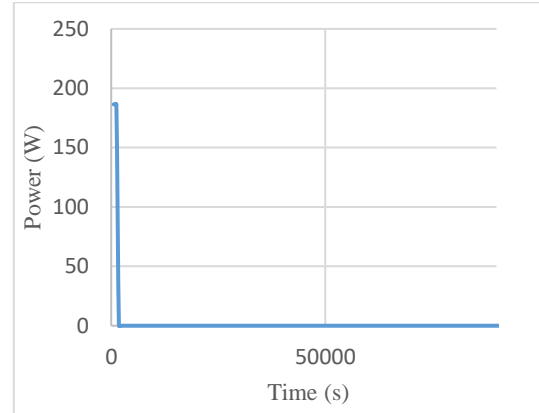**Figure 3.** Dynamic behavior of the compressor energy consumption from system 1

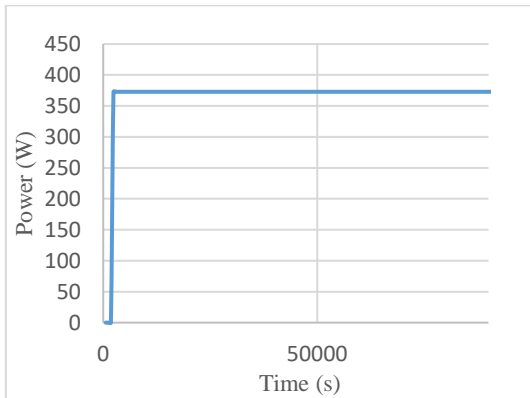**Figure 4.** Dynamic behavior of the compressor energy consumption from system 2

Figure 5. Dynamic behavior of the vacuum pump energy consumption

Figure 6. Dynamic behavior of the heat source energy consumption.
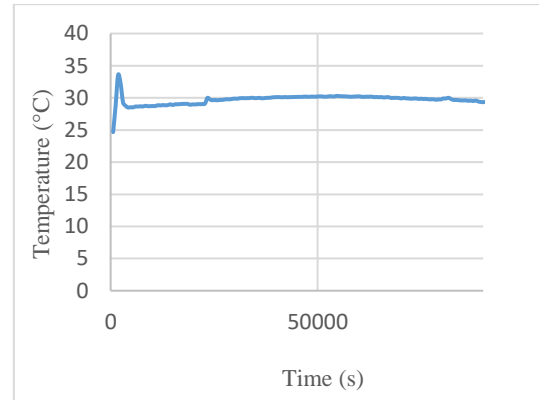
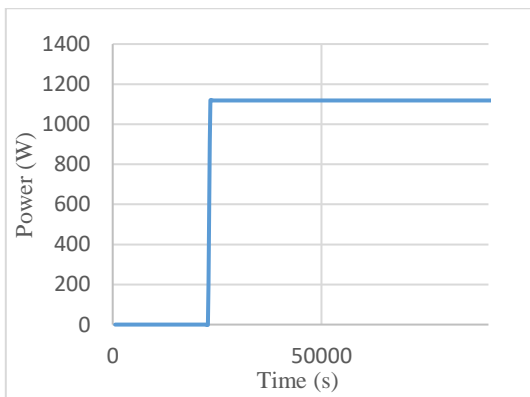
Figure 7. Dynamic behavior of the ambient temperature.
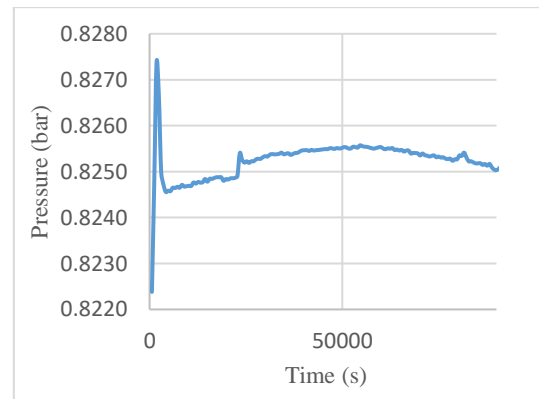
Figure 8. Dynamic behavior of the atmospheric pressure.
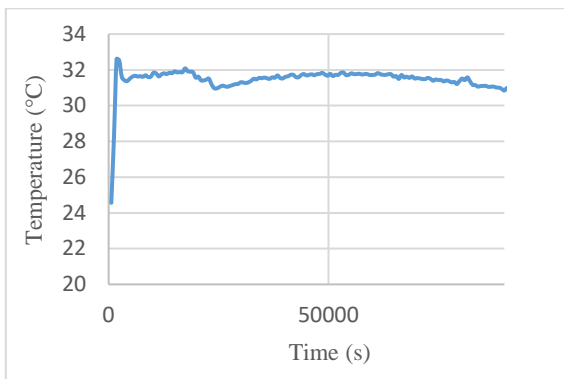
Figure 9. Dynamic behavior of the high temperature at high pressure on system 1.

Figure 10. Dynamic behavior of the low temperature at high pressure on system 1.

Figure 11. Dynamic behavior of the low temperature at low pressure on system 1.

Figure 12. Dynamic behavior of the common high temperature at low pressure.

Figure 13. Dynamic behavior of the high temperature at high pressure on system 2.
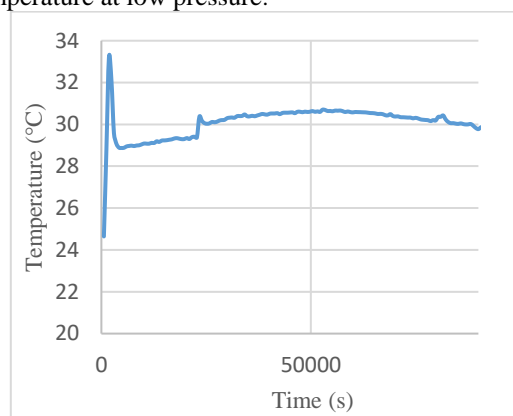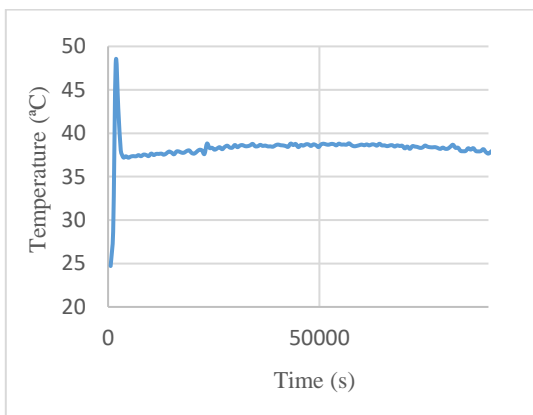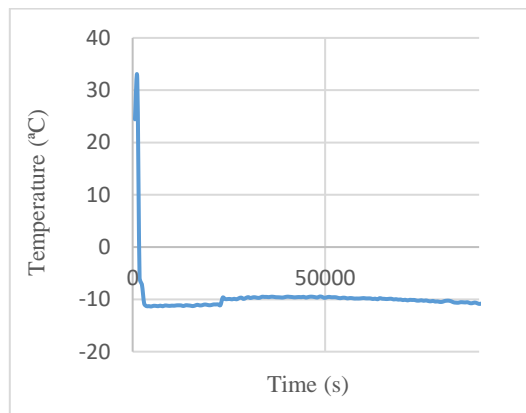

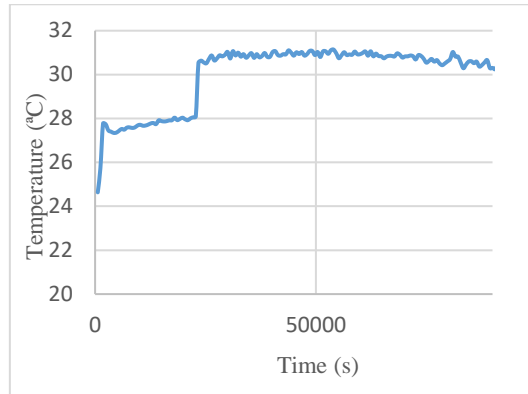**Figure 14.** Dynamic behavior of the low temperature at high pressure on system 2.

Figure 15. Dynamic behavior of the low temperature at low pressure on system 2.

Then by considering the transfer functions from each relationships of these experimental results, their magnitude spectra was estimated by a Fast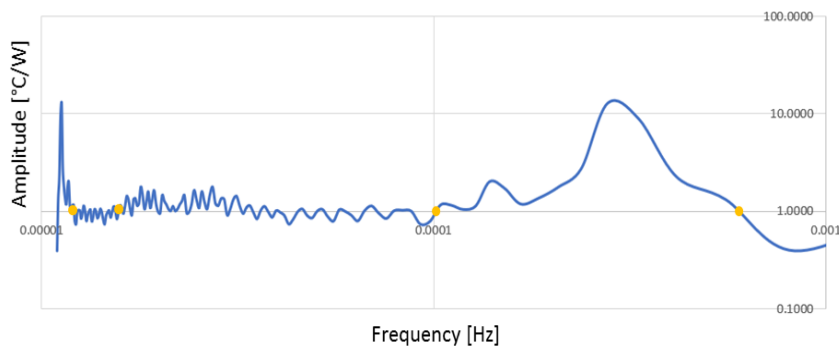 Fourier Transform algorithm, and by analyze them graphically the poles (maximums) and zeros (crosses) were estimated as well as Figs. 16 and 17 show it. Therefore the resulted poles polynomial is

$$P(\omega) = (\omega - 2\pi[0.0002778])(\omega - 2\pi[0.0001388])(\omega - 2\pi[0.0000273])$$
$$(\omega - 2\pi[0.0000245])(\omega - 2\pi[0.0000193])(\omega - 2\pi[0.0000179])$$
$$(\omega - 2\pi[0.00000112])$$

(4)
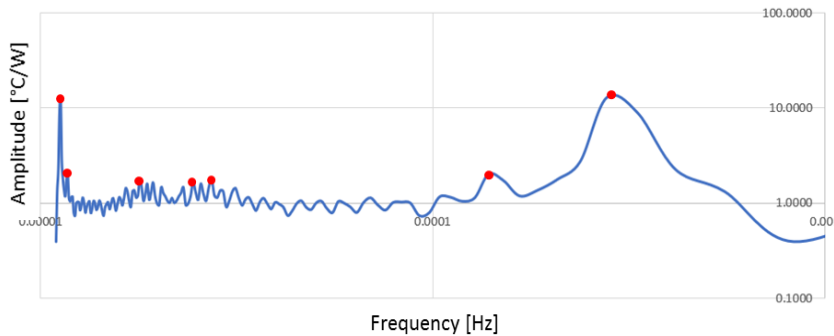
POLES
0.000277778
0.000138889
1.12613E-05
1.17371E-05
1.79211E-05
2.73224E-05
2.4510E-05

Figure 16. Graphical analysis from a transfer function spectra to get the poles location.

Figure 17. Graphical analysis from a transfer function spectra to get the zeros location.

And the concerned zeros for each variables relationship are listed on table 2.

Table 2. Model zeros list.

	Z1 [Hz]	Z2 [Hz]	Z3 [Hz]	Z4 [Hz]	Z5 [Hz]	Z6 [Hz]
$Z_{1,1}$	1.1990E-05	1.6181E-05	3.0303E-05	0.00010417		
$Z_{1,2}$	1.1820E-05	1.5015E-05	2.9240E-05	5.7471E-05	0.00033333	
$Z_{1,3}$	1.3123E-05	2.1368E-05	6.4103E-05	0.00055556		
$Z_{1,4}$	1.1655E-05	1.5432E-05	1.9841E-05	2.4155E-05	0.00166667	
$Z_{1,5}$	1.4749E-05	3.9683E-05	9.8039E-05	0.000555556		
$Z_{1,6}$	1.1494E-05	1.5873E-05	3.4722E-05	9.8039E-05	0.000416667	
$Z_{2,1}$	1.1261E-05	1.5015E-05	3.6232E-05	0.00033333		
$Z_{2,2}$	1.1038E-05	1.4493E-05	1.8315E-05	2.8736E-05	3.7879E-05	0.00055556
$Z_{2,3}$	1.4124E-05	2.1368E-05	7.5758E-05	0.00055556		
$Z_{2,4}$	1.1038E-05	1.5576E-05	2.6042E-05	0.00041667		
$Z_{2,5}$	1.1037E-05	1.5723E-05	3.3333E-05	0.000111111	0.000166667	0.000208333
$Z_{2,6}$	1.2073E-05	1.5576E-05	3.3333E-05	0.000128205	0.000208333	0.000416667
$Z_{3,1}$	1.2531E-05	1.7921E-05	3.4722E-05	0.00033333		
$Z_{3,2}$	1.1111E-05	1.4124E-05	1.7361E-05	2.9240E-05	5.9524E-05	0.00041667
$Z_{3,3}$	1.3550E-05	1.9841E-05	0.00011905	0.00055556		
$Z_{3,4}$	1.1111E-05	1.1820E-05	1.4368E-05	1.9608E-05	2.4876E-05	0.00041667
$Z_{3,5}$	1.0964E-05	0.001666667	3.7788E-05	0.00023810	0.00033333	0.00041667
$Z_{3,6}$	1.1737E-05	3.8760E-05	0.0002381	0.0003333	0.0005556	
$Z_{4,1}$	1.1338E-05	1.3550E-05	1.9608E-05	2.4876E-05	9.2593E-05	0.00041667
$Z_{4,2}$	1.1038E-05	1.4124E-05	1.8727E-05	2.6882E-05	4.3860E-05	0.00055556
$Z_{4,3}$	1.5015E-05	2.0833E-05	5.9524E-05	0.00055556		
$Z_{4,4}$	1.1038E-05	1.2438E-05	1.6835E-05	1.8315E-05	2.4155E-05	0.00055556
$Z_{4,5}$	1.263E-05	1.9380E-05	2.4876E-05	5.9524E-05	0.000238095	0.000333333
$Z_{4,6}$	1.1905E-05	1.9380E-05	2.5641E-05	8.7719E-05	0.000333333	0.001666667

$Z_{5,1}$	1.1905E-05	1.2438E-05	1.5723E-05	1.9157E-05	2.5253E-05	0.00041667
$Z_{5,2}$	1.1111E-05	1.3661E-05	1.7921E-05	2.8249E-05	3.7879E-05	0.00041667
$Z_{5,3}$	1.2438E-05	1.8939E-05	2.0576E-05	6.1728E-05	0.00055556	
$Z_{5,4}$	1.3228E-05	1.7921E-05	1.9608E-05	2.4510E-05	0.00033333	
$Z_{5,5}$	0.0000142	0.0000192	0.0000249	0.0000476	0.0002083	0.0003333
$Z_{5,6}$	1.1494E-05	1.5873E-05	3.4722E-05	9.8039E-05	0.000416667	
$Z_{6,1}$	1.1261E-05	1.3550E-05	2.0325E-05	2.4876E-05	8.3333E-05	0.00055556
$Z_{6,2}$	1.1111E-05	1.4493E-05	1.7007E-05	2.7778E-05	3.7037E-05	
$Z_{6,3}$	1.5576E-05	2.1097E-05	5.5556E-05	0.00055556		
$Z_{6,4}$	1.1111E-05	1.8315E-05	2.4876E-05	0.00012821	0.00055556	0.00166667
$Z_{6,5}$	1.1494E-05	1.4881E-05	1.9380E-05	2.4510E-05	0.000119	0.000417
$Z_{6,6}$	1.1111E-05	1.6340E-05	3.1447E-05	9.8039E-05	0.000333333	5.556E-04
$Z_{7,1}$	1.4124E-05	2.6042E-05	0.00016667	0.00083333		
$Z_{7,2}$	1.1261E-05	1.4006E-05	1.7361E-05	2.8736E-05	3.7037E-05	0.00033333
$Z_{7,3}$	1.4881E-05	2.1368E-05	6.4103E-05	0.00055556		
$Z_{7,4}$	1.1038E-05	1.3774E-05	3.2051E-05	0.00166667		
$Z_{7,5}$	1.1737E-05	1.4881E-05	3.2680E-05	0.000111111	0.000333333	0.000416667
$Z_{7,6}$	1.1111E-05	1.6340E-05	3.1447E-05	9.8039E-05	0.000333333	0.000555556

Due to the sample time of the data acquisition system the all frequency measurements have ± 0.0016 Hz of uncertainty. Fig. 18 shows the experimental setup of the lyophilization device used in this work.

Figure 18. Experimental setup of the lyophilization device.

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5.   Results analysis

Based on the transfer function matrix from the complete dynamical system of the analyzed lyophilization process, the model simulation results (output variables performance) for the same experimental scenario (temporal behavior of the set of input variables) was predicted. Figs. 19 and 20 show the temporal confrontation between the experimental results and the model simulation, for the internal temperature and pressure from the concerned lyophilization chamber.

Conclusion

Due to the zeros location listed on Table 2, it is true that electrical inertial moments are not significant for the spectral of interest in this study case. As the poles location dictates, the dynamic link between ambient temperature and pressure is strong and cannot be decoupling, statement that reinforces the thermodynamics in the between.
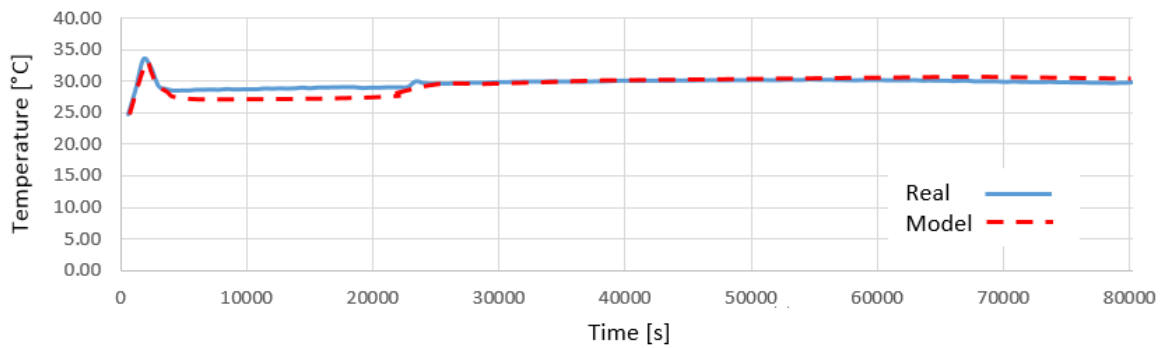

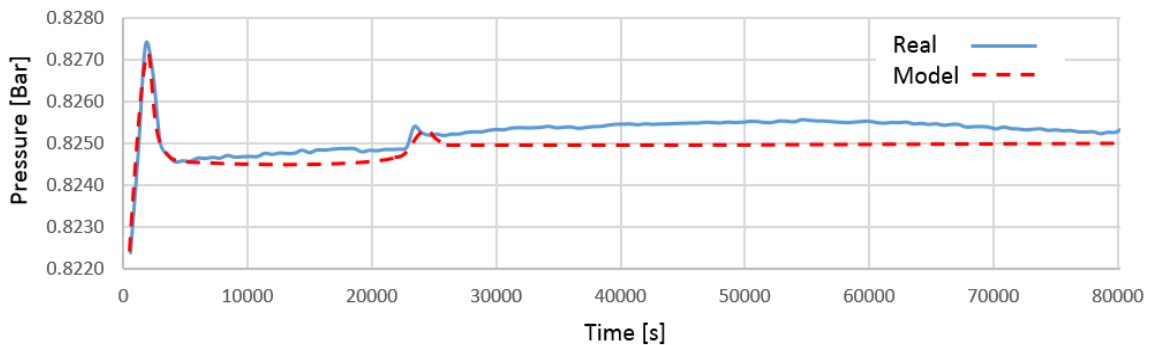**Figure 19.** Results of the temperature prediction from the lyophilization chamber.

Figure 20. Results of the pressure prediction from the lyophilization chamber.

It is shown the poles location does not depend of the three sub-process of the

lyophilization process, because they are define based on the transfer function and

do not on the activation time of the input variables which define the lyophilization device energetic performance.

The strongest dynamics links between input and output variables are the ones which present almost the same zeros location, and they came from the variables that represents the same energetic stage of the refrigerant gas in both vapor compression systems.

The results analysis statement listed before, has been reviewed by a refrigeration specialist board conformed by Technologists from the several Companies as BOHN, MABE, Guentner and by Scientifics from LaNITeF; then the most important results of this work is that this model can work as the baseline for the future development of a control algorithm and an energy optimization strategy consequently, because results are according with the time response expected from the lyophilization system.

Acknowledgment

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